NPS Phase III Remedial Investigation Sampling and Analysis Plan

Wrangell-St. Elias National Park and Preserve

Kennecott Mines and Mill Town Site
EDL # 5AKR3347

Prepared by

April 24, 2024

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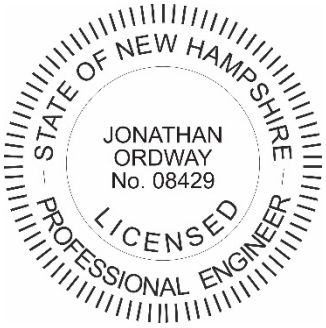

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LIST OF ABBREVIATIONS AND ACRONYMS

ACM	Asbestos Containing Material
ADEC	Alaska Department of Environmental Conservation
ANILCA	Alaska National Interest Lands Conservation Act
amsl	Above Mean Sea Level
bgs	Below Ground Surface
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
COC	Chain of Custody
COPC	Contaminant of Potential Concern
COPEC	Contaminant of Potential Ecological Concern
CSM	Conceptual Site Model
DOC	Dissolved Organic Carbon
DQO	Data Quality Objective
DQI	Data Quality Indicator
DU	Decision Unit
ECCD	Environmental Compliance and Cleanup Division
EDL	Environmental and Disposal Liabilities
ESV	Ecological Screening Value
EU	Estimation Unit
F	Fahrenheit
FEMA	Federal Emergency Management Agency
FSP	Field Sampling Plan
H_A	Alternative Hypothesis
HASP	Health and Safety Plan
H_0	Null Hypothesis
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation Derived Waste
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology Regulatory Council

IVBA	In Vitro Bioaccessibility Assay
KNHL	Kennecott Mines National Historic Landmark
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
mg/kg	Milligram per kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NELAP	National Environmental Laboratory Accreditation Program
NPS	National Park Service
NRWQC	National Recommended Water Quality Criteria
ORP	Oxidation Reduction Potential
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Level
PCB	Polychlorinated Biphenyl
PEF	Particulate Emission Factor
PIQ	Principal Investigation Question
PVC	Polyvinyl Chloride
QAPP	Quality Assurance Project Plan
QA	Quality Assurance
QC	Quality Control
QL	Quantitation Limit
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SIM	Selected Ion Monitoring
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedures
SU	Sampling Unit

SVOC	Semi-volatile Organic Compound
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
THQ	Target Hazard Quotient
TOC	Total Organic Carbon
TR	Target Risk
UCL	Upper Confidence Limit
µm	Micron
U.S.C.	United States Code
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
VHB	Vanasse Hangen Brustlin, Inc.
VOC	Volatile Organic Compound
WRST	Wrangell-St. Elias National Park and Preserve
XRF	X-Ray Fluorescence

1 INTRODUCTION

This Sampling and Analysis Plan (SAP) was prepared to support Phase III of the multiple-phase, multiple-year Remedial Investigation (RI) for the Kennecott Mines and Mill Town Site (the Site[1]). During previous investigations, including the Preliminary Assessment (Amec, 2018), the National Park Service (NPS) determined that releases of contaminants associated with historical mining and milling activities (including metals, semi-volatile organic compounds [SVOCs][2], and volatile organic compounds [VOCs]) have occurred at the Site and concluded that additional exploration and testing is needed to:

- Evaluate the nature and extent of the release of hazardous substances, pollutants or contaminants that has occurred at the Site by sampling environmental media;
- Evaluate the nature of threats to public health or welfare or the environment related to the release or threatened release of hazardous substances, pollutants or contaminants, including an assessment as to whether hazardous substances, pollutants or contaminants are migrating to the surrounding environment; and
- Provide the basis for recommending a remedial action for the Site.

The RI is being performed pursuant to the delegated authority provided to NPS under Section 104 of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) 42 United States Code (U.S.C.) §§ 9601, *et. seq.* The Site is located in the eastern portion of Alaska, approximately 100 miles from the Pacific coast and 230 miles from Anchorage in the Wrangell-St. Elias National Park and Preserve (WRST, Park) (**Figure 1**). The Site Environmental and Disposal Liabilities (EDL) number is 5AKR3347. The activities conducted during the RI correspond to the activities conducted under Phase 2 (Cleanup Action Selection), Sub-phase 2a, (Site Investigation Planning) and Sub-phase 2b, (Site Fieldwork), of the NPS Contaminated Site Cleanup Framework.

The key components of the SAP include the Data Quality Objectives (DQOs), the Field Sampling Plan (FSP), and the Quality Assurance Project Plan (QAPP). DQOs (**Section 4**) are the basis for developing principal investigation questions (PIQs), which are used to identify what data are required to complete the Phase III RI. The FSP (**Section 5**) describes the number, types, and locations of samples and the types of analyses that will be conducted on the samples. The QAPP includes a series of tables (attached **Tables 1 through 10**) that conform to the Uniform Federal Policy for QAPPs. The QAPP describes the project's policy, organization, and functional activities relative to the DQOs and identifies measures necessary to achieve the goals of the study.

This SAP was prepared by Vanasse Hangen Brustlin, Inc. (VHB) under Contract No. 140P2121A0026. The SAP was prepared in accordance with the United States Environmental Protection Agency's (USEPA) *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006), *Guidance for Quality Assurance Project Plans* (USEPA, 2002), *EPA Requirements for Quality Assurance*

[1] Note that the Site definition and boundaries may require modification as investigations into the nature and extent of contamination progress.

[2] Polycyclic aromatic hydrocarbons (PAHs) are a subset of SVOCs that result from burning coal, oil, gas, wood, garbage, and tobacco.

Project Plans (USEPA, 2001), and the Intergovernmental Data Quality Task Force's (IDQTF) *Uniform Federal Policy for Quality Assurance Project Plans* (IDQTF, 2005).

2 BACKGROUND

2.1 SITE DESCRIPTION

As shown on the Site Location Map (**Figure 1**), the Site is located within the 13.2 million-acre WRST in Alaska, approximately 3.5 miles north of the town of McCarthy. The following information can be used to locate the Site:

- Site entrance is located at coordinates 61.485167, -142.888722, McCarthy Road, Kennecott, Alaska 99566.
- Government employee, contractor, and local community vehicle access via private toll bridge (permit required).
- Visitor access via the Kennicott River footbridge (no vehicle access for visitors).

As shown on the Site Layout and Land Ownership Map (**Figure 2**), much of the Site is located within the Kennicott Subdivision, which comprises five subdivision units with interspersed public (NPS-managed) and privately owned lands crossed by road easements that provide access to lot owners and guests. Management of the Kennicott Subdivision is not under the authority of NPS. Within the Kennicott Subdivision, NPS has primary authority only on properties owned by the United States and NPS is legally bound to the restrictions noted on the plat and deed covenants.

While the Site includes the historic Mill Town and mines associated with former Kennecott operations, Phase III RI activities will be conducted in the vicinity of the approximately 80-acre Mill Town portion of the Site (see **Figure 2**). The Mill Town functions as a center for exploring the surrounding Park. Visitors walk throughout the Mill Town and the mines on both self-guided and commercially guided tours.

2.1.1 Topography and Drainage

The Mill Town is located on the western slope of Bonanza Ridge near the confluence of the Root and Kennicott glaciers, on the valley wall above glacial moraine. The forested area east of the Mill Town is characterized by steep mountains climbing in elevation from 2000 feet above mean sea level (amsl) near the Mill Town to nearly 6000 feet amsl towards the east in the area where the historical mines operated within Bonanza Ridge. Glacial moraine is located immediately west and downslope of the Mill Town and varies in elevation. Glacial moraine is located along the margins of the Kennicott Glacier. Surface runoff from precipitation and snowmelt likely flows from the topographic highs within the eastern areas of the mountains towards National Creek and the glacial moraine. See **Figure 3** for topography and inferred surface water runoff pathways.

2.1.2 Surface Water and Wetlands

The Site is located within the Lower Kennicott Glacier watershed. The occurrence of surface water at the Site is likely due to glacial melt, groundwater discharge, precipitation runoff, and snow melt. National

Creek bisects the Mill Town running from east to west and is a source of drinking water for staff, residents, and visitors of the Site. Where National Creek flows through the moraine field adjacent to and downstream of Mill Town, it creates a dynamically changing system of channels, ponds, and small lakes among the moraine deposits. During the Phase II RI, National Creek discharged to the moraine field and was no longer visible on the surface approximately one-half mile southwest of the Mill Town. The runoff from the moraine field flows into a proglacial lake approximately 3 miles to the south prior to discharging to the Kennicott River.

A group of spring and creeks collectively referred to as Clear Creek are also located within the Kennicott Glacier outwash zone and are used as a drinking water source for the Town of McCarthy (Hecht & LaChapelle, 1999; Amec, 2018). The approximate location of Clear Creek is identified on **Figure 1**. Clear Creek is fed from an aquifer of unconsolidated glacial outwash recharged principally from permeable terrace deposits north and east of McCarthy and from dissected Kennecott Glacier outwash gravels visible along and to the west of the road to Kennecott. Additional recharge originates directly or indirectly from the slopes of Porphyry Mountain to the northeast of the McCarthy airstrip (Hecht & LaChapelle, 1999). It is unknown whether National Creek is hydraulically connected to the downstream drinking water sources, however, a 1999 hydrologic assessment of Clear Creek found that surface and subsurface drainage from within the Kennecott Mill Town flow considerably westward of inferred recharge areas for Clear Creek, and therefore, were considered unlikely to be a source of potential contamination to the drinking water supply (Hecht & LaChapelle, 1999). McCarthy Creek drains the eastern side of Bonanza Ridge and joins the Kennicott River approximately 1.5 miles south of the town of McCarthy.

2.1.3 Structures and Utilities

The Kennecott Mill Town is located within the "Millsite Unit" of the Kennicott Subdivision and is comprised of over 100 historic structures. The Mill Town was built to receive, concentrate, store, and transport copper ore mined from Bonanza, Jumbo, Erie, and Glacier Mines. A tramway transported material from the mines to the Mill Town, and the railroad was used to transport the concentrated ore from the mill to Cordova, Alaska. Substantial facilities were developed around the mining operation including the local Power Plant (Building 9 on **Figure 2**), a Mill Building (Building 3 on **Figure 2**), and an ammonia Leaching Plant (Building 15 on **Figure 2**), all of which supported the milling operation. The Power Plant used a fuel believed to be a heavy oil similar to Bunker C for heating and electrical power supply. Machine shops, repair shops, administrative offices, material stores, large bunk houses, single staff housing, and private residences for management were developed around the main milling facilities (Gilbert et al., 2001).

Visitor exhibits and staff offices are located within several NPS-managed historic structures. Visitor use of the Mill Town is generally unrestricted, except for the area immediately north of the Mill Building, an area west of the Leaching Plant, and the interiors of some buildings (most notably the Mill Building and Power Plant). These interior spaces are accessible only on paid, guided tours (NPS, 1996; EMCON, 1992). A number of historic buildings have been renovated for private residential use. Privately owned buildings are generally used as summer or seasonal residences or businesses. A privately-owned lodge, constructed in 1981 (Building 17, **Figure 2**), attracts domestic and international visitors. See **Figure 2** for the layout of the Mill Town buildings, other Site features, and identification of public and private lands.

Buried and aboveground utilities are present at the Site. **Figure 2** compiles known and/or suspected utility locations based on historical fire insurance maps and Park records.

2.1.4 Regional and Local Geology

The origins and composition of the bedrock below and surrounding the Mill Town are complex due to the active thermal features and fault lines in the area. Bedrock in the vicinity of the Mill Town is composed of several igneous and sedimentary units crosscut by both valley and cirque glaciers that largely define localized topography (Trop et al., 2021). The Mill Town is primarily sited over two bedrock units: the Nikolai Greenstone; and the Chitistone and Nizina Limestones (Wilson et al., 2015; see **Figure 4**). The Nikolai Greenstone is an igneous basalt deposited in a sub-aerial to sub-marine depositional environment. The Chitistone and Nizina Limestones formed in a marine environment and are characterized by stromatolites (layered sedimentary formations created by photosynthetic cyanobacteria) and evaporites (water-soluble mineral sediment resulting from concentration and crystallization by evaporation from aqueous solutions). These formations include rocks associated with the copper sulfide mineral deposits (e.g., chalcocite) mined by the high-grade copper operation on Bonanza Ridge located to the east of the Mill Town. Alkaline conditions associated with calcium carbonate from limestone rocks in the area may neutralize acidic drainage typically associated with sulfide ore mine waste and may explain why characteristic signs of acid mine drainage (low pH, staining/discoloration) have not been encountered at the Site.

Valley and cirque glaciation play a significant role in the geomorphology of the Site. As the glaciers advance and recede, they weather, mix, transport, and deposit sediments scoured from the geologic units they cut through along their path. The Root and Kennicott Glaciers dominate the landscape and geomorphologic activity to the west of the Mill Town, and historically under the Mill Town. The terminal moraine deposited at the southern end of the glacier, near McCarthy, and the lateral moraines deposited parallel to the valley wall along the Mill Town are composed of a mixture of glacial till left by the receding glacier, colluvium, alluvium, and mine tailings. The receding cirque glaciers on the ridges above the Mill Town also weather, transport, and deposit sediments at the Mill Town and surrounding areas.

The depth to bedrock is expected to be relatively shallow (zero to tens of feet) over much of the Mill Town; however, thicker overburden deposits may be present in certain areas. Unconsolidated materials are present where bedrock is not exposed at the surface. These materials are sourced from several geologic units, including those from which ore was mined. In addition to the typical mechanical and chemical weathering processes that turn bedrock into sediment and the processes that transport and deposit sediment throughout a watershed (including creeks), the Mill Town also has two relatively unique transportation and deposition features: glaciation; and anthropogenic mining and transportation of rocks and minerals to the Mill Town. Therefore, overburden in the Mill Town includes mine tailings that likely overlay glacial till and bedrock.

2.1.5 Regional Hydrogeology

The Nikolai Greenstone and the Chitistone and Nizina Limestones are not considered productive aquifers (Amec, 2018; Winkler et al., 2000). Fractures in the bedrock may act as conduits for groundwater flow and contaminant transport. Groundwater is inferred to flow from the higher elevation mountains towards the moraine field in the Kennicott Glacier valley, and at times is likely feeding surface water and National Creek (see **Figure 3**) (Amec, 2018). Groundwater elevation is expected to vary throughout the year depending on precipitation levels. The occurrence of seeps or springs and the interaction between groundwater and surface water at the Site are likely dynamic and vary temporarily due to seasonal changes in precipitation, snowmelt, and groundwater elevations. Seeps were observed in multiple locations within the Mill Town adjacent to bedrock outcrops during the Phase I RI (see **Figure 3**) but were not observed during the Phase II RI. Groundwater may flow into creeks draining the ridge, seeps or springs, or into the mix of surface water and glacial melt water within the glacial moraine along the margin of the Kennecott Glacier.

2.1.6 Sensitive Environments

Sensitive environments are defined by USEPA as terrestrial or aquatic resources, a fragile natural setting, or other areas with unique or highly valued environmental or cultural features; USEPA considers all National Parks sensitive environments (USEPA, 1992).

The FWS Information for Planning and Consultation tool was used to identify listed species and migratory birds that may be present at the Site, using the Mill Town and its vicinity as the search area (Fish & Wildlife Service, n.d.). The Kennicott and Nizina Rivers were reported as migration routes for the sockeye and king salmon in the 1995 Site Inspection Report; however, harvest data for fisheries are not reported by the Alaska Department of Fish and Game for these rivers nor was any fishing observed along these rivers in the 2017 Site Reconnaissance (Amec, 2018). While National Creek bisects the Site, the National Wetland Inventory does not identify wetlands at the Site (Fish & Wildlife Service, 2004). Migratory birds may be present (lesser yellowlegs, olive-sided flycatcher), but endangered species are not reported to be located in the Mill Town or vicinity (Amec, 2018; Fish & Wildlife Service, n.d.).

2.1.7 Historically and Culturally Significant Resources

Kennecott Mines and Mill Town played an important role in the history of Alaska and the American mining industry; therefore, the buildings and other features present at the time of historical operations are considered historically and culturally significant. The mines that contributed to the operation were designated a National Historic Landmark in 1986 and the Mill Town followed in 1998.

NPS manages the Kennecott Mines National Historic Landmark (KNHL) to protect the cultural and natural resources of the historic mining district and the surrounding glacial landscape; and provide a safe, educational, and rewarding experience for the area's visitors and residents. A focus for management of the KNHL is to stabilize, preserve, and interpret the key patterns, relationships, and remaining structures and features that define the historic, cultural and natural character of the KNHL. NPS preserves

archeological resources contributing to the character and interpretation of the KNHL. If removal of such resources is required for stabilization projects, archeological objects or features are returned to their original location upon completion of the project when feasible (NPS, 2014).

2.1.8 Local Climate and Natural Hazard Assessment

This region of Alaska has a subarctic climate characterized by long, cold winters and short, cool to mild summers. Precipitation is relatively low, but snowfall can be significant. The Kennecott Mill Town is open to the public from late May to mid-September when the weather is more moderate than during the winter months. Based on climate data for nearby McCarthy, July is the hottest month with an average high temperature of 71 degrees Fahrenheit (F); January is the coldest month with an average low temperature of -9 degrees F. The average annual precipitation and snowfall are 18.32 inches and 69 inches, respectively (United States Climate Data, 2023).

According to the Federal Emergency Management Agency (FEMA) National Risk Index for the Copper River Census Area, Alaska, the risk for avalanches, earthquakes, flooding, wildfires, and volcanic activity is low (FEMA, 2023).

2.1.9 Current and Anticipated Future Land Use

The Kennecott Mill Town is a popular tourist destination and the most visited area within WRST. The expected future use of the Site is historic interpretation and outdoor recreation, in addition to the subsistence uses provided for under the Alaska National Interest Lands Conservation Act (ANILCA) of 1980, 16 U.S.C. § 410hh(9).

2.2 OPERATIONAL HISTORY

The Kennecott Mines and Mill Town were operated during the first four decades of the twentieth century (1911 to 1938). The initial discovery of the Kennecott copper deposits was made in 1900 on Bonanza Ridge. The Kennecott Mill Town was built in 1907 by the Kennecott Copper Corporation to receive, concentrate, store, and transport the ore mined from Bonanza, Jumbo, Erie, and Glacier Mines. Bonanza Mine was the first to be developed. Mill Town processing of the ore from Bonanza Mine and shipment of processed ore began in 1911 when the railroad from Kennecott Mill Town to Cordova was completed. Jumbo Mine was opened in 1913, followed by Erie Mine in 1916, and eventually Glacier Mine in 1920. Production peaked in 1923 to 1924 (Gilbert et al., 2001). Tramways transported material from several of the mines to the Mill Town, and the railroad was used to transport the concentrated ore from the mill to Cordova, Alaska. From Cordova, the ore was transported via steamship line to the American Smelting and Refining Company, a smelter located in Tacoma, Washington (Gilbert et al., 2001).

Substantial and sophisticated facilities were developed around the mining operation to advance the work and support the workforce. The largest facilities were the Mill Building (Building 3 on **Figure 2**), the local Power Plant (Building 9 on **Figure 2**), which used a fuel believed to be a heavy oil similar to

Bunker C for heating and electrical power supply, an ammonia Leaching Plant (Building 15 on **Figure 2**), and the North Sacking Shed and Flotation Plant (Building 21 on **Figure 2**), all of which supported the milling operation. Machine shops, repair shops, administrative offices, material stores, large bunk houses, single staff housing, and private residences for management were developed around the main milling facilities (Gilbert et al., 2001).

Copper production declined in the 1930s, and the mines were temporarily shut down between 1932 and 1935. Low prices for copper and the depletion of the ore body finally caused the closure of the mines in 1938 and abandonment of the town. Eventually, the rails and ties were removed from the railroad grade, leaving behind graded vehicle access between McCarthy and Kennecott Mill Town (Gilbert et al., 2001).

In 1965, the Consolidated Wrangell Mining Company acquired rights to mine copper from surface deposits below the Bonanza Mine. The company installed a bunkhouse and a heavy media separation plant, but the operation proved unsuccessful, and the plant was dismantled and removed. Two years later, the company installed a more traditional mechanical separation unit and again attempted operations for a brief time. Most of the milling machinery was left behind at the closure in the mid-1970s and remains scattered throughout the Mill Town (Gilbert et al., 2001).

The land rights were eventually acquired by the Great Kennicott Land Company in 1976. The land was subdivided for sale to the public and within the Mill Town almost every building was designated its own lot. During this time of ownership, several renovations and demolitions occurred (Gilbert et al., 2001). Although WRST was created in 1980 by the United States Congress, the Kennecott Mill Town was not acquired by the Federal Government until 1998. Preservation measures have been taken by NPS and Friends of Kennicott throughout the years to increase visitation (Gilbert et al., 2001; NPS, 1996).

2.3 PREVIOUS INVESTIGATIONS AND RESPONSE ACTIONS

2.3.1 Pre-RI Investigation and Remediation Activities

Multiple environmental investigations and remediation activities were undertaken at the Site prior to initiation of the CERCLA RI. Section 2.2 of the Kennecott Mines and Mill Town Phase I RI SAP (VHB, 2021) contains a detailed summary of these activities, as well as an assessment of the quality and usability of the resulting data (e.g., environmental sample analytical results, etc.). Applicable data/information compiled as part of the historical review process were taken into consideration during the scoping of the RI activities completed to date, as appropriate. Summaries of the Phase I and II RIs are provided below.

2.3.2 2021 Phase I RI

In August 2021, VHB, on behalf of NPS, completed the first phase of the multiple phase RI. The primary purpose of the Phase I RI was to collect data to evaluate the nature and extent of potential contaminants in air, surface soil, sediment, and surface water in the Mill Town portion of the Site. The data collected during the Phase I RI was also intended to support future risk assessments and decision making regarding

potential response actions that may be undertaken by NPS or other parties. In general, sample results were compared to risk-based Project Action Levels (PALs) and/or preliminary field background values to identify the potential extent of impacts. PALs represent conservative screening levels; exceedance of PALs does not necessarily indicate there is a threat to human health or the environment. See **Section 4** for more information regarding PALs and **Tables 6a, 6b, 6c, and 6d** for a listing of PALs. Six DQO investigations were conducted under the Phase I RI. See **Figure 5** for Phase I RI sample locations.

DQO 1 – Ambient Air Investigation

The overall goal of the DQO 1 field program was to collect sufficient data to allow assessment of standard CERCLA risk assessment modeling approaches for outdoor ambient air, given the Site's unusual physical/climate setting. These approaches rely on soil analytical data and assumptions associated with airborne particulate emission rates to estimate ("model") outdoor air concentrations and related inhalation risks.

The DQO 1 investigation was designed to monitor the range of outdoor air preliminary contaminant of potential concern (COPC) concentrations under a variety of conditions (e.g., wind/weather, traffic, construction activities) and compare those results to applicable PALs, as well as "modeled" outdoor air preliminary COPC concentrations. Air samples were collected from six air stations and analyzed for 23 target analyte list (TAL) metals and asbestos. DustTrak aerosol monitors were also located at each air station to collect real-time airborne particulate (dust) measurements and a weather station was deployed to record Site meteorological conditions.

The DQO 1 investigation findings are as follows:

- Although multiple rain events occurred during the field program, air samples were collected during brief dry periods. Copper, calcium, and asbestos were detected at only one air station and concentrations were less than PALs (or the detected preliminary COPCs do not have PALs).
- Particulate matter concentrations were similar across air stations and corresponded with a "good" air quality index.
- Air and correlated weather data indicated that wind was a primary generator of airborne dust during the Phase I RI field program. Due to the wet conditions, the lack of sustained traffic, and absence of ongoing construction work, dust generation during this period was relatively low and high dust conditions were not observed and/or sampled during the Phase I RI.
- As a result of these conditions and the limited number of detections in air samples, robust statistical comparison of measured air concentrations to modeled air concentrations could not be completed.
- As an alternative method of assessment, a sensitivity analysis was performed of one of the key model input parameters: the particulate emission factor (PEF) recommended by the Alaska Department of Environmental Conservation (ADEC) (ADEC, 2018).
    - The results of this analysis indicated that the ADEC PEF is a conservative representation of typical Site conditions under ambient wind conditions and additional data collection to develop a Site-specific PEF is not required to support future evaluations of the inhalation exposure pathway using the standard CERCLA risk assessment approach.

DQO 2 – Historical Paint Investigation

The DQO 2 investigation was designed to assess the horizontal extent of surface soil with metals concentrations that exceed preliminary "field" background levels in the vicinity of known or suspected lead- and arsenic-based paint use areas. Handheld X-ray fluorescence (XRF) analyzers were used to field screen discrete surface soil samples collected from 0 to 6 inches below ground surface (bgs) for metals concentrations around ten buildings primarily associated with Kennecott Mill Town operations and ore processing in the northern portion of the Site. In order to inform sampling extent of the adaptive field program, metals concentrations detected with the XRF analyzers were compared to mean field (XRF) background concentrations established at the outset of the field program using samples collected north of the Kennecott Mill Town[1]. Confirmation samples were sent to a National Environmental Laboratory Accreditation Program (NELAP) accredited environmental laboratory to assess the performance of the XRF. Sampling for VOC and SVOC analyses was also conducted around the Oil House to assess potential impacts to surface soil from historical operations within the building.

The DQO 2 investigation findings are as follows:

- The XRF appeared to be an effective field screening tool for 11 metals (antimony, arsenic, barium, cadmium, copper, iron, lead, mercury, nickel, silver, and zinc).
- Lead and arsenic concentrations were observed to be elevated in surface soil samples collected within 20 feet of buildings assessed during the Phase I RI.
    - The full extent of preliminary COPC concentrations greater than field background could not be delineated based on the data collected during the Phase I RI.
- Impacts of metals from historic paint and tailings (DQO 3 investigation) are intermixed and cannot be differentiated.
- Low level PAL exceedances were noted for SVOCs in surface soil samples collected at the Oil House (benzo(a)pyrene and naphthalene). Benzene was detected at a concentration greater than the PAL in one of the three samples collected from the Oil House (at a sample collected within one foot of the building). Other VOCs were not detected at concentrations greater than PALs.

DQO 3 – Mill Tailings and Ore/Ore Concentrates Investigation

The DQO 3 investigation was designed to assess the horizontal extent of historical tailings and tailings-impacted surficial soil in the vicinity of previously mapped tailings piles as indicated by metals concentrations relative to preliminary field (XRF) background levels. Handheld XRF analyzers were used to field screen discrete surface soil samples (0 to 6 inches bgs) for metals concentrations around previously mapped tailings piles in the northern portion of the Site. Confirmation soil samples were analyzed by a NELAP accredited environmental laboratory to assess the performance of the XRF.

The DQO 3 investigation findings are as follows:

[1] Eighteen soil samples were collected from two areas north of the Site during the Phase I RI and analyzed using the XRF analyzer to establish preliminary "field" background metal preliminary COPC concentrations to preliminarily inform sampling extents given the adaptive nature of the field investigation. A formal background study has not been conducted to date.

- Metals concentrations were observed to be elevated in surface soil samples collected in the vicinity of previously mapped tailings piles in the northern portion of the Kennecott Mill Town.
  - The full extent of preliminary COPC concentrations greater than field background could not be delineated based on the data collected during the Phase I RI.
- Copper, lead, and arsenic concentrations are elevated in surface soil samples collected from within visible tailings deposits, as reported in XRF and laboratory analytical data.
- Given the orders of magnitude difference between copper concentrations in field background samples and in tailings samples, copper may be used as an "indicator metal" to assess the extent of tailings-impacted soil relative to field background concentrations.

Because impacts of metals from historic paint and tailings are intermixed and appear to extend over a significant area, it was recommended that future investigations should evaluate metals in surface soil using XRF field screening tools and a methodology consistent with that used for DQO 3, regardless of the suspected source of metals.

DQO 4 – Blasting Cap Disposal Area Investigation

The DQO 4 investigation was designed to assess the horizontal extent of surface soil with concentrations of preliminary COPCs (metals, VOCs, SVOCs, and explosives) that exceed PALs in the Blasting Cap Disposal Areas (see **Figure 5**). Incremental sampling methodology (ISM) was utilized to collect surficial soil samples from 0 to 6 inches bgs in areas of known/observed blasting caps disposal (Blasting Cap Disposal Area-East and Blasting Cap Disposal Area-West).

The DQO 4 investigation findings are as follows:

- Explosives and VOCs were not detected at concentrations greater than PALs in samples collected from the Blasting Cap Disposal Areas.
- Metals (aluminum, antimony, arsenic, barium, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, silver, vanadium, and zinc) and SVOCs (benzo(a)pyrene and naphthalene) were detected at concentrations exceeding PALs.
  - The extent of metals and SVOCs in surface soil at concentrations exceeding PALs could not be fully delineated based on the data collected during the Phase I RI.
- Concentrations of metals and SVOCs detected in DQO 4 soil samples were similar to concentrations detected in the DQO 2 and DQO 3 soil samples collected near the Blasting Cap Disposal Areas.

DQO 5 – Transformer House Investigation

The DQO 5 investigation was designed to assess the horizontal extent of surface soil (0 to 6 inches bgs) with transformer oil preliminary COPCs (TAL metals, VOCs, SVOCs, and polychlorinated biphenyls [PCBs]) in the vicinity of the Transformer Building. Given the historical building use/operation, surface soil samples were collected from around and under the Transformer House for laboratory analysis.

The DQO 5 investigation findings are as follows:

- PCB Aroclors were not detected in surface soil samples collected at the Transformer House, and therefore, it is unlikely that PCB-containing transformer oil was used in the building.

- Metals (aluminum, antimony, arsenic, barium, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, silver, vanadium, and zinc) and SVOCs (benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, chrysene, dibenz(a,h)anthracene, indeno(1,2,3-cd)pyrene, naphthalene, pyrene) were detected at concentrations exceeding PALs.
  - The extent of metals and SVOCs in surface soil at concentrations exceeding PALs could not be fully assessed based on the data collected during the Phase I RI.
- VOCs were not detected at concentrations exceeding PALs.

DQO 6 – National Creek Investigation

The DQO 6 investigation was designed to:

- Assess whether preliminary COPCs are present in National Creek surface water and sediment samples at concentrations exceeding PALs.
- Assess whether preliminary COPCs are present at greater concentrations in downstream surface water and sediment samples than upstream samples in National Creek.
- Identify locations of groundwater seeps, springs, or storm water run-off conveyances in the vicinity of the Kennecott Mill Town that could act as preliminary COPC sources to National Creek.

Collocated samples of surface water and sediment were collected from National Creek at upstream, midstream, and downstream locations (three samples per location). Two sampling events were conducted; one targeted event after a rainstorm (surface water samples collected for total and dissolved metals analysis only) and one scheduled event (full suite of preliminary COPCs collected in surface water and sediment[1]). Sediment samples were collected from 0 to 6 inches below the stream bed surface at locations containing fine-grained sediment and representing depositional environments. Where observed, the locations of seeps were recorded.

The DQO 6 investigation findings are as follows:

- Metals were detected at concentrations exceeding PALs in surface water (aluminum, arsenic, barium, copper, iron, manganese, silver) and sediment samples (aluminum, antimony, arsenic, barium, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, vanadium, and zinc) collected from National Creek.
- Concentrations in downstream surface water and sediment samples were greater than concentrations in upstream samples, indicating the Kennecott Mill Town may be a source of preliminary COPCs to National Creek.
  - The downstream extent of elevated preliminary COPCs in surface water and sediment could not be fully delineated using the data collected during the Phase I RI.
- Downstream impacts in surface water were most pronounced in samples collected after a rain event, indicating runoff may be an active migration pathway for preliminary COPCs.

[1] VOCs were not sampled in surface water during the Phase I RI because vials were mistakenly not provided by the laboratory.

- SVOCs, VOCs[1], and PCBs were either not detected or not detected above PALs in surface water and sediment (with the exception of chlorobenzene in two sediment samples).
  - Additional data are needed to confirm this finding under different streamflow conditions.
- Several seeps were identified in the vicinity of National Creek during the Phase I RI.

2.3.3 2023 Phase II RI

In July 2023, on behalf of NPS, VHB completed the second phase of the multiple phase RI. The primary purpose of the Phase II RI was to obtain data to further the understanding of the nature and extent of contaminants in surface soil, sediment, and surface water in the Mill Town portion of the Site. The data collected during the Phase II RI was also intended to support future risk assessments and decision making regarding potential response actions that may be undertaken by NPS or other parties. Similar to the Phase I RI, sample concentrations were compared to risk-based PALs and/or preliminary field background values to identify the potential extent of impacts. As noted above, PALs represent conservative screening levels; exceedance of PALs does not necessarily indicate there is a threat to human health or the environment. Two DQO investigations were conducted under the Phase II RI. See **Figure 5** for Phase II RI sample locations.

DQO 2/3 – Surface Soil Investigation

Because the Phase I RI found impacts from historical paint (DQO 2) and mill tailings/ore/ore concentrates (DQO 3) were intermixed, these DQOs were combined into a single DQO (DQO 2/3 Surface Soil) for the Phase II RI. The DQO 2/3 investigation was designed to assess the horizontal extent of surface soil with metal and SVOC concentrations that exceed PALs and/or background levels in areas of the Mill Town that were not investigated during the Phase I RI or where the Phase I RI indicated that contamination extended beyond the Phase I RI sampling area. Handheld XRF analyzers were used to field screen surface soils for metals concentrations around the Site. Two methods of field screening were employed during the Phase II program: (1) XRF screening of the ground surface and (2) XRF analysis of discrete surface soil samples. To assess whether field XRF results are consistent with laboratory results, 20% of the discrete surface soil samples analyzed by XRF were analyzed by a NELAP accredited environmental laboratory for TAL metals. To evaluate the broader spatial distribution of SVOCs in surface soil, 20 discrete surface soil samples were collected across the Site and analyzed by the environmental laboratory for SVOCs.

The DQO 2/3 findings are as follows:

- Metals, including but not limited to copper, arsenic, and lead, are present at concentrations that exceed PALs in surface soil across the Site.
- Metals concentrations are higher near former operational areas in the Mill Town portion of the Site, adjacent to historical buildings where lead- or arsenic-based paint was applied, in the tailings piles, and along Mill Town Roads.

[1] VOCs were not sampled in surface water during the Phase I RI because vials were mistakenly not provided by the laboratory.

- The horizontal extent of elevated metals in Site surface soil relative to field background concentrations has largely been identified.
  - Notable exceptions to this finding include areas under private land ownership, the major Mill Town roadways/transportation routes, the aerial tramways, and the meandering downstream channel(s) of National Creek.
- The distribution of low-level PAH concentrations in Site surface soil is relatively broad and interspersed throughout the developed portions of the Site.

DQO 6 – Surface Water and Sediment Investigation

The surface water and sediment investigation was designed to:

- Assess whether concentrations of COPCs in National Creek surface water and sediment samples exceed PALs during different stream stage/flow conditions.
- Assess whether COPCs in National Creek are present at greater concentrations in downstream surface water and sediment samples than upstream samples during different stream stage/flow conditions.
- Identify locations of groundwater seeps, springs, or storm water run-off conveyances in the vicinity of the Kennecott Mill Town that could act as COPC sources to National Creek.
- Assess the extent of downstream surface water and sediment COPC concentrations in National Creek that exceed PALs and/or upstream (reference) sample concentrations.
- Identify locations along the banks of National Creek downstream of the Mill Town where tailings are visibly present and could be acting as COPC sources to National Creek.

Collocated surface water and sediment samples were collected from National Creek at the approximate upstream, midstream, and downstream locations that were sampled during the Phase I RI (three samples per area). Because elevated concentrations of metal preliminary COPCs were observed in the downstream sample location during the Phase I RI, two additional downstream sampling locations (three samples per area) were included in the Phase II RI sampling scheme. Surface water and sediment samples were analyzed for the full suite of COPCs outlined in the 2022 Phase II RI SAP.

The DQO 6 surface water investigation findings are as follows:

- Seventeen of the 23 TAL metals were not detected at concentrations exceeding PALs;[1] aluminum, barium, cadmium, copper, iron, and mercury were detected at concentrations exceeding their respective PALs in at least one surface water sample.
- Concentrations of copper are greater in downstream surface water samples than in upstream and midstream samples. Similar concentration trends were observed in total (unfiltered) metals and dissolved (filtered) metals concentrations for copper.
- The trend of increased concentrations in downstream samples is less apparent with metals other than copper. Total mercury and cadmium concentrations are elevated in one downstream sample

[1] Arsenic was not detected in surface water samples collected during the Phase II RI. The laboratory detection limit was greater than the PAL.

(NCW-D5) compared to upstream and midstream results, and total lead and chromium are elevated in one midstream sample (NCW-M3) compared to upstream results.

- Six PAHs including anthracene, benzo(a)pyrene, benzo(b)fluoranthene, chrysene, dibenz(a,h)anthracene, and indeno(1,2,3-cd)pyrene were detected at concentrations exceeding PALs in at least one surface water sample (ranging from marginal exceedances to 150 times PALs). The PAL exceedances were associated with 7 of the 15 samples collected from upstream, midstream, and downstream locations. All detected concentrations of PAHs exceeding PALs were flagged as estimated by the laboratory.
- Chloromethane was the only VOC detected in surface water samples; all detections were flagged as estimated by the laboratory and the concentrations were less than the PAL.
- Similar to the Phase I RI findings, PCBs were not detected in surface water samples during the Phase II RI. [1]

The DQO 6 sediment investigation findings are as follows:

- Twelve of the 23 TAL metals were not detected in sediment samples at concentrations exceeding PAL (or do not have PALs); 11 metals, including aluminum, arsenic, cadmium, cobalt, copper, iron, manganese, mercury, nickel, silver, and vanadium, were detected at concentrations exceeding PALs in at least one sediment sample.
- Concentrations of arsenic, cadmium, calcium, copper, magnesium, mercury, and silver are greater in downstream and midstream sediment samples than upstream samples.
- SVOCs were not detected at concentrations exceeding PALs with the exception of di-n-butyl phthalate, which appears to be a laboratory contaminant (based on laboratory quality control (QC) data) and not Site related.
- VOCs were not detected at concentrations exceeding PALs, with the exception of toluene, which was detected at a concentration (0.000593 milligrams per kilogram [mg/kg]) greater than the PAL at one midstream duplicate sample location (NCS-L1). Toluene was not detected in the parent sample; however, it was detected in the equipment blank sample at a concentration lower than found in the NCS-L1 sample.
- Similar to the 2021 Phase I RI findings, PCBs were not detected in sediment samples during the Phase II RI. [2]

Groundwater seeps, springs, or storm water run-off conveyances were not observed during the Phase II RI field program. Tailings deposits were visible along the banks of National Creek extending from the Mill Town to the terminus of National Creek. Visible tailings deposits were observed to extend to McCarthy (see Figure 1) and may have been deposited by National Creek along historical flow paths or through glacial reworking or possibly through direct disposal in some areas.

[1] The surface water method detection limits for PCBs are greater than the PALs; therefore, it is possible that PCBs are present at concentrations greater than PALs but were not detected by this analysis.

[2] PALs are greater than the sediment method detection limits for PCBs.

3 CONCEPTUAL SITE MODEL

NPS updated the Conceptual Site Model (CSM) based on findings of this Phase II RI. **Figure 6** is a pictorial illustration of the CSM used to help visualize Site conditions. Supplemental discussion of the CSM based on both historical information and the results of the RI to date follows. A CSM evolves as additional data are collected; NPS anticipates that the CSM presented herein may continue to evolve as data are collected and analyzed during future phases of the RI.

3.1 KNOWN AND SUSPECTED CONTAMINANTS OF POTENTIAL CONCERN

Based on previous investigations, six primary sources of contamination have been identified at the Site:

- Mill tailings/ore & ore concentrates:
  - Tailings contain arsenic, cadmium, lead, and mercury (Amec, 2018).
- Blasting caps:
  - Suspected explosives classified as Resource Conservation and Recovery Act (RCRA) D003 Reactive Waste (Amec, 2018).
  - Chemicals associated with the blasting caps are unknown, but may include mercury fulminate, potassium chlorate, tetryl, lead azide, lead styphnate, and pentaerythritol tetranitrate.
  - Fuses are wrapped in tar; the chemical makeup of the tar is unknown but may be associated with SVOCs.
- Garbage dumps:
  - Reportedly contained a variety of debris including building materials, empty drums, gas cans, lead and zinc batteries, historical equipment associated with Site operations.
  - Piles suspected to have asbestos containing material (ACM) and lead- and arsenic-based paint associated with building debris.
  - Oily water mixtures observed among the debris.
- Building materials (historical paint, asbestos):
  - Lead-based paint has been applied to several buildings in the Mill Town.
  - Chipped paint samples have contained lead concentrations between 50 and 525,000 mg/kg, with lead concentrations being highest in white paint samples and lowest in red paint samples.
  - Paint chips from building materials processed using toxicity characteristic leaching procedure (TCLP) analysis have exceeded the lead RCRA regulatory level for waste disposal. Arsenic has been detected in paint samples as well, up to 17,000 mg/kg.
  - Surface soil samples in the vicinity of Mill Town buildings have elevated concentrations of lead and arsenic, likely as a result of peeling paint (Amec, 2018).
  - Chrysotile was located in pipe insulation, fiberboard, and around boiler tanks.
- Fuel/oil:
  - Reported releases of Bunker C, lubricating oil, residual fuel oil, PCBs, and petroleum hydrocarbons.
- Boiler ash:
  - RCRA hazardous waste; exceeded the RCRA TCLP limit for lead.
  - Historical samples of the ash also reported contained barium, cadmium, chromium, lead, and silver (Amec, 2018).

Considering the results of the Phase I and II RI summarized in **Section 2.3**, the COPCs/contaminants of potential ecological concern (COPECs) for the Site and their suspected sources are as follows:

- Metals:
  - Aluminum, arsenic, barium, cadmium, chromium, cobalt, copper, iron, lead, manganese, mercury, nickel, selenium, silver, vanadium, and zinc were detected at concentrations exceeding PALs in surface soil, surface water, and/or sediment in 2021 and/or 2023.
  - Suspected sources include mill tailings/ore & ore concentrates, blasting caps, garbage dumps, building materials (historical paint), and boiler ash.
- SVOCs:
  - Anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluoranthene, indeno(1,2,3-cd)pyrene, phenanthrene, pyrene and naphthalene were detected at concentrations exceeding PALs in surface soils, surface water, and/or sediment in 2021 and/or 2023.
  - Suspected source is incidental fuel/oil releases.
- VOCs:
  - Toluene[9] and chlorobenzene were detected at concentrations above their respective PALs in sediment in 2023 and 2021, respectively.
  - Benzene was detected in one surface soil sample at the Oil House at a concentration greater than PALs in 2021.
  - Suspected source is fuel/oil releases.
- Explosives:
  - Not detected in surface soil in the Blasting Cap Disposal Areas in 2021.
  - Additional information is needed to assess subsurface soil for explosives.
  - Suspected source is blasting caps.

3.2 LOCATIONS OF PROBABLE SOURCE AREAS

Known source areas associated with the Mill Town portion of the Site are shown on **Figure 7**. Phases I and II of the RI have largely focused on the extent of COPCs in Site surface soils and the extent of COPC impacts to National Creek. The nature and extent of contamination, as evaluated using information collected to date, is discussed by medium in **Section 3.5** below.

- Mill tailings/ore & ore concentrates:
  - Six areas previously reported to have extensive piles of mill tailings and ore concentrates.
  - Two long piles run parallel to one another north of the Mill Building; the northernmost pile is suspected to extend into private land adjacent to the Mill Building.
  - Two piles identified between the Mill Building and the Hospital.
  - Two largest piles are located on the western side of the Site; the northernmost pile is suspected to surround the Leaching Plant, while also extending downslope and south towards National Creek.
  - National Creek flows through tailings.
  - Phase II RI confirmed that tailings are present along the banks of National Creek extending from the Mill Town to the terminus of National Creek. Visible tailings deposits were observed to extend to McCarthy.

[9] Toluene was detected at a concentration exceeding the PAL at one midstream sample in 2023; it was not detected in the parent sample, but was also detected in the equipment blank.

- Blasting caps:
  - Blasting caps and fuses identified in two separate debris scatter areas; scatter areas are heterogenous, and the extent of caps has not been fully delineated.
  - Eastern area is on the steep slope, north of the Mill Building.
  - Western area is on the western side of the north Sacking Shed and Floatation Building.
- Garbage dumps:
  - Large areas predominantly on the western side of the Site, downslope of the main roads in the moraine fields
  - Waste piles are located along a steep slope, remain covered with debris, and are difficult to access (Amec, 2018).
- Building materials (historical paint, asbestos):
  - Associated with historical buildings and their immediate vicinity.
  - A 1992 report estimated 70,000 square feet of building surface area consisted of badly deteriorated paint.
  - Reportedly found in garbage dumps.
  - ACM was identified and quantified in 23 buildings and utilidors (utility corridors) in the Mill Town during a 1991 survey. In 1993 and 1994, asbestos was abated from most buildings and accessible utilidors in the Mill Town. ACM is reportedly still found in crawl spaces and in foundations and walls by NPS workers during rehabilitation of Mill Town buildings (Amec, 2018).
- Fuel/oil:
  - Heavy fuel oil west of the Power Plant near three aboveground storage tanks (ASTs):
    - Reported to ADEC (case number 912412211) and the National Response Center (case number 83218) in 1991 by the Kennecott Corporation and EMCON (EMCON, 1992).
    - Surface staining and pooling of Bunker C oil reported in an area behind the Power Plant.
    - Total area of surface staining estimated to be 79,000 square feet and observed to extend approximately 125 feet downslope of the fuel tanks.
    - Depth of contamination reportedly from two inches to two feet.
    - Estimated 1,000 cubic yards of contaminated soil was present; remedial action to stabilize the pools of Bunker C completed in 1994.
    - Buried piping and utilidors that connect the ASTs reported to contain residual fuel oil(ADEC, n.d.a).
  - Heavy fuel oil on the north side of the Mill Building
    - 50 square yard area of stained soil reported by EMCON in 1992.
    - Lubricating oil from Mill Building operations reported as source of staining (EMCON, 1992).
    - A 1995 letter from ADEC indicated that no further action was needed for this spill area (GeoEngineers, 2006).
    - If, in the future, the buildings are moved or demolished, impacted soils may need to be addressed (ADEC, n.d.b).
  - Used-oil disposal pit located next to a generator at Building 24.
    - Used-oil disposal pit reportedly located adjacent to the Kennecott Wilderness Guides (Building 24).
    - Exact location of this impacted area not identified in historical reports and therefore not shown on **Figure 7**.
  - Monroe Tank 4(1) and utilidor between the Power Plant and the AST
    - AST previously emptied and cleaned.
    - High levels of petroleum contamination were noted in 1992.
    - Status of the site is listed as “active” and ADEC site report indicates cleanup may be needed (ADEC, n.d.c).
- Boiler ash:

- An estimated volume of 1,443 cubic feet (49,000 pounds) of boiler ash located southwest of the Power Plant; was capped with concrete in 1994.

3.3 MECHANISMS AND TIMING OF HISTORICAL AND SUSPECTED RELEASES

A description of each of the waste types and a summary of the mechanisms and timing of suspected releases is provided below.

- Mill tailings/ore & ore concentrates:
  - Generated during Mill Town operations in the early 20th century.
  - Direct disposal is the primary release mechanism.
- Blasting caps:
  - Disposed of during Mill Town operations in the early 20th century.
  - Direct disposal is the primary release mechanism.
- Garbage dumps:
  - Materials disposed in garbage dumps during Mill Town operations in the early 20th century.
  - Lead acid batteries and possibly ACM were removed from these areas prior to NPS's acquisition of the Site.
  - Direct disposal is the primary release mechanism.
- Building materials (historical paint, asbestos):
  - Lead-based paint applied to several buildings in the Mill Town in the early 20th century during Mill Town operations. Painted surfaces have weathered/deteriorated with time and have resulted in chipped and peeling paint.
  - ACM used in building materials in the Mill Town in the early 20th century during Mill Town operations. In 1993 and 1994, asbestos was abated from most buildings and accessible utilidors in the Mill Town.
  - Direct disposal and weathering are the primary release mechanisms.
- Fuel/oil:
  - Multiple releases associated with bulk fuel/oil storage/conveyance features identified prior to NPS acquisition of the Site but anticipated to have occurred coincident with Mill Town operations in the early 20th century.
- Boiler ash:
  - Likely generated during Mill Town operations in the early 20th century.
  - Capped with concrete in 1994.
  - Direct disposal and weathering/cap deterioration are the primary release mechanisms.

3.4 AFFECTED ENVIRONMENTAL MEDIA

Previous investigations and historical records of on-Site activity have detected COPCs in environmental media including soil, surface water, and sediment. There has not been an in-depth analysis of the groundwater at the Site; however, the surface water infiltration observed in the moraine field suggests groundwater may be a media of potential concern.

3.5 CONTAMINANT DISTRIBUTION

3.5.1 Surface Soil

Phase I and II RI data (XRF and laboratory analytical data) indicate elevated metals concentrations in surface soil (0 to 6 inches bgs) are widespread in the vicinity of the Mill Town. The concentrations of the most prevalent metals (copper, lead, and arsenic) are greater in the operational areas of the Mill Town, in the tailings piles, and along Mill Town roads; concentrations of these constituents generally decrease with distance from these features. The approximate extent of elevated metals in Site surface soils relative to field background concentrations has largely been identified (see **Figure 7**). Shading of the approximate area where metals have been detected at concentrations greater than field background concentrations in **Figure 7** reflects which data were used to inform the evaluation and the level of certainty. Solid shading represents areas where analytical and/or XRF results are available to inform the evaluation. Cross hatching represents areas where insufficient analytical and/or XRF results are available and where patterns of results collected at other comparable areas of the Site have been used to infer where metals concentrations greater than field background concentrations are likely present.

The approximate extent of elevated metals relative to field background has not been identified in the area south of the Site in the glacial moraine; it is evident from field observations and collected data that National Creek has transported and intermixed tailings and moraine material at the Site. Ground surface XRF measurements collected within the moraine field as far south as McCarthy indicated metals were detected at up to 10 times the preliminary field background concentrations in this area.

PAHs were detected in surface soil at the Oil House, the Transformer House, and the Blasting Cap Disposal Areas at relatively low concentrations but exceeding PALs during the Phase I RI. The highest measured concentrations of PAHs during the Phase I RI were detected beneath the Transformer House. PAHs were detected in a subset of the additional surface soil samples collected within the Mill Town during the Phase II RI at low level concentrations that exceed PALs. Naphthalene and benzo(a)pyrene were the most prevalent PAHs that exceeded PALs. The results of the Phase I and II RI suggest the distribution of low levels of PAHs is relatively broad and interspersed throughout the developed portions of the Site. Outside of known/mitigated release areas (such as the stabilized fuel oil release and capped ash pile) and former operational areas associated with more significant petroleum usage (e.g., Transformer House, Oil House), the distribution of these constituents is generally consistent with conditions associated with incidental spills/releases.

Low level VOC PAL exceedances were measured in surface soil at the Oil House during the Phase I RI. PCBs were not detected in surface soil samples collected from the Transformer House during the Phase I RI, suggesting PCB oil was not likely used and/or PCBs have not been released at this location. Blasting caps were not encountered in surface soil in the east or west Blasting Cap Disposal Areas during the Phase I RI and explosives were not detected in surface soil samples collected in the Blasting Cap Disposal Areas. Based on the Phase I findings, surface soil samples were not analyzed for VOCs, PCBs, or explosives as part of the Phase II RI.

3.5.2 Surface Water

Metals were detected at concentrations that exceed PALs in surface water samples collected during the Phase I and II RI in upstream, midstream, and downstream locations. Elevated concentrations of certain metals were detected in downstream surface water samples compared to midstream and upstream surface water samples during the Phase I and Phase II RI. These results indicate the Site may be impacting surface water quality in downstream National Creek. Downstream concentrations of select metals were elevated in surface water samples after a rain event during the Phase I RI, suggesting surface runoff may be transporting COPCs from Mill Town surface soil to downstream surface water.

During the Phase II RI, PAHs were detected in surface water samples at concentrations exceeding PALs (ranging from marginal exceedances to 150 times PALs). Samples with concentrations of PAHs exceeding PALs were collected from upstream, midstream, and downstream locations. Clear trends in concentrations of these constituents in surface water are not yet apparent. Other SVOCs were not detected in surface water samples at concentrations exceeding PALs during the Phase I or II RI. PCBs were not detected in surface water samples collected during the Phase I or II RI. VOCs were not detected at concentrations exceeding PALs during the Phase II RI. VOCs were not analyzed during the Phase I RI due to a laboratory supply issue.

3.5.3 Sediment

Metals were detected at concentrations that exceed PALs in sediment samples collected during the Phase I and II RI in upstream, midstream, and downstream locations. Elevated concentrations of several metals were detected in downstream sediment samples compared to midstream and upstream sediment samples. These results indicate the Site may be impacting sediment quality in downstream National Creek.

During the two phases of the RI, a limited number of PAHs and VOCs (several of which are known to be common laboratory contaminants) were detected at concentrations exceeding PALs in a subset of upstream, midstream, and downstream samples. These results are not indicative of significant or widespread presence of these constituents in sediment. PCBs have not been detected in sediment samples collected from National Creek.

3.5.4 Air

Based on historical data and the results of the Phase I and II RI, COPCs, primarily metals, are present in surface soils and tailings across the Mill Town portion of the Site. COPCs in surface soil have the potential to become airborne due to natural wind events or human activities. Air samples collected and analyzed for metals and asbestos during the Phase I RI did not contain these COPCs at concentrations greater than PALs. Additionally, an exposure assessment conducted in September 2019 indicated that employee occupational exposures to target airborne contaminants (metals, asbestos, respirable crystalline silica, dust, VOCs, and PAHs) are below the applicable occupational exposure limits for the work tasks evaluated (Occupational Safety and Health Administration Permissible Exposure Limits, National Institute of Occupational Safety and Health Recommended Exposure Limits, American Conference of

Governmental Industrial Hygienists Threshold Limit Values). Although there are no established exposure limits applicable to the general public for the contaminants evaluated, results from a "simulated" visitor airborne contaminant exposure scenario were also well below occupational exposure limits and thus not expected to present an airborne hazard to short duration child or adult visitors (W&C, 2020). Air sampling during both the Phase I RI and 2019 Exposure Assessment was completed in the months of August and September when weather conditions were wet from frequent rain. Park personnel have reported drier conditions that could represent high airborne dust conditions during earlier summer months (e.g., July).

As an alternative method of assessment, a sensitivity analysis was performed of one of the key model input parameters – the PEF recommended by ADEC (ADEC, 2018). The results of this analysis indicate that the ADEC PEF is a conservative representation of typical Site conditions under ambient wind conditions and additional data collection to develop a Site-specific PEF is not required to support future evaluations of the inhalation exposure pathway using the standard CERCLA risk assessment approach. Air sampling was not conducted during the Phase II RI.

3.5.5 Other Media

Subsurface soil sampling was not conducted during the Phase I or II RI. Subsurface soil is likely to contain elevated metals concentrations in the tailings piles areas, as the tailings piles themselves are many feet in depth. Additional information is needed to evaluate the nature and extent of contamination in subsurface soil.

Groundwater has not been sampled to date. Concentrations of metal COPCs in soil exceed Alaska's migration to groundwater cleanup levels for arsenic, cadmium, copper, silver and thallium, suggesting groundwater is a medium of potential concern. Based upon the presence of natural seeps observed in 2021, it appears that the depth to groundwater is shallow in some areas and could be impacted by soil contamination at the Site. Two known groundwater wells are present in the Mill Town. Two Class B wells (transient, non-community water system) were drilled for the Kennicott Glacier Lodge on lot 26 in 2001 (well logs 31843 and 31844). These wells were drilled to 140 feet bgs and 105 feet bgs, respectively (Alaska Department of Natural Resources, 2016a; Alaska Department of Natural Resources, 2016b).

3.6 POTENTIAL MIGRATION PATHWAYS

COPCs may be released to surface and subsurface soil by direct disposal, weathering, deterioration, and spills/accidental releases. COPCs in surface soil may be suspended by wind or other mechanical processes (traffic, construction, etc.) and redeposited onto surface soil and indoors on surfaces across the Site, spreading contamination away from source areas. Rainfall erosion of contaminated surface soils may transport COPCs to downgradient surface soil locations and/or National Creek, particularly in areas of the Mill Town with extreme slopes.

National Creek flows through mill tailings and impacted soils in the Kennecott Mill Town and continues to flow toward the moraine field to the west and south of the Site. Surface water flow at the Site is dynamic, at times creating new primary flow channels and infiltrating into groundwater within the

moraine field. Runoff may transport COPCs from soil near National Creek to surface water and sediment in National Creek. Concentrations of metals in surface water were elevated in samples collected immediately after a rain event during the Phase I RI, indicating that runoff may be an active migration pathway for COPCs, carrying suspended particles and dissolved COPCs downstream to the west and south of the Site. COPCs may be transported between surface water and sediment through dissolution and adsorption.

Infiltration and percolation of rainfall may dissolve COPCs in soil and transport them vertically to underlying groundwater. COPCs in groundwater may be transported further downgradient through advection and dispersion. Sorption of COPCs to soil (notably metals, PAHs) may retard COPC movement through groundwater; whereas acidic conditions or reduced environments may result in increased mobility of metal COPCs. Diffusion may allow for groundwater flowing through contaminated soils to transport contaminants from areas of high concentration to areas of low concentration resulting in a more widespread distribution of contaminants across the Site.

Groundwater beneath the Site discharges at the ground surface in seep locations, where it becomes surface water; several seeps were observed at the Site during the Phase I RI. These groundwater seeps may discharge COPCs to the ground surface and/or National Creek. Limited information is available regarding groundwater flow conditions at the Site; however, regional groundwater flow direction is inferred to flow toward the southwest, following the surface topography of the Site. Receptors downgradient and at a lower elevation than the Site may be exposed to contaminated groundwater, if present.

Potential contaminant migration pathways associated with human and ecological receptors are illustrated on **Figures 8 and 9**, respectively.

3.7 POTENTIAL RECEPTORS

Potential human receptors identified at the Site are as follows:

- Site visitors
- Site workers
- Maintenance/construction worker
- Residents
- Subsistence users

As shown in **Figure 8**, human receptors could be exposed to contaminants through: inhalation of dust/volatiles in ambient air and subsurface soils; ingestion of fugitive dust, surface soil, subsurface soil, surface water, and sediment; and direct contact with fugitive dust (e.g., dust that becomes airborne from disturbances such as foot traffic or wind), surface soil, subsurface soil, groundwater (via contact with groundwater that emanates at the ground surface at seep locations where it becomes surface water), surface water, and sediment. Human receptors may also be exposed to contaminants through ingestion of impacted aquatic biota (fish), terrestrial biota (meat), and plants. National Creek is used as a drinking water source within the Mill Town. Groundwater remains a potential exposure medium with potential

ingestion and direct contact exposure routes. Two Class B wells (transient, non-community) were drilled at lot 26 for the Kennicott Glacier Lodge in 2001; whether these wells are currently in use is unknown.

Potential ecological receptors for this Site are divided into the following categories:

- Plants
- Invertebrates
- Amphibians
- Fish
- Birds and mammals

As shown in **Figure 9**, ecological receptors could become exposed to contaminants through: inhalation of dust/volatiles in ambient air; ingestion of fugitive dust, surface soil, subsurface soil, groundwater (as surface water via seeps), surface water, and sediment; and direct contact with/uptake from fugitive dust, surface soil, subsurface soil, groundwater (as surface water via seeps), surface water, and sediment. Ecological receptors also may be exposed to contaminants through bioaccumulation within the food chain. No known threatened or endangered species are present on the Site or in off-Site areas where hazardous substances from the Site could have migrated. Migratory birds (lesser yellowlegs, olive-sided flycatcher) may be present at the Site.

3.8 PRELIMINARY EXPOSURE MODEL

Preliminary human and ecological exposure models are presented as **Figures 8 and 9**, respectively. Exposure models will continue to be refined based on data obtained throughout the investigation. Along with the results from screening-level risk assessments, these models will form the basis for detailed risk assessments to be conducted during a future phase of the RI. NPS will consider the concentrations of COPCs and COPECs in the context of various Site-specific exposure scenarios. For humans, this will include the frequency, duration, and intensity of potential exposure. For example, a visitor to the Site for a few days in their lifetime will have a lower potential exposure than a Site worker or a full-time resident. For ecological receptors, the risk assessment takes into consideration what receptors are present at the Site and considers what concentration levels may lead to ecological health effects.

3.9 DATA GAP ASSESSMENT

Based on the results of the Phase I and II RI, the following data gaps have been identified for the Site:

- Mill Town roads and aerial tramways served as the transportation routes for ore minerals and concentrate. Results of the Phase II RI indicate Mill Town roads and aerial tramways are associated with elevated metal COPCs. The approximate extent of elevated COPCs in surface soil have not been fully evaluated along all roads and aerial tramways, but Phase II RI results indicate COPC concentrations decrease within a short distance from Mill Town roads.
- The horizontal extent of COPCs in surface soil in the area south of the Site in the glacial moraine has not been fully evaluated; it is evident from field observations and collected data that National Creek has transported and intermixed tailings and moraine material at the Site.

- The farthest upstream surface water and sediment sample location appears to be located within the extent of metals impacts observed in surface soil as part of the Phase II RI. Soil quality in this area may be contributing to the concentrations of COPCs detected in upstream samples – as a result the data obtained from this sample location may not be representative of actual conditions upstream of the Site.
- Given the dynamic conditions associated with National Creek, the nature and downstream extent of metals, VOCs, and SVOCs in surface water and sediment (and as well as surface soil in the vicinity of the creek) may vary temporally. The current temporal data set is limited to two field seasons.
- The nature and extent of COPCs in subsurface soil is unknown. Data collected to date indicate elevated concentrations of COPCs in surface soils; based on what is known about the Site CSM, it is possible these contaminants are leaching into the subsurface soils. In certain areas of the Site, (e.g., tailings piles), COPCs are expected to be elevated in subsurface soils due to direct disposal of tailings. Subsurface soil data is needed to evaluate the vertical extent of impacts and inform future risk assessment.
- The nature and extent of COPCs in groundwater is unknown. Groundwater is believed to be relatively shallow based on information provided in historical investigations (Amec, 2018), and evidence of seeps and springs was observed in the moraine area west of the Mill Town during the Phase I RI. COPCs may be leaching from Site surface soils into groundwater at the Site.
- The nature of COPCs present in the environment in areas unimpacted by historical mining and milling operations is unknown. Metal COPCs can be naturally occurring in the environment.

4 DATA QUALITY OBJECTIVES

The DQO process specifies anticipated project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. DQOs were established by the DQO Planning Team following the seven steps listed below, which are consistent with the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006).

1. State the problem (**Section 4.1**)
2. Identify the goal of the investigation (**Section 4.2**)
3. Identify the information inputs (**Section 4.3**)
4. Define the boundaries of the investigation (**Section 4.4**)
5. Develop the analytic approach (**Section 4.5**)
6. Specify performance or acceptance criteria (**Section 4.6**)
7. Develop the plan for obtaining data (**Section 5**)

The DQO Planning Team for this project is shown on **Text Table 1**.

Text Table 1: DQO Planning Team

Name	Project Role	Technical Specialty
Sarah Venator	NPS Contaminated Site Project Manager	Geologist, Phase I and II RI NPS Project Manager
Rene Nahlik	Cleanup Lead/VHB Project Manager at the time of DQO planning	Environmental Engineer, Phase I RI Technical Advisor, Phase II RI Cleanup Lead
Todd Hall	DQO Team Member	Engineer/Hydrogeologist, Phase II RI Technical Advisor
Brie Hicknell	DQO Team Member	Environmental Engineer, Phase I and II RI Field Team Leader
Lynn Woodbury	DQO Team Member	Risk Assessment & DQO Development, Technical Advisor for Phase I and II RI

4.1 PROBLEM IDENTIFICATION

Several historical investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations, along with the Phase I and Phase II RI completed in August 2021 and July 2023, respectively, identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include:

- Metals (paint, blasting caps, tailings)
- SVOCs, including PAHs (petroleum, creosote, other operations)
- VOCs (petroleum, creosote, other operations)[1]
- Explosives (blasting caps)[1]
- Asbestos[1]

The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors.

Similar to previous phases of the RI, the Phase III RI will be subdivided into several DQOs:

- DQO 2/3 Surface Soil (continued from Phase I and II RI)
- DQO 6 Surface Water/Sediment (continued from Phase I and II RI)
- DQO 7 Groundwater
- DQO 8 Subsurface Soil
- DQO 9 Background Study

Details of the DQOs required to achieve these objectives are provided in **Text Tables 2 through 6** (embedded after Section 4).

4.2 INVESTIGATION GOALS

The goal of the Site-wide RI is to provide sufficient data to characterize the nature and extent of contamination in environmental media and determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. As noted previously, a multiple phase RI approach is necessary to achieve the goal of the Site-wide RI. The focus of the Phase III RI will be addressing several of the data gaps noted for the Kennecott Mill Town in Section 3.9.

Due to the significant scope associated with addressing the data gaps and the short season during which field work can be conducted at the Site, the priorities for the Phase III RI are as follows:

- Estimate exposure point concentrations for human and ecological receptors potentially exposed to COPCs in surface soil at residential properties within the Mill Town.
- Add to the temporal data set used to characterize COPCs in surface water and sediment.
- Identify additional groundwater seeps, springs, or stormwater conveyances that could act as additional COPCs sources to National Creek.

[1] These COPCs were not detected or detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI.

- Estimate exposure point concentrations for human and ecological receptors potentially exposed to COPCs in surface water and sediment in National Creek.
- Assess the nature and extent of COPCs in groundwater in the vicinity of the Mill Town.
- Assess groundwater flow direction.
- Assess the nature and extent of COPCs in subsurface soil.
- Assess the concentrations of COPCs naturally present in media from similar geomorphologic environments as the Mill Town that are unimpacted by historical mining and Mill Town operations.

Consistent with Step 2 of the DQO process, a detailed listing of the decision/estimation[1] PIQs, potential outcomes, and the key decision/estimation statements are provided in **Text Tables 2 through 6**.

4.3 INFORMATION INPUTS

The purpose of this step is to identify the data required to answer the PIQs and to determine which inputs require environmental measurements. A detailed listing of information required to answer the PIQs and the key sources of this information are provided in Section 3 of **Text Tables 2 through 6** - Identify Information Inputs. The data required to answer the PIQs are detailed in the "Sampling and Analytical Requirements" Section of **Text Tables 2 through 6**.

4.4 BOUNDARIES OF THE INVESTIGATION

The objective of this step is to identify the decision units (DUs) and sampling units (SUs) to define the spatial and temporal elements of the investigation area. The focus of the Phase III RI is limited to the Kennecott Mill Town and the areas where the background study will be conducted; see Section 5 for location specifics. The details of the spatial and temporal boundaries of each PIQ are defined in Section 4 of **Text Tables 2 through 6** – Define the Boundaries of the Investigation.

4.5 ANALYTIC APPROACH

The population parameters that will be applied to the sample populations to answer the PIQs and the screening values these parameters will be compared to are defined in Section 5 of **Text Tables 2 through 6** – Develop the Analytic Approach. Detection limits are based on human health and ecological risk-based screening levels. DQO-specific screening values, decision rules, and specification of the estimators applicable to each DQO are detailed in **Text Tables 2 through 6**.

[1] Two types of PIQs are specified in the DQOs – decision questions and estimation questions. Decision questions make a choice between two alternate conditions, whereas estimation questions evaluate the magnitude of an environmental parameter or characteristic for future use. In the DQO tables, decision PQIs are denoted with a "D" prefix and estimation PQIs are denoted with an "E" prefix.

4.6 PERFORMANCE OR ACCEPTANCE CRITERIA

The purpose of this step is to establish the criteria needed to evaluate whether the data will be of sufficient quality to answer the PIQs with confidence.

4.6.1 Quality Assurance/Quality Control and Data Validation and Usability

Project Quality Assurance (QA)/QC measures are detailed in attached **Table 2** (Sample Types and Total Number of Containers), **Table 4** (Standard Operating Procedures [SOPs]), **Table 5** (Sample Containers, Preservation, and Hold Times), **Tables 6a, 6b, 6c, and 6d** (Screening Levels and Laboratory-Specific Detection/Reporting Limits), and **Table 7** (Laboratory Quality Control and Data Validation Criteria). Project-specific field QA/QC procedures, including decontamination procedures; instrument inspection, calibration, testing, and maintenance; and inspection of supplies and consumables are detailed in the FSP (see **Section 5**). Field and laboratory audits are discussed in **Section 7** (Assessment and Oversight).

Data usability will be assessed mainly using data quality indicators (DQIs), which will be represented by the following and are presented for each analytical method in attached **Table 7**.

- **Precision**: field precision will be assessed using field duplicates for discrete samples and field replicates for ISM samples; laboratory precision will be assessed using laboratory control samples and duplicates (LCS/LCSD); and precision in the sample matrix will be assessed using matrix spike (MS) and matrix spike duplicates (MSD).
- **Accuracy**: field accuracy will be assessed using equipment blanks; laboratory accuracy will be assessed using method blanks, calibration criteria, and LCS recoveries; and accuracy in the sample matrix will be assessed using the MS recovery results.
- **Representativeness**: representativeness will be assessed quantitatively through the relative percent difference for duplicates, through relative standard deviation for ISM triplicates, and qualitatively for all data by verifying that sampling procedures and locations were followed as designed, validating the data received for COPCs, and checking that DQOs were met.
- **Completeness**: completeness will be assessed by comparing the number of samples that were not rejected to the planned number of samples; if fewer than 95 percent of sample results are usable, the program should be reevaluated to determine if additional samples are required.
- **Comparability**: comparability will be assessed by evaluating whether DQOs were achieved and SOPs were followed.
- **Sensitivity**: field sensitivity will be assessed using the field equipment blanks; laboratory sensitivity will be assessed using the laboratory method blanks, LCS, and calibration criteria; and sensitivity in the sample matrix will be assessed based on sample-specific method detection limit (MDL) and quantitation limit (QL) achieved for COPCs. MDLs and QLs are compared to PALs in **Table 6**. For a select number of analytes and media, MDLs and QLs may be greater than PALs. MDLs and QLs vary by laboratory; given the remote nature of the Site and the short hold times associated with select analyses, sending samples to laboratories with lower MDLs and QLs is not a practical option. Analytes that have not been detected and that have MDLs and QLs greater than PALs will be discussed in the risk assessment as part of the uncertainty assessment.

The following items will be reviewed by the VHB Project Manager to verify data:

- Generated by the field team: field notes, chain of custody (COC) records, and daily progress reports.
- Generated by the Data Validator: data validation report and validated data tables.

The following items will be reviewed by the Contract Laboratory Project Manager or their designated representative:

- Data generated by the laboratory (e.g., equipment calibration, testing, maintenance, and inspection logs).

The following items will be reviewed by the Data Validator, Laboratory Project Manager, and Contractor Project Manager to verify data:

- Generated by the field team: COC upon sample receipt.
- Generated by the laboratory: sample check-in confirmation report and laboratory reports (including raw data and reported results for samples, standards, QC checks, and QC samples).

The Data Validator will spot check results provided in the laboratory report against results provided in the EDD for one analyte per laboratory method per medium. The Data Validator will prepare a report documenting completion of the steps outlined in attached **Tables 9 and 10**.

4.6.2 Decision Error Limits and Uncertainty Evaluation

The project decision makers will use analytical results to decide whether additional investigations may be needed. The tolerable limits on decision errors used to establish performance goals for the data collection design are typically specified in this step. Upon the receipt of sample analytical results, decision PIQs will be addressed using the "Statistical Hypothesis Testing" method (if applicable), as outlined in *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006).

The probability of a decision error is controlled by adopting a scientific approach in which the data are used to select between the null hypothesis [H_0] (e.g., the mean Site concentration of a particular analyte is significantly higher than the mean background/reference concentration) and the alternative hypothesis [H_A] (e.g., the mean Site concentration of a certain analyte is less than or equal to the mean background/reference concentration). The null hypothesis is presumed to be true (not rejected) in the absence of evidence to the contrary. A false rejection, or "Type I" decision error, refers to the type of error made when the null hypothesis is rejected when it is actually true (e.g., concluding that the mean Site concentration for a particular analyte is naturally occurring when it is actually elevated due to a release). A false acceptance, or "Type II" decision error, refers to the type of error made when the null hypothesis is accepted when it is actually false (e.g., concluding that the mean Site concentration of a particular analyte is higher than the background/reference when it is actually not significantly different). Null and alternative hypotheses and acceptable Type I and Type II errors for each of the decision PIQs are provided in **Text Tables 2 through 6**.

Text Table 2: DQO 2/3 Surface Soil Data Quality Objectives

DQO: DQO Step:	Surface Soil
1 – State the Problem	
Problem Description	Several investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include: • Metals (paint, blasting caps, tailings) • SVOCs, including PAHs (petroleum, creosote, other operations) • VOCs (petroleum, creosote, other operations)* • Explosives (blasting caps)* • Asbestos* *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI. The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. Surface soil: • Phase I and II RI data indicate elevated concentrations of metals in surface soil (0 to 6 inches bgs) are widespread in the vicinity of the Mill Town. • Concentrations of the most prevalent metals (copper, lead, and arsenic) are greater in the operational areas of the Mill Town, in the tailings piles, and along Mill Town roads. • The approximate extent of elevated metals relative to field background has not been identified in the area south of the Site in the glacial moraine, along aerial tramways, along road/transportation corridors, and in the vicinity of the mines. • Phase I and II RI results suggest the distribution of low levels of PAHs is relatively broad and interspersed throughout the developed portions of the Site. Low level exceedances of VOC PALs were measured in surface soil at the Oil House during the Phase I RI.
Planning Team Members	NPS is the lead regulatory agency for the CERCLA activities at the Site. NPS contractors providing technical support for SI planning include VHB and CDM Smith. Members of the DQO Planning Team and their respective roles are listed in **Text Table 1**.
Resources, Constraints, Schedule	Available resources for this project include the planning team entities listed above, as well as the NPS and VHB subcontractors that will be employed and equipment that will be used to perform the activities required for this investigation. NPS is currently targeting spring and summer 2024 to complete the Phase III RI field activities. One of the primary constraints on completing the work is the relatively short field season, which is generally between the Memorial Day and Labor Day holidays; power and other Site services are shutdown outside of this period. Other

Text Table 2: DQO 2/3 Surface Soil Data Quality Objectives

DQO: DQO Step:	Surface Soil
	key considerations include: the remote location (significant mobilization time/distance; extremely limited availability of goods/services, including medical services and sample shipping options); the presence of private property interspersed within Federal land; ongoing renovation/rehabilitation efforts; presence of managed ruins/debris fields; steep topography; and historical/archeological significance of various Site features.
2 – Identify Goals of the Investigation	
PIQs	E.2.1: What concentrations of COPCs in surface soil are a reasonable estimate of exposure point concentrations for human and ecological receptors at residential properties within the Mill Town?
Alternative Outcomes	E.2.1: not applicable
Decision/Estimation Statements	E.2.1: Estimate the concentrations of COPCs in surface soil that represent the reasonable average exposure to human and ecological receptors at residential properties within the Mill Town.
3 – Identify Information Inputs	
Information Required to Answer PIQs	E.2.1: • Average COPC (metals, PAHs) concentrations in surface soil at the Silk Stocking Cottages located on federal land (see **Figure 2**) • In Vitro Bioaccessibility Assay (IVBA) analyses of surface soil from Silk Stocking Cottages to assess lead and arsenic bioavailability for consideration during risk assessment • Georeferenced estimation unit (EU) boundaries
Sources of Information	• Measured surface soil concentrations for samples collected from the Silk Stocking Cottages located on federal land • Measured IVBA in soil • Soil classification SOP • ADEC Field Sampling Guidance • ADEC Guidance for Evaluating Metals at Contaminated Sites • Interstate Technology Regulatory Council (ITRC) ISM Technical/Regulatory Guidance • USEPA Superfund Lead-Contaminated Residential Sites Handbook • USEPA Guidance for Sample Collection for IVBA for Arsenic and Lead in Soil and Applications of Relative Bioavailability Data in Human Health Risk Assessment
Sampling and Analytical Requirements	E.2.1: • NPS prefers the use of ISM when characterizing soil EUs, since this sampling methodology provides a robust estimate of the mean and reduces the influence of within-EU heterogeneity. However, because ISM samples are typically not split in the field, the collection of separate non-ISM (discrete or composite) samples may be necessary to accommodate when specialized laboratory analyses are necessary (i.e., two different analytical laboratories are performing analyses for the same EU). • Surface soil ISM samples should be collected from various EUs within the Silk Stocking Cottages parcels in triplicate at the following depths:

Text Table 2: DQO 2/3 Surface Soil Data Quality Objectives

DQO: DQO Step:	Surface Soil
	– 0 to 1 inches within dripline areas and yard areas[1] for TAL metals analysis (USEPA methods 6020B, 1631E) and PAH analysis (USEPA method 8270-selected ion monitoring [SIM]). A subset of the dripline and yard area EUs should be sampled for IVBA analysis (USEPA method 1340). – 0 to 12 inches in areas where gardening occurs for TAL metals (USEPA methods 6020B, 1631E) and PAHs (USEPA method 8270-SIM) analyses – Samples collected for metals analysis should be sieved in the field using No. 4 or No. 10 sieve screens and sieved by the laboratory using a 150-micron (µm) sieve prior to analysis; samples collected for PAH analysis should not be sieved – Samples collected for IVBA analysis should be sieved in the field using No. 4 or No. 10 sieve screens and sieved by the laboratory using a 150 µm sieve prior to analysis
4 - Define the Boundaries of the Investigation	
Spatial Bounds	E.2.1: • Areal/horizontal bounds: EUs should be located within the Silk Stocking Cottages parcels located on federal land within cleared areas (excludes forested portions of the parcels) • Vertical bounds: soil samples shall be collected between 0 and 12 inches bgs
Temporal Bounds	E.2.1: Concentrations of COPCs in surface soil are unlikely to vary seasonally; therefore, samples may be collected at any time during the summer season (May to September), during which the Mill Town is most easily accessed.
Scale of Inference/Estimation (DU/SU)	E.2.1: • EUs: sized according to the use/geometry of the yard areas. EUs should be established around Silk Stocking Cottages between the sides of the structures and the dripline of the roof, within the remaining yard area surrounding the cottages, within the garden established in the parcel across from the cottages, and the cleared area surrounding the garden. EUs are between 0.004 and 0.1 acres in size
5 – Develop the Analytic Approach	
Statistical Parameter of Interest	E.2.1: • The mean COPC concentration of samples collected within each EU, as a reasonable estimate for the “true mean” exposure point concentration
Investigation-specific Decision Thresholds (Screening Levels)	E.2.1: not applicable
Decision Rule	E.2.1: not applicable
Specification of the Estimators	E.2.1: This investigation will estimate reasonable COPC exposure point concentrations that represent human and ecological receptor exposure to surface soil at the Silk Stocking Cottages located on federal land, for use in risk assessment
6 – Specify Performance/Acceptance Criteria	

[1] Consistent with USEPA’s *Superfund Residential Lead Sites Handbook* (USEPA, 2024), samples from dripline areas should be kept separate from yard areas since dripline samples have the potential for higher contamination from outdoor painted surfaces.

Text Table 2: DQO 2/3 Surface Soil Data Quality Objectives

DQO: DQO Step:	Surface Soil
Investigation QA/QC	• Adherence to Field SOPs (provided in the QAPP tables and Appendix A), Site-specific methods as defined in Project FSP, and field equipment manufacturer's instructions as appropriate • Collection of field and laboratory QC samples as prescribed in the SAP • Verification of laboratory certifications • Assessment of DQIs and their associated measurement performance criteria for precision, accuracy, representativeness, comparability, completeness, and sensitivity metrics (provided in the Project QAPP) • Implementation of data verification and validation
Statistical Hypothesis/Tolerable Limits for Decision Errors	E.2.1: not applicable
Acceptable Limits on Estimation Uncertainty	E.2.1: Uncertainty in the estimation of average exposure point concentrations will be accounted for by calculating the upper confidence limit (UCL) on the mean COPC concentration of the triplicate samples collected from each EU. 95% UCL on the mean is selected based on EPA guidance (i.e., to limit the potential that the true mean is higher than the 95% UCL to less than 5%). A sufficient number of samples should be collected to ensure the 95% UCL is no more than 50% higher than the arithmetic mean estimate.
7 – Develop Plan for Obtaining Data	
	See **Section 5** of the SAP.

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
1 – State the Problem	
Problem Description	Several investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include: • Metals (paint, blasting caps, tailings) • SVOCs, including PAHs (petroleum, creosote, other operations) • VOCs (petroleum, creosote, other operations)* • Explosives (blasting caps)* • Asbestos* *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI. *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the Phase I RI; however, additional testing may be conducted in future phases of the RI. The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. Surface water/sediment: • Metals were detected at concentrations that exceed PALs in surface water and sediment samples collected during the Phase I and II RI in upstream, midstream, and downstream locations. • Elevated concentrations of certain metals were detected in downstream surface water and sediment samples compared to midstream and upstream surface water samples. • Downstream concentrations of select metals were elevated in surface water samples after a rain event during the Phase I RI; surface runoff may transport COPCs from Mill Town surface soil to surface water. • During the Phase II RI, PAHs were detected in surface water samples at concentrations exceeding PALs in upstream, midstream, and downstream locations. Clear concentration trends for these constituents in surface water are not yet apparent. A limited number of SVOCs and VOCs (several of which are known to be common laboratory contaminants) were detected at concentrations exceeding PALs in a subset of upstream, midstream, and downstream sediment samples. Results are not indicative of significant or widespread presence of these constituents in sediment.
Planning Team Members	NPS is the lead regulatory agency for the CERCLA activities at the Site. NPS contractors providing technical support for SI planning include VHB and CDM

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
	Smith. Members of the DQO Planning Team and their respective roles are listed in **Text Table 1**.
Resources, Constraints, Schedule	Available resources for this project include the planning team entities listed above, as well as the NPS and VHB subcontractors that will be employed and equipment that will be used to perform the activities required for this investigation. NPS is currently targeting spring and summer 2024 to complete the Phase III RI field activities. One of the primary constraints on completing the work is the relatively short field season, which is generally between the Memorial Day and Labor Day holidays; power and other Site services are shutdown outside of this period. Other key considerations include: the remote location (significant mobilization time/distance; extremely limited availability of goods/services, including medical services and sample shipping options); the presence of private property interspersed within Federal land; ongoing renovation/rehabilitation efforts; presence of managed ruins/debris fields; steep topography; and historical/archeological significance of various Site features.
2 – Identify Goals of the Investigation	
PIQs	D.6.1: Do the concentrations of COPCs in National Creek surface water/sediment exceed PALs? E.6.1: Where are locations (in the vicinity of the Mill Town) of groundwater seeps, springs, or storm water run-off conveyances that could act as COPC sources to National Creek? E.6.2: What concentrations of COPCs in National Creek surface water/sediment are reasonable estimates of exposure point concentrations for human and ecological receptors?
Alternative Outcomes	D.6.1: • Eliminate COPCs from further consideration; or • Plan and conduct additional investigation, as needed, to obtain sufficient data to conduct a risk assessment E.6.1: not applicable E.6.2: not applicable
Decision/Estimation Statements	D.6.1: Determine if COPC concentrations in surface water/sediment in National Creek are greater than the PALs, which would indicate that evaluation of potential risks to human and/or ecological receptors is needed; or if COPC concentrations are less than the PALs, which would result in re-evaluation of data in regard to temporal and physical considerations to confirm its representativeness and identify data gaps (if any) that need to be filled prior to determining that data are representative of actual COPC concentrations E.6.1: Potential exists that groundwater seeps, springs, or storm water run-off conveyances may be sources of contamination to National Creek. If COPC concentrations in National Creek surface water/sediment are greater than PALs, locating these areas will support future RI efforts and/or CSM development E.6.2: Estimate the concentrations of COPCs in National Creek surface water/sediment that represent exposures to human and ecological receptors
3 – Identify Information Inputs	

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
Information Required to Answer PIQs	D.6.1: • COPC (metals, PAHs, VOCs) concentrations in surface water/sediment at multiple locations in the vicinity and downstream of the Mill Town • water quality indicator parameters (e.g., temperature, oxidation reduction potential [ORP], specific conductivity, etc.), hardness, and copper biotic ligand model input parameters (temperature, pH, dissolved organic carbon [DOC], major cations [calcium, magnesium, sodium, potassium], major anions [sulfate, chlorine], alkalinity, sulfide) in surface water • total organic carbon (TOC) in sediment • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) • remote sensing imagery (from Park) • regional meteorological data and Site-specific stream flow data E.6.1: georeferenced locations for and photographs of new seep, springs, or storm water run-off conveyance locations E.6.2: COPC (metals, PAHs, VOCs) concentrations in surface water/sediment at multiple locations in the vicinity and downstream of the Mill Town
Sources of Information	• Measured surface water and sediment concentrations for samples collected from National Creek • USEPA Regional Screening Levels (RSLs) (Target Risk [TR]=1E-06; Target Hazard Quotient [THQ]=0.1) for tapwater and resident soil • Maximum Contaminant Levels (MCLs) • National Recommended Water Quality Criteria (NRWQC) • NPS Screening Level Ecological Risk Assessment (SLERA) COPEC Selection Ecological Screening Values (ESV) • State surface water protection standards • Aerial photographs and topographic maps • Remote Automatic Weather Stations data • United States Geological Survey (USGS) surface water station data (USGS Station 15209770 National Creek near McCarthy, Alaska)
Sampling and Analytical Requirements	D.6.1 and E.6.2: • Co-located samples of surface water and sediment should be collected from National Creek at multiple locations and during various stream stage/flow conditions • Surface water samples should be field-filtered for dissolved metals and DOC analysis. Unfiltered samples should be collected for total metals, PAHs, VOCs, hardness, sulfate, chloride, alkalinity, and sulfide analyses • Samples should be collected from downstream to upstream locations. Water samples should be collected before sediment samples to limit turbidity in the water column. Samples should be collected in areas of fine-grained sediment deposition to target appropriate grain size distribution for analysis and high-end COPC concentrations (COPCs tend to sorb to finer-grained sediments) • Surface water and sediment samples should be collected for analysis of TAL metals (USEPA methods 6020B, 200.8, 1631E), PAHs (USEPA method 8270-SIM), VOCs (USEPA method 8260D), TOC/DOC (USEPA method 9060A modified), hardness (calculations), sulfate & chloride (USEPA method 300.0), alkalinity (SM 2320B), and sulfide (SM 4500S D) at an environmental laboratory

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
	• Field measurements (e.g., temperature, pH, ORP, specific conductivity, etc.) should be collected coincident with samples for laboratory analysis E.6.1: To support identifying locations of groundwater seep discharges, springs, or storm water run-off conveyances in the vicinity of the Mill Town and National Creek, aerial photographs and topographic maps, combined with on-Site visual examinations, should be used to search for likely locations of natural seeps and/or springs, and surface water run-off tributaries that feed National Creek
4 - Define the Boundaries of the Investigation	
Spatial Bounds	D.6.1 and E.6.2: • Surface water and sediment sampling should be conducted in the portion of National Creek in the vicinity and downstream of the Mill Town • Areal/horizontal bounds: – Approximate upstream, midstream, and downstream locations sampled during the Phase II RI. If possible, sampling locations should be co-located with previously sampled locations to allow comparison of results – Vertical bounds: Sediment samples should be collected from the top 0-6 inches of sediment (considered representative of most recent sediment deposited in stream bed); multiple sample events will allow qualitative evaluation of changes in sediment quality over time. Water samples should be collected from the middle of the water column (or the top 0-2 inches if the water depth is less than 1-foot). These water depths will support sampling representative water flowing at the time of sampling while limiting the potential for collecting sediment in the water samples E.6.1: • Areal/horizontal bounds: National Creek should be assessed from 300 feet upstream of the former National Creek dam to 3500 feet downstream of the leaching plant, and to a distance of at least 15 feet beyond the banks of National Creek • Vertical bounds: not applicable
Temporal Bounds	D.6.1, E.6.2: COPC concentrations in surface water are expected to vary at different river stage/flow conditions. Sediment concentrations are considered less likely to have temporal variation than surface water E.6.1: Observe/identify groundwater seep discharges, springs, or storm water run-off conveyances when conditions are wet (during spring melt and/or following precipitation events)
Scale of Inference/Estimation (DU/SU)	D.6.1 • SUs: The SUs for COPC concentrations will include the 15 SUs for the Phase I/II RIs (three upstream, three midstream, and nine downstream samples) • DUs: National Creek surface water and sediment from a point 300 feet upstream of the former National Dam to a point 3500 feet downstream of the leaching plant E.6.1: • SUs: not applicable. • EUs: same as D.6.1 DUs E.6.2: • SUs: same as D.6.1 SUs

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
	• EUs: same as D.6.1 DUs
5 – Develop the Analytic Approach	
Statistical Parameter of Interest	D.6.1: • The population mean; the maximum sample COPC concentration in the DU shall be a conservative estimate for the "true mean" exposure point concentration E.6.1: • Elevation, latitude, and longitude (X,Y,Z) of each groundwater seep, spring, and/or storm water run-off conveyance location E.6.2: • The mean and maximum COPC concentrations in EU, as reasonable estimates for the applicable exposure point concentrations (depending on exposure media and receptor)
Investigation-specific Decision Thresholds (Screening Levels)	D.6.1: • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) E.6.1: not applicable E.6.2: not applicable
Decision Rule	D.6.1: • If the maximum concentration is greater than the PAL, then evaluate data gaps and assess the DU further relative to this COPC • If the maximum concentration is not greater than the PAL, then no further assessment is required relative to this COPC
Specification of the Estimators	E.6.1: Specify the elevation, latitude, and longitude (X, Y, Z) of each groundwater seep, spring, and/or storm water run-off conveyance location E.6.2: This investigation will estimate the National Creek surface water/sediment concentrations that represent human and ecological receptor exposures to COPCs, for use in risk assessment
6 – Specify Performance/Acceptance Criteria	
Investigation QA/QC	• Adherence to Field SOPs (provided in the QAPP tables and Appendix A), Site-specific methods as defined in Project FSP, and field equipment manufacturer's instructions as appropriate • Collection of field and laboratory QC samples as prescribed in the SAP • Verification of laboratory certifications • Assessment of DQIs and their associated measurement performance criteria for precision, accuracy, representativeness, comparability, completeness, and sensitivity metrics (provided in the Project QAPP) • Implementation of data verification and validation
Statistical Hypothesis/Tolerable Limits for Decision Errors	D.6.1: A "yes/no" decision will be made by comparing the COPC concentrations to the PALs, without considering the variability associated with the analytical results. As described in USEPA's DQO Guidance (USEPA 2001), in this situation, no formal statistical hypothesis tests are performed; therefore, it is not necessary to specify tolerable limits on making a wrong decision E.6.1: not applicable E.6.2: not applicable

Text Table 3: DQO 6 Surface Water/Sediment Data Quality Objectives

DQO: DQO Step:	Surface Water/Sediment
Acceptable Limits on Estimation Uncertainty	E.6.1: The acceptable limit of estimation of uncertainty of latitude, longitude, and elevation estimates should be within +/- 15 feet E.6.2: Uncertainty in the estimation of average exposure concentrations will be accounted for by calculating the UCL on the mean concentration of EUs. 95% UCL on the mean is selected based on USEPA guidance (i.e., to limit the potential that the true mean is higher than the 95% UCL to less than 5%). A sufficient number of samples should be collected to ensure the 95% UCL is no more than 50% higher than the arithmetic mean estimate
7 – Develop Plan for Obtaining Data	
	See **Section 5** of the SAP.

Text Table 4: DQO 7 Groundwater Data Quality Objectives

DQO: DQO Step:	Groundwater
1 – State the Problem	
Problem Description	Several investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include: • Metals (paint, blasting caps, tailings) • SVOCs, including PAHs (petroleum, creosote, other operations) • VOCs (petroleum, creosote, other operations)* • Explosives (blasting caps)* • Asbestos* *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI. The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. Groundwater: • Groundwater has not been sampled to date. Concentrations of metal COPCs in soil exceed ADEC's migration to groundwater cleanup levels for arsenic, cadmium, copper, silver and thallium, suggesting groundwater is a medium of potential concern. • Based upon the presence of natural seeps observed in 2021, it appears that the depth to groundwater is shallow in some areas and could be impacted by soil contamination at the Site. • Two Class B wells (transient, non-community water system) were drilled at lot 26 for the Kennicott Glacier Lodge in 2001.
Planning Team Members	NPS is the lead regulatory agency for the CERCLA activities at the Site. NPS contractors providing technical support for SI planning include VHB and CDM Smith. Members of the DQO Planning Team and their respective roles are listed in **Text Table 1**.
Resources, Constraints, Schedule	Available resources for this project include the planning team entities listed above, as well as the NPS and VHB subcontractors that will be employed and equipment that will be used to perform the activities required for this investigation. NPS is currently targeting spring and summer 2024 to complete the Phase III RI field activities. One of the primary constraints on completing the work is the relatively short field season, which is generally between the Memorial Day and Labor Day holidays; power and other Site services are shutdown outside of this period. Other key considerations include: the remote location (significant mobilization time/distance; extremely limited availability of goods/services, including medical services and sample shipping options); the presence of private property

Text Table 4: DQO 7 Groundwater Data Quality Objectives

DQO: DQO Step:	Groundwater
	interspersed within Federal land; ongoing renovation/rehabilitation efforts; presence of managed ruins/debris fields; steep topography; and historical/archeological significance of various Site features. Sampling for the Phase III RI is restricted to public land.
2 – Identify Goals of the Investigation	
PIQs	D.7.1: Do concentrations of COPCs in groundwater in the vicinity of the Mill Town exceed PALs? E.7.1: What is the horizontal and vertical extent of groundwater with concentrations of COPCs greater than PALs? E.7.2: What is the groundwater flow direction in the vicinity of the Mill Town?
Alternative Outcomes	D.7.1: • Eliminate COPCs from further consideration; or • Plan and conduct additional investigation, as needed, to obtain sufficient data to conduct a risk assessment E.7.1: not applicable E.7.2: not applicable
Decision/Estimation Statements	D.7.1: Determine if COPC concentrations in groundwater in the vicinity of the Mill Town are greater than the PALs, which would indicate that evaluation of potential risks to human and/or ecological receptors is needed; or if COPC concentrations are less than the PALs, which would result in re-evaluation of data in regard to temporal and physical considerations to confirm its representativeness and identify data gaps (if any) that need to be filled prior to determining that data are representative of actual COPC concentrations E.7.1: Estimate the horizontal and vertical extent of groundwater with COPC concentrations that exceed PALs E.7.2: Estimate the groundwater flow direction in the vicinity of the Mill Town
3 – Identify Information Inputs	
Information Required to Answer PIQs	D.7.1 and E.7.1: • COPC (metals, SVOCs, VOCs) concentrations in groundwater samples collected from groundwater monitoring wells installed in the vicinity of the Mill Town • Water quality indicator parameters (e.g., temperature, ORP, specific conductivity, etc.), hardness, and copper biotic ligand model input parameters (temperature, pH, DOC, major cations [calcium, magnesium, sodium, potassium], major anions [sulfate, chlorine], alkalinity, sulfide) in groundwater • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) • Surveyed monitoring well locations • Depth to groundwater measurements E.7.2: • Surveyed monitoring well locations • Depth to groundwater measurements
Sources of Information	• Measured groundwater concentrations for samples collected from monitoring wells installed in the vicinity of the Mill Town

Text Table 4: DQO 7 Groundwater Data Quality Objectives

DQO: DQO Step:	Groundwater
	• USEPA RSLs (TR=1E-06; THQ=0.1) for tapwater • Maximum Contaminant Levels (MCLs) • NRWQC • Vapor Intrusion Screening Levels (VISLs) • NPS SLERA COPEC Selection ESVs • State surface water protection standards
Sampling and Analytical Requirements	D.7.1 and E.7.1: • Groundwater samples should be collected from multiple locations in the vicinity of the Mill Town using low flow sampling techniques • Groundwater samples for DOC analysis should be field-filtered; samples for all other analyses should not require field-filtering (given the use of low-flow sampling techniques) • Groundwater samples should be collected for analysis of TAL metals (USEPA methods 200.8, 1631E), SVOCs (USEPA method 8270 and 8270-SIM), VOCs (USEPA method 8260D), DOC (USEPA method 9060A modified), hardness (calculations), sulfate & chloride (USEPA method 300.0), alkalinity (SM 2320B), and sulfide (SM 4500S D) at an environmental laboratory • Field measurements (e.g., temperature, pH, ORP, specific conductivity, etc.) should be collected prior to groundwater sample collection E.7.2: not applicable
4 - Define the Boundaries of the Investigation	
Spatial Bounds	D.7.1, E.7.1 and E.7.2: • Areal/horizontal bounds: groundwater monitoring wells should be installed within the Millsite subdivision unit in locations where groundwater has the highest potential for Site-related contamination, and/or the highest likelihood for use as drinking water, and/or in areas that will provide good spatial distribution of results • Vertical bounds: groundwater samples shall be collected at the first occurrence of groundwater (i.e., the water table); the depth to the water table is unknown but expected to be relatively shallow
Temporal Bounds	D.7.1, E.7.1, E.7.2: COPC concentrations in groundwater and groundwater flow direction may vary seasonally. Several sampling events should be conducted in multiple months and years to capture seasonal variability in groundwater COPC concentrations and groundwater flow direction
Scale of Inference/Estimation (DU/SU)	D.7.1: • SUs/DUs: individual samples collected from each monitoring well location E.7.1: • SUs: same as D.7.1 • EU: Millsite subdivision unit E.7.2: • SUs: depth to water readings collected from each monitoring well location • EU: Millsite subdivision unit
5 – Develop the Analytic Approach	
Statistical Parameter of Interest	D.7.1: • The population mean; the maximum sample COPC concentration in the DU, shall be a conservative estimate for the “true mean” exposure point concentration

Text Table 4: DQO 7 Groundwater Data Quality Objectives

DQO: DQO Step:	Groundwater
	E.7.1: • Elevation, latitude and longitude (X,Y,Z) of each groundwater monitoring well E.7.2: • Depth to water for each groundwater monitoring well
Investigation-specific Decision Thresholds (Screening Levels)	D.7.1: • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) E.7.1: not applicable E.7.2: not applicable
Decision Rule	D.7.1: • If the maximum concentration is greater than the PAL, then evaluate data gaps and further assess the DU relative to this COPC • If the maximum concentration is not greater than the PAL, then no further assessment is required relative to this COPC
Specification of the Estimators	E.7.1: This investigation will estimate the vertical and horizontal extent of COPC concentrations in groundwater that exceed PALs. Each SU concentration will be compared to PALs and if the SU concentration is greater than PALs, then this area will be considered impacted and included in the vertical and horizontal extent of the impacted area for the Site E.7.2: This investigation will estimate the groundwater flow direction. Depth to water measurements will be compared to infer groundwater flow direction
6 – Specify Performance/Acceptance Criteria	
Investigation QA/QC	• Adherence to Field SOPs (provided in the QAPP tables and Appendix A), Site-specific methods as defined in Project FSP, and field equipment manufacturer's instructions as appropriate • Collection of field and laboratory QC samples as prescribed in the SAP • Verification of laboratory certifications • Assessment of DQIs and their associated measurement performance criteria for precision, accuracy, representativeness, comparability, completeness, and sensitivity metrics (provided in the Project QAPP) • Implementation of data verification and validation
Statistical Hypothesis/Tolerable Limits for Decision Errors	D.7.1: A "yes/no" decision will be made by comparing the COPC concentrations to the PALs, without considering the variability associated with the analytical results. As described in USEPA's DQO Guidance (USEPA 2001), in this situation, no formal statistical hypothesis tests are performed; therefore, it is not necessary to specify tolerable limits on making a wrong decision E.7.1: not applicable E.7.2: not applicable
Acceptable Limits on Estimation Uncertainty	E.7.1: The acceptable limit of estimation uncertainty for areal/horizontal delineation of groundwater with concentrations of COPCs greater than PALs is 3 feet. The acceptable limit of estimation uncertainty for vertical delineation of groundwater with COPC concentrations greater than PALs is 0.1 feet

Text Table 4: DQO 7 Groundwater Data Quality Objectives

DQO: DQO Step:	Groundwater
	E.7.2: The acceptable limit of estimation uncertainty for horizontal location accuracy of monitoring wells is 2 feet. The acceptable limit of estimation uncertainty for monitoring well reference point elevations and water level measurements in 0.01 feet
7 – Develop Plan for Obtaining Data	
	See **Section 5** of the SAP.

Text Table 5: DQO 8 Subsurface Soil Data Quality Objectives

DQO: DQO Step:	Subsurface Soil
1 – State the Problem	
Problem Description	Several investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include: • Metals (paint, blasting caps, tailings) • SVOCs, including PAHs (petroleum, creosote, other operations) • VOCs (petroleum, creosote, other operations)* • Explosives (blasting caps)* • Asbestos* *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI. The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. Subsurface soil: • Subsurface soil sampling was not conducted during the Phase I or II RI. • Subsurface soil is likely to contain elevated concentrations of metals in the tailings piles areas, as the tailings piles themselves are many feet in depth. • Direct disposal (on ground surface) of other wastes has been documented at the Site as well (garbage, blasting caps, etc.); contaminants associated with these wastes may be transported to the subsurface through subsequent ground disturbance (redevelopment and biological activity) and/or infiltration of precipitation/runoff. • Additional information is needed to evaluate the nature and extent of contamination in subsurface soil.
Planning Team Members	NPS is the lead regulatory agency for the CERCLA activities at the Site. NPS contractors providing technical support for SI planning include VHB and CDM Smith. Members of the DQO Planning Team and their respective roles are listed in **Text Table 1**.
Resources, Constraints, Schedule	Available resources for this project include the planning team entities listed above, as well as the NPS and VHB subcontractors that will be employed and equipment that will be used to perform the activities required for this investigation. NPS is currently targeting spring and summer 2024 to complete the Phase III RI field activities. One of the primary constraints on completing the work is the relatively short field season, which is generally between the Memorial Day and Labor Day holidays; power and other Site services are shutdown outside of this period. Other key considerations include: the remote location (significant mobilization time/distance; extremely limited availability of goods/services, including medical

Text Table 5: DQO 8 Subsurface Soil Data Quality Objectives

DQO: DQO Step:	Subsurface Soil
	services and sample shipping options); the presence of private property interspersed within Federal land; ongoing renovation/rehabilitation efforts; presence of managed ruins/debris fields; steep topography; and historical/archeological significance of various Site features.
2 – Identify Goals of the Investigation	
PIQs	D.8.1: Do concentrations of COPCs in subsurface soil in the vicinity of the Mill Town exceed PALs? E.8.1: What is the horizontal and vertical extent of subsurface soil with COPCs concentrations that exceed PALs?
Alternative Outcomes	D.8.1: • Eliminate COPCs from further consideration; or • Plan and conduct additional investigation, as needed, to obtain sufficient data to conduct a risk assessment E.8.1: not applicable
Decision/Estimation Statements	D.8.1: Determine if COPC concentrations in subsurface soil in the vicinity of the Mill Town are greater than the PALs, which would indicate that evaluation of potential risks to human and/or ecological receptors is needed; or if COPC concentrations are less than the PALs, which would result in re-evaluation of data in regard to temporal and physical considerations to confirm its representativeness and identify data gaps (if any) that need to be filled prior to determining that data are representative of actual COPC concentrations E.8.1: Estimate the horizontal and vertical extent of subsurface soil with COPC concentrations that exceed PALs
3 – Identify Information Inputs	
Information Required to Answer PIQs	D.8.1 and E.8.1: • COPC (metals, SVOCs, VOCs) concentrations in subsurface soil samples collected from various depths during installation of groundwater monitoring wells installed in the vicinity of the Mill Town • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) • Georeferenced monitoring well locations
Sources of Information	• Measured subsurface soil concentrations for samples collected from various depths during installation of groundwater monitoring wells installed in the vicinity of the Mill Town • Soil classification SOP • ADEC Field Sampling Guidance • ADEC Guidance for Evaluating Metals at Contaminated Sites • USEPA RSLs (TR=1E-06; THQ=0.1) for resident soil • NPS SLERA COPEC Selection ESVs • State cleanup standards (human health and migration to groundwater)
Sampling and Analytical Requirements	D.8.1 and E.8.1: • Subsurface soil samples should be collected from multiple depths in soil borings at multiple locations in the vicinity of the Mill Town • Soil samples should be collected for analysis of TAL metals (USEPA methods 6020B, 1631E), SVOCs (USEPA method 8270 and 8270-SIM), and VOCs (USEPA method 8260D) at an environmental laboratory
4 - Define the Boundaries of the Investigation	

Text Table 5: DQO 8 Subsurface Soil Data Quality Objectives

DQO: DQO Step:	Subsurface Soil
Spatial Bounds	D.8.1 and E.8.1: • Given the difficulty of collecting subsurface soil samples at this Site and to maximize resources, subsurface soil samples should be collected from soil borings advanced as part of the DQO 7 investigation. Areal/horizontal bounds same as D.7.1 and E.7.1. • Vertical bounds: subsurface soil samples shall be collected from depths greater than 6 inches bgs to the top of bedrock/refusal
Temporal Bounds	D.8.1 and E.8.1: Concentrations of COPCs in subsurface soil are unlikely to vary seasonally; therefore, samples may be collected at any time during the summer season (May to September), during which the Mill Town is most easily accessed, but should be collected during installation of groundwater monitoring wells
Scale of Inference/Estimation (DU/SU)	D.8.1: • SUs/DUs: individual samples collected from each boring location E.8.1: • SUs: same as D.8.1 • EU: Millsite subdivision unit
5 – Develop the Analytic Approach	
Statistical Parameter of Interest	D.8.1: • The population mean; the maximum sample COPC concentration in the DU, shall be a conservative estimate for the “true mean” exposure point concentration E.8.1: • Elevation, latitude, and longitude (X,Y,Z) of each subsurface soil sample
Investigation-specific Decision Thresholds (Screening Levels)	D.8.1: • PALs (i.e., lowest of the screening levels listed in *Sources of Information*) E.8.1: not applicable
Decision Rule	D.8.1: • If the maximum concentration is greater than the PAL, then evaluate data gaps and assess the DU further relative to this COPC • If the maximum concentration is not greater than the PAL, then no further assessment is required relative to this COPC
Specification of the Estimators	E.8.1: This investigation will estimate the vertical and horizontal extent of subsurface soil concentrations that exceed PALs. Each SU concentration will be compared to PALs and if the SU concentration is greater than PALs, then this area will be considered impacted and included in the vertical and horizontal extent of the impacted area for the Site
6 – Specify Performance/Acceptance Criteria	
Investigation QA/QC	• Adherence to Field SOPs (provided in the QAPP tables and Appendix A), Site-specific methods as defined in Project FSP, and field equipment manufacturer’s instructions as appropriate • Collection of field and laboratory QC samples as prescribed in the SAP • Verification of laboratory certifications • Assessment of DQIs and their associated measurement performance criteria for precision, accuracy, representativeness, comparability, completeness, and sensitivity metrics (provided in the Project QAPP) • Implementation of data verification and validation

Text Table 5: DQO 8 Subsurface Soil Data Quality Objectives

DQO: DQO Step:	Subsurface Soil
Statistical Hypothesis/Tolerable Limits for Decision Errors	D.8.1: A "yes/no" decision will be made by comparing the COPC concentrations to the PALs, without considering the variability associated with the analytical results. As described in USEPA's DQO Guidance (USEPA 2001), in this situation, no formal statistical hypothesis tests are performed; therefore, it is not necessary to specify tolerable limits on making a wrong decision E.8.1: not applicable
Acceptable Limits on Estimation Uncertainty	E.8.1: The acceptable limit of estimation uncertainty for areal/horizontal delineation of subsurface soil with COPC concentrations greater than PALs is 3 feet. The acceptable limit of estimation uncertainty for vertical delineation of subsurface soil with COPC concentrations greater than PALs is 2 feet
7 – Develop Plan for Obtaining Data	
	See **Section 5** of the SAP.

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
1 – State the Problem	
Problem Description	Several investigations were conducted by the NPS within the Mill Town area of the Kennecott Mines and Mill Town Site (the Site) to assess environmental contamination and Site hazards. These investigations identified potential and actual sources and releases of contaminants to air (fugitive dust), soil, surface water, sediment, and groundwater/seeps. Based upon prior Site investigations and historical information/operations, COPCs include: • Metals (paint, blasting caps, tailings) • SVOCs, including PAHs (petroleum, creosote, other operations) • VOCs (petroleum, creosote, other operations)* • Explosives (blasting caps)* • Asbestos* *These COPCs were not detected or were detected at concentrations less than PALs in samples collected during the completed phases of the RI; however, additional testing may be conducted in future phases of the RI. The CSM identifies transport/exposure pathways with the potential to expose human and ecological receptors to contaminated media. However, available data are insufficient to characterize the nature and extent of contamination in all Site media and whether contaminants are present above concentrations that pose an unacceptable risk to human and ecological receptors. Therefore, measured concentrations of COPCs in Site media are needed to characterize the nature and extent of contamination, and ultimately to determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. Background study: • Arsenic, chromium and many other metals are naturally occurring throughout Alaska (ADEC, 2018). The bedrock formations responsible for the copper sulfide mineral deposits mined on Bonanza Ridge are associated with naturally elevated levels of various metals. • PAHs may be present in the environment because of non-site-related ubiquitous anthropogenic impacts. • A background study is required to estimate the concentrations of COPCs (metals, PAHs) that are naturally present in environmental media (soil, surface water, sediment, and groundwater) at the Site to determine the extent of site-related contamination. • Background concentrations of COPCs in environmental media can be used to provide a frame of reference for interpreting risks and identifying which COPCs are site-related. • Background concentrations of COPCs in environmental media will help inform the selection of remedial goals.
Planning Team Members	NPS is the lead regulatory agency for the CERCLA activities at the Site. NPS contractors providing technical support for SI planning include VHB and CDM Smith. Members of the DQO Planning Team and their respective roles are listed in **Text Table 1**.
Resources, Constraints, Schedule	Available resources for this project include the planning team entities listed above, as well as the NPS and VHB subcontractors that will be employed and equipment that will be used to perform the activities required for this investigation. NPS is currently targeting spring and summer 2024 to complete the Phase III RI field

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
	activities. One of the primary constraints on completing the work is the relatively short field season, which is generally between the Memorial Day and Labor Day holidays; power and other Site services are shutdown outside of this period. Other key considerations include: the remote location (significant mobilization time/distance; extremely limited availability of goods/services, including medical services and sample shipping options); the presence of private property interspersed within Federal land; ongoing renovation/rehabilitation efforts; presence of managed ruins/debris fields; steep topography; and historical/archeological significance of various Site features.
2 – Identify Goals of the Investigation	
PIQs	D.9.1: Do background soil COPC concentrations differ between the different geomorphologic environments present at the Site? D.9.2: Are Site exposure area concentrations of COPCs in environmental media (soil, surface water, sediment, groundwater) higher than background? E.9.1: What are high-end background threshold concentrations for each COPC that can be used to delineate the site-related contamination extent in environmental media?
Alternative Outcomes	D.9.1: • If there are differences in COPC concentrations between different geomorphological environments, then Site and background datasets for each geomorphologic environment will need to be kept separate and statistics calculated separately for each type. If not, then it is not necessary to distinguish between geomorphologic environments when making comparisons to background. D.9.2: • If Site COPC concentrations within an exposure area are higher than background, then the COPC will be identified as a Site-related contaminant. If not, then the COPC can be excluded as a Site-related contaminant. E.9.1: not applicable
Decision/Estimation Statements	D.9.1: Determine if COPC concentrations for different geomorphologic environments are statistically different from each other, which would indicate that Site and background datasets for each geomorphologic environment will need to be kept separate and statistics calculated separately for each type. D.9.2: Determine if Site exposure area COPC concentrations are statistically higher than background, which would indicate that the COPC is a Site-related contaminant. E.9.1: Estimate a high-end threshold value from the background COPC concentration distribution that can be used to make comparisons to Site samples to establish the Site-related contamination extent in environmental media.
3 – Identify Information Inputs	
Information Required to Answer PIQs	D.9.1: • Concentration distribution for each COPC in surface and subsurface soil samples within background study areas for each geomorphological environment

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
	• Geomorphologic information for Mill Town and background areas (e.g., soil type, bedrock classification, thickness of soil horizon, particle size distribution, well screening interval, etc.) D.9.2: • Average COPC concentrations in surface soil, subsurface soil, surface water, sediment, and groundwater collected from background study areas • Average COPC concentrations in surface soil, subsurface soil, surface water, sediment, and groundwater collected from Site exposure areas for human and ecological receptors • Water quality indicator parameters (e.g., temperature, ORP, specific conductivity, etc.) and hardness for groundwater and surface water samples • Copper biotic ligand model input parameters (temperature, pH, DOC, major cations [calcium, magnesium, sodium, potassium], major anions [sulfate, chlorine], alkalinity, sulfide) for surface water samples E.9.1: Background concentration distribution for each COPC in surface soil, subsurface soil, surface water, sediment, and groundwater
Sources of Information	• Measured surface soil, subsurface soil, surface water, sediment, and groundwater COPC concentrations in samples collected from background study areas • Measured surface soil, subsurface soil, surface water, sediment, and groundwater COPC concentrations in samples collected from site exposure areas (collected in DQOs 2/3, 6, 7, and 8) • Soil descriptions for Mill Town and background areas • USGS Geologic Map of Alaska • USFS Alaska Soil Survey • ADEC Guidance for Evaluating Metals at Contaminated Sites • ITRC Soil Background and Risk Assessment guidance document • Dataset visualization tools (e.g., scatter plots, quartile box plots, etc.)
Sampling and Analytical Requirements	D.9.1: • Surface and subsurface soil samples should be collected from background study locations areas representative of each geomorphological environment for analysis of TAL metals (USEPA methods 6020B, 1631E), PAHs (USEPA method 8270-SIM) at an environmental laboratory • Sampling design needs to characterize the full distribution of concentrations across each background area (i.e., incremental, composite sampling methods cannot be employed) – discrete samples should be collected D.9.2: • Surface and subsurface soil samples should be collected from site and background exposure areas for analysis of TAL metals (USEPA methods 6020B, 1631E) and PAHs (USEPA method 8270-SIM) at an environmental laboratory • Surface water and sediment samples should be collected from background (upstream) study locations for analysis of TAL metals (USEPA methods 6020B, 200.8, 1631E), PAHs (USEPA method 8270-SIM), and TOC (sediment only; USEPA method 9060A modified) at an environmental laboratory. DOC (USEPA method 9060A modified), hardness (calculations), sulfate & chloride (USEPA method 300.0), alkalinity (SM 2320B), and sulfide (SM 4500S D) should also be analyzed for surface water samples

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
	• Surface water samples to be field-filtered for dissolved metals analysis. Unfiltered samples to be collected for total metals and PAHs analyses • Groundwater samples should be collected from wells within the same aquifer but upgradient of the site for analysis of TAL metals (USEPA methods 200.8, 1631E) and PAHs (USEPA method 8270-SIM) at an environmental laboratory – see Text Table 4 for additional sampling and analytical requirements for groundwater samples • Field measurements (e.g., temperature, pH, ORP, specific conductivity, etc.) should be collected coincident with surface water and groundwater samples for laboratory analysis • Sampling design needs to provide an estimate of the overall average concentration across each site and background exposure area (i.e., incremental, composite sampling methods can be ideal for this purpose); the same sampling design should be conducted in both the site exposure areas and background study areas – discrete and ISM samples should be collected to mirror samples collected at the Site E.9.1: • Samples of surface soil, subsurface soil, surface water, sediment, and groundwater should be collected from background study areas for analysis of TAL metals and PAHs (see above for methods) at an environmental laboratory • Sampling design needs to characterize the full distribution of concentrations across each background area (i.e., incremental, composite sampling methods cannot be employed) to allow for a determination of the high-end of the concentration distribution
4 - Define the Boundaries of the Investigation	
Spatial Bounds	General: • Background study areas should be representative of locations with similar environments as the Mill Town that are unimpacted by historical mining and Mill Town operations • Background study areas should encompass the full range of COPC concentration distribution variability • Background soil ISM DUs should be 0.25 acres in size. Discrete soil samples should be collected from within the boundaries of the ISM DUs • Vertical bounds: Surface and subsurface soil samples should be collected from the same depth intervals at both background and site locations; surface water samples should be collected from the middle of the water column (or the top 0-2 inches if the water depth is less than 1-foot); sediment samples should be collected from the top 0-6 inches of sediment; groundwater samples should be collected at the water table (the depth to the water table is unknown but expected to be relatively shallow) D.9.1: • Background samples will be collected from locations with similar geomorphologic environments as the Mill Town that are unimpacted by historical mining and Mill Town operations; soil background locations will not be collected from within 150 feet of a rural road or 300 feet of a building or structure (consistent with USGS background study methodology); background areas for different geomorphologic environments should be sized similarly

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
Temporal Bounds	General: Concentrations of COPCs in surface and subsurface soil, and sediment are unlikely to vary seasonally; therefore, samples may be collected at any time during the summer season (May to September), during which the Mill Town and vicinity is most easily accessed. COPC concentrations in surface water and groundwater may vary seasonally. Several sampling events should be conducted in multiple months and years to capture seasonal variability in surface water and groundwater COPC concentrations. Background sampling efforts should be conducted simultaneous with Site sampling efforts.
Scale of Inference/Estimation (DU/SU)	D.9.1: • DUs: the size of the background DUs for each geomorphologic environment should encompass the full range of background COPC concentrations; background areas for different geomorphologic environments should be sized similarly • SUs: should be sized to capture the concentration variability within each DU D.9.2: • DUs: The site DU should be equal to the receptor-specific exposure area; the site and background exposure areas (DUs) should be sized similarly • SUs: because the goal is to estimate the DU-wide average, the SU can be set equal to the DU E.9.1: • DUs: the size of the background DUs for each environmental medium should encompass the full range of background COPC concentrations • SUs: should be sized to capture the concentration variability within each DU; systematic or random sampling designs should be employed
5 – Develop the Analytic Approach	
Statistical Parameter of Interest	D.9.1: • Mean/median COPC concentrations for each geomorphologic background area DU D.9.2: • Mean COPC concentrations across the entire exposure area DU (for both site and background exposure areas) E.9.1: • 95th percentile of the background concentration distribution for each environmental medium
Investigation-specific Decision Thresholds (Screening Levels)	D.9.1 and D.9.2: • Two-sample hypothesis tests; Probability value (p-value) E.9.1: • Estimation of a background threshold value (BTV) that represents the high-end of the background distribution
Decision Rule	D.9.1: • If COPC soil concentrations from different geomorphologic environments are statistically different from each other, then site soil comparisons to background will need to be assessed specific to each geomorphologic environment. If COPC soil concentrations from different geomorphologic environments are not statistically different from each other, then the datasets from the different

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
	geomorphologic environments can be combined for the purposes of making site comparisons D.9.2: • If COPC concentrations within site exposure areas are statistically higher than background, then the COPC will be deemed a site-related contaminant. If COPC concentrations within site exposure areas are not statistically higher than background, then the COPC will not be identified as a site-related contaminant
Specification of the Estimators	E.9.1: This investigation will estimate an upper-bound threshold from the background concentration distribution. A BTV is needed for each environmental medium so that individual site samples can be classified as either contaminated (site > BTV) or not contaminated (site ≤ BTV)
6 – Specify Performance/Acceptance Criteria	
Investigation QA/QC	• Adherence to Field SOPs (provided in the QAPP tables and Appendix A), Site-specific methods as defined in Project FSP, and field equipment manufacturer's instructions as appropriate • Collection of field and laboratory QC samples as prescribed in the SAP • Verification of laboratory certifications • Assessment of DQIs and their associated measurement performance criteria for precision, accuracy, representativeness, comparability, completeness, and sensitivity metrics (provided in the Project QAPP) • Implementation of data verification and validation
Statistical Hypothesis/Tolerable Limits for Decision Errors	D.9.1: • When comparing background datasets between two different geomorphologic environments, it will be assumed that COPC concentrations in the two areas are different until proven otherwise. Thus, the null and alternative hypotheses are as follows. H_0: The COPC concentration distribution in geomorphologic environment #1 is significantly different from the COPC concentration distribution in geomorphologic environment #2 (based on an appropriate two-sample hypothesis test comparison given the distribution of the datasets); the datasets should be kept separate. H_A: The COPC concentration distributions in the two geomorphologic environments are not statistically different from each other; the datasets can be combined. When making comparisons to background concentrations, the probability of a Type I error should not exceed 5% (i.e., $\alpha = 0.05$). The probability of a Type II error is not specified; additional lines of evidence should be evaluated when the null hypothesis is not rejected. D.9.2: Multiple lines of evidence will be considered when comparing Mill Town data to background data: • When comparing background data to Mill Townsite data, it will be assumed that Site COPC concentrations within the exposure area are "Site-related" contaminants until proven otherwise (i.e., a Form II hypothesis test). Thus, the null and alternative hypotheses are as follows.

Text Table 6: DQO 9 Background Study Data Quality Objectives

DQO: DQO Step:	Background Study
	H_0: The mean COPC concentration in the Site samples exposure area is significantly higher than the mean COPC concentration in the background samples exposure area (based on an appropriate two-sample hypothesis test statistical comparison appropriate given the distribution of the datasets); the analyte COPC may be a Mill Town is a Site-related contaminant. H_A: The mean COPC concentration in the Site samples exposure area is less than or equal to the mean COPC concentration in the background samples exposure area; the analyte COPC is not a Mill Town Site-related contaminant. A Type I error is the more severe decision error (i.e., an analyte would be dismissed as being not Mill Town-related when it could be present due to the release). Therefore, when making comparisons to background concentrations, the probability of a Type I error should not exceed 5% (i.e., $\alpha = 0.05$). The probability of a Type II error is not specified; additional lines of evidence will be evaluated when the null hypothesis is not rejected.
Acceptable Limits on Estimation Uncertainty	E.9.1: Individual site samples will be compared to the BTV to make determinations about the location being contaminated or uncontaminated. To ensure the contamination extent does not inadvertently include concentrations representative of background, a high-end statistic from the background distribution (95^{th} percentile) should be selected for each environmental medium. Uncertainty in the 95^{th} percentile should be accounted for by calculating an UCL on this statistic, i.e., computing the 95% UCL on the 95^{th} percentile, which is the 95-95 upper tolerance limit (95-95UTL).
7 – Develop Plan for Obtaining Data	
	See **Section 5** of the SAP.

5 FIELD SAMPLING PLAN

The FSP guides data collection. This section includes subsections describing the planned investigation including:

- An explanation of sampling activities to be conducted with reference to figures illustrating the planned sample locations,
- A brief rationale for the sampling design and the sampling activities based on the DQOs established in **Section 4**,
- Detailed protocols that reference relevant SOPs for the sample collection methodologies, and
- A list of measurements to be conducted and how each measurement will be used.

5.1 GENERAL SITE PROCEDURES

This subsection describes procedures that will be performed before or during the field investigation that are not specific to the sampling medium. Because the Site is within a unit of the National Park System, field activities will be planned and implemented to protect public safety and preserve public enjoyment in close coordination with the NPS.

5.1.1 Site Preparation and Restoration Procedures

VHB will coordinate with Park staff to ensure boring locations are not within 10 feet of any known utilities. VHB will contact 811 Alaska Digline at least 15 days before mobilization to assess whether buried utilities exist at the Site and will either pre-screen boring locations using a private utility locator or will hand dig borings to 5 feet bgs to limit the likelihood of damaging unidentified buried utilities.

To keep the public safe during field work, an exclusion zone will be established around active sampling locations using barricades, safety cones, stakes, and/or caution tape, as needed.

Efforts will be made to minimize impacts to work sites. After completing ground-disturbing activities, reasonable efforts will be made to restore locations and disturbed ground surfaces to a natural condition. Following the completion of work at each location, investigation derived waste (IDW) will be transferred to appropriate labeled containers. See **Section 5.3** for a detailed discussion of IDW handling.

5.1.2 Historical and Archeological Recordation

Historically significant artifacts may be encountered during the Phase III RI. If VHB encounters objects other than soil or rock during sampling efforts in areas not previously assessed by the WRST archaeologist, the object will be left in place and the archaeologist will be notified. The archaeologist will assist in temporary removal of any artifacts found during sampling activities and will return them to their original location upon completion. If artifacts are found that cannot be removed, VHB will record the

artifact location and adjust sampling areas to avoid damaging artifacts, in consultation with the NPS archaeologist.

5.1.3 Equipment Decontamination Procedures

Hand-held equipment will be decontaminated at the point-of-use before introducing it into a sampling point, and after completion of work at a sampling point. Trace decontamination of small equipment will follow removal of solids and gross contamination and generally consists of washing and scrubbing with a laboratory-grade detergent, such as Liquinox, and rinsing with a large volume of tap or distilled water followed by a final triple rinse with deionized or distilled water, in accordance with *SOP-JCO-027: SOP for Decontamination of Field Equipment*. Equipment may be air dried or wiped dry with paper towels. Decontamination water from hand-held equipment will be contained in a 5-gallon bucket and transferred to a drum with a tight-sealing lid for characterization and disposal.

The drill rig will arrive on-Site clean and free from mud, soil, and vegetation. A centralized IDW staging area for the drilling rig and equipment will be established in consultation with the NPS. A staging area adjacent to the decontamination area will be large enough to allow storage of cleaned equipment and materials prepared for use, as well as to stage water tanks, fuel, and drums of decontamination IDW. The decontamination pad will be lined with heavy gauge plastic sheeting and designed with a collection system to capture decontamination water. The 55-gallon drums containing solid or liquid IDW will be placed on heavy gauge plastic sheeting or similar and covered with plastic sheeting when active field investigation is not occurring. Smaller decontamination areas for personnel and portable equipment will be established adjacent to each investigation area as necessary. These locations will include buckets to capture decontamination fluids, which will be transferred to 55-gallon drums.

Drilling equipment such as rods and casing will be decontaminated upon completion of drilling at each boring. A steam cleaner may be used to clean core barrels and steel casing on the decontamination pad. Solids and mud may be removed from equipment using a brush, paper towels, and clean water. Equipment blanks will be collected from decontaminated equipment to verify decontamination efficacy. All decontamination water will be contained in a bucket or on plastic sheeting and transferred to drums.

Non-IDW trash, such as wrappers and unused tubing trimmings, will be disposed of at a location specified by the NPS.

5.1.4 Mapping and Surveying

Final geographic coordinates and elevations of monitoring wells will be surveyed by a licensed land surveyor or marked by high-resolution GPS equipment. Surveyed locations will be determined to a horizontal accuracy of 0.1 feet referenced to the 1983 State Plane Alaska Zone 2 (feet). The water level measuring point and ground surface elevations at all monitoring wells will be included in the survey, determined to the nearest 0.01 feet based on NAVD88.

Geographic coordinates of locations that require a lower degree of precision, such as ISM DU boundaries, locations of individual soil samples, seeps/springs, etc.) will be recorded using a Global Positioning System (GPS) with submeter accuracy if satellite reception is adequate.

5.1.5 Dust, Vapor, Odor, and Noise Control

Field activities will be limited to normal work hours (i.e., between 7:00 am and 6:00 pm, but only during daylight hours) to minimize potential noise impact to residents and Park visitors. Work may occur seven days per week. Sampling activities are not anticipated to create excessive dust, vapor, odor, or noise; however, engineering controls (e.g., spraying the source of dust with potable water) will be applied if needed to manage dust, vapors, odor, and noise emitted during the field activities. These activities will be qualitatively monitored. Noise, odor, or dust complaints received by the public will be evaluated immediately, and practices will be changed to mitigate the situation, if practicable.

5.1.6 Damage to Site Structures

The Phase III RI field activities will involve subsurface investigations and the use of heavy equipment. During the field program, precautions will be taken to avoid damage to structures including utilities, roads and paths, trees, and buildings. Plywood, plastic mats, or similar materials will be used as needed to prevent excessive rutting or similar damage. Cones, stakes, and/or flagging will be placed around investigation locations and travel corridors as necessary to protect sensitive objects (e.g., large trees or unstable slopes). Contingency planning for damage includes the following:

- The Field Team Leader will be responsible for assessing and documenting damage and repairs in the daily reports. Documentation will include reports of interviews with those present and photographs of the damage. The NPS Contaminated Site Project Manager and the Cleanup Lead will be immediately notified of the damage.
- Depending on the scope of the damage, repairs will be made in a timeframe commensurate with the degree of damage and planning required for mitigation.
- Spills of petroleum that cause a film or sheen on surface water and spills of other hazardous substances in reportable quantities will be reported to the National Response Center at (800) 424-8802 and the Alaska Spill Prevention and Response Central Office at (907) 269-3063 as soon as possible. Spill kits will be available at the Site.

5.2 SAMPLE HANDLING

The construction material of the sampling devices (e.g., plastic, polyvinyl chloride [PVC], metal) will be appropriate for the COPCs being sampled and will be selected so as not to interfere with the chemical analyses being performed. Sampling equipment dedicated for use at one sampling location will be clean, unused and will be protected from contamination until ready for use. Non-dedicated sampling equipment

will be decontaminated according to the specifications in **Section 5.1.3** before initiating sampling activities and will be protected from contamination until ready for use.

Monitoring equipment used on-Site will be maintained, calibrated, and operated properly with consideration of the manufacturer's recommendations and the relevant SOPs. All field water quality measurements and related instrument calibrations will be executed in accordance with manufacturer's guidelines and SOPs.

Sample containers will be clean, unused, and supplied by the environmental laboratory. Samples will be packed in coolers with bagged wet ice or dry ice and sufficient padding to prevent breakage. A temperature blank will be provided in each cooler. Coolers will be sealed with a signed custody seal and packing tape. The custody seal will be attached such that the cooler cannot be opened without breaking the seal. Samples will be delivered to the laboratory upon demobilization of the field team. The laboratory will be responsible for notifying VHB as soon as possible of any inconsistencies or breakage upon receipt, and within one day at the latest. The laboratory will obtain approval from VHB prior to disposing of any samples.

5.2.1 Sample Labeling

Samples will be assigned a unique sample identifier shown on the COC form and sample container labels. **Text Table 7** summarizes the sample nomenclature for all samples, including duplicates, MS/MSDs, and equipment blanks.

Sample container labels will indicate the sample identifier, date and time of collection (24-hour time), initials of the sample collector, and the type of analysis required. Labels will be completed using waterproof ink unless impracticable because of weather conditions such as rain. The source of blind duplicate field samples will be provided with a fictional sample time, be identified only on field forms and/or in field books, and the information will not be provided to the laboratory.

Text Table 7: Sample Nomenclature

DQO 2/3 Surface Soil					
ISM Samples					
Medium- (surface soil sample "S-")	**Building/Lot Number** (e.g., 32C, 32D, 53)	**DU Identifier-** (e.g., dripline "drip-", yard "yard-", garden "gard-")	**Depth Interval** (e.g., 0 to 1 inches "0-1")	**Sample Replicate** (ISM replicate "A", "B", or "C")	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
Discrete Samples					
Medium- (surface soil sample "S-")	**Building/Lot Number** (e.g., 32C, 32D, 53)	**DU Identifier-** (e.g., dripline "drip-", yard "yard-", garden "gard-")	**Depth Interval-** (e.g., 0 to 1 inches "0-1-"	**Sample Type** (discrete sample "D")	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
DQO 6 Surface Water and Sediment					

Medium- (National Creek surface water "NCW-" or sediment "NCS-")	**Segment of National Creek** (upstream "U", midstream "M", downstream "D", or L for duplicate)	**Location Numerical Identifier-** (e.g., 1-, 2-, 3-, 4-, etc.)	**Sample Collection Date** (DDMMYY)	**-Filtered** (if filtered "-F")	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
DQO 7 Groundwater					
Sample Type- (groundwater sample "MW-")	**Location Numerical Identifier-** (e.g., 01-, 02-, 03-, 04-, etc.)		**Sample Collection Date** (DDMMYY)	**-Filtered** (if filtered "-F")	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
DQO 8 Subsurface Soil					
Sample Type (soil boring "SB")	**Location Numerical Identifier-** (e.g., 01-, 02-, 03-, 04-, etc., 50- through 55- for duplicate)		**Depth Interval-** (e.g., 2 feet "2-"	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")	
DQO 9 Background Study					
Soil ISM Samples					
DQO- (Background soil sample "BG-")	**DU Identifier** (e.g., 01, 02, 03, 04)		**Sample Replicate** (ISM replicate "A", "B", or "C")		**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
Soil Discrete Samples					
DQO- (Background soil sample "BG-")	**DU Identifier-** (e.g., 01-, 02-, 03-, 04-, 20- for duplicate)		**Numerical Identifier-** (discrete sample number e.g., 1-, 2-, 3-, 4-, etc.)		**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
Surface Water and Sediment Samples					
Medium- (National Creek surface water "NCW-" or sediment "NCS-")	**Segment of National Creek** (background "B", "K" for duplicate)	**Location Numerical Identifier-** (e.g., 1-, 2-, 3-, 4-, etc.)	**Sample Collection Date** (DDMMYY)	**-Filtered** (if filtered "-F")	**-QA/QC Identifier** (e.g., MS/MSD "-MS/MSD")
Groundwater and subsurface soil sample nomenclature identified under DQO 7 and 8, respectively.					
QC					
Sample Type- (equipment blank "EB-")		**Applicable DQO/Medium-** (e.g., surface soil "S-", surface water "NCW-", sediment "NCS-", groundwater "GW-", subsurface soil "SB-", background surface soil "BGS-", background water "BGS", background sediment "61ppro-		**Sample Collection Date** (DDMMYY)	

Sample Type- (trip blank "TB-")	**Numerical Identifier-** (e.g., 01-, 02-, 03-, 04-, etc.)	**Sample Collection Date** (DDMMYY)
Sample Type- (drilling water "DW-")	**Numerical Identifier-** (e.g., 01-, 02-, 03-, 04-, etc.)	**Sample Collection Date** (DDMMYY)

5.2.2 Sample Handling and Chain of Custody

COC records will be completed in accordance with *SOP-VHB-007: SOP for COC Records*. Field forms and COC records will be used to maintain a comprehensive record of sample collection, custody, and receipt by the laboratory. A COC form supplied by the laboratory will list the sampler's initials, sample identifier, date and time of collection, matrix, preservative, and requested analyses. Accompanying COC form(s) will be completed for each cooler of samples. The original form will be placed in the cooler in a waterproof plastic bag and a copy or photograph will be retained by the sampler.

Samples will be packed in coolers with bagged wet ice or dry ice and sufficient padding to prevent breakage. A temperature blank will be provided in each cooler. Coolers will be sealed with a signed custody seal and packing tape. The custody seal will be attached such that the cooler cannot be opened without breaking the seal. Samples will be delivered to the laboratory after the field program.

5.2.3 Documentation and Records

A daily record of field activities will be recorded by the Field Team Leader using the Daily Log form provided in Appendix B; the Daily Log will be transmitted to the Cleanup Lead before 10:00 am on the next business day when possible. Additional field forms will be completed by VHB personnel to document specific tasks, such as equipment calibration and sample collection. Additional notes may be kept in field logbooks. Blank copies of field forms are included in Appendix B.

5.3 INVESTIGATION DERIVED WASTE SAMPLING AND DISPOSAL

IDW includes all soil that has been removed from its original location and not used to fill sample containers. In accordance with NPS policy, all IDW will be placed in appropriate containers, such as sealed buckets or drums, and will not be placed back on the ground. Containers will be consolidated and secured in an area approved by NPS within the Site property. Soil samples collected in plastic bags will be placed in sealed buckets or steel drums after they have been appropriately logged and sampled. A representative composite sample will be collected for IDW characterization. No decontamination liquids will be released to the ground or to surface waters. Water used for decontaminating equipment will be placed in sealed buckets or drums. A minimum of one liquid IDW sample will be collected for characterization.

IDW samples will be submitted to the laboratory and analyzed for parameters required by the selected disposal facility using methods identified in the QAPP. It is anticipated that the following parameters will be required:

- The solid IDW samples will be extracted by TCLP and analyzed for RCRA 8 metals (arsenic, barium, cadmium, total chromium, lead, mercury, selenium, and silver), VOCs, SVOCs, and total PCB Aroclors.
- The liquid IDW samples will be analyzed for RCRA 8 metals, VOCs, SVOCs, and PCB Aroclors.

NPS shall be identified as the generator of wastes for the purpose of regulatory or policy compliance. A bill of lading (for non-hazardous wastes) or manifest (for hazardous wastes) will be prepared for each shipment of IDW and will be signed by NPS. Waste will be removed as soon as possible for disposal at an appropriate facility, based on the analytical results. Final transporter and disposal facility selection will be based on waste acceptance policies, capacity, and pricing, and will be approved by NPS in advance. All project waste operations will be conducted in accordance with applicable regulations and requirements, including state and federal regulations such as RCRA, Toxic Substances Control Act, and Department of Transportation regulations. The shipping documents will also be signed by the driver prior to the waste leaving the Site; these documents will accompany all shipments of waste while in transit at all times. The certified disposal facility tickets will be collected by VHB and provided to NPS to maintain information related to these wastes, including all disposal analyses by waste type, manifests/bills of lading, and disposal facility weight tickets.

Non-soil and water waste, such as wrapping and non-hazardous debris generated during RI activities will be placed in bins or dumpsters provided by or at a location specified by NPS for disposal off-Site at a licensed landfill.

5.4 HEALTH AND SAFETY

A Health and Safety Plan (HASP) that covers all VHB employees is provided in Appendix C. Each subcontractor who will be performing activities that could result in contact with or release of contamination is responsible for preparing a separate HASP that covers their own specific activities, and for providing staff who are appropriately trained in the Occupational Safety and Health Administration's Hazardous Waste Operations and Emergency Response 1910.120 requirements. Safety at the Site is a top priority.

5.5 DQO 2/3 SURFACE SOIL INVESTIGATION

Surface soil samples will be collected from the Silk Stocking Cottages to answer the following PIQs:

- What concentrations of COPCs in surface soil are a reasonable estimate of exposure point concentrations for human and ecological receptors at residential properties within the Mill Town?

5.5.1 DQO 2/3 Surface Soil Sampling Locations

Surface soils around the Silk Stocking Cottages will be sampled and analyzed using ISM techniques in accordance with ITRC guidance. This structured composite sampling processing procedure is designed to reduce data variability, increase sample representativeness, and increase reproducibility (ITRC, 2020). ISM samples will be collected from seven Eus. The proposed boundaries for each EU are identified on **Figure 10**.

Proposed sampling locations are summarized in **Table 1A** and presented graphically on **Figure 10**. Locations of Eus were selected considering the USEPA Superfund Lead-Contaminated Residential Sites Handbook (USEPA, 2024) and the ITRC Technical and Regulatory Guidance for ISM (ITRC, 2020). The seven proposed Eus include the following:

- The dripline for Silk Stocking Cottages 32C (sample collected between the building walls and the dripline of the building);
- The dripline for Silk Stocking Cottage 32D (sample collected between the building wall and the dripline of the building);
- The dripline for the garage located directly south of building 32D (sample collected between the building wall and the dripline of the building);
- The yard encompassing Silk Stocking Cottage 32C (the cleared portions of the parcel only – not including the dripline areas around the building),
- The yard encompassing Silk Stocking Cottage 32D and the garage (the cleared portions of the parcel only – not including the dripline areas around each building);
- The garden located on lot 53, directly west of Silk Stocking Cottage 32C; and
- The yard area of lot 53 (the cleared portion of the parcel, only – not including the garden).

In addition to DQO considerations discussed in Section 4, the rationale for selecting each soil sampling location is provided in **Table 1A**. There are seven ISM Eus for DQO 2/3. EU boundaries may be altered based on field conditions and accessibility; alterations from the proposed EU boundaries will be documented in field notes and daily reports.

5.5.2 DQO 2/3 Surface Soil Sampling Protocol

GPS will be used to locate the boundaries of the Eus mapped in **Figure 10** in the field. The boundaries of the Eus will be marked using flagging tape, pin flags, or stakes and recorded in ArcGIS Field Maps using GPS. Markers will be removed after sample collection.

Surface soil ISM samples will be collected from 0 to 1 inches bgs within each EU in triplicate, apart from the garden EU, which will be sampled from 0 to 12 inches bgs in triplicate. The number of increments to be collected within each EU will be calculated based on the area of the EU; an increment density of approximately 100 square feet per increment will be maintained for each EU (consistent with the approach used during the Phase I RI in the DQO 4 Blasting Caps Disposal Areas investigation). Each ISM replicate will be composed of a minimum of 30 increments. **Table 1A** summarizes samples that will be collected from each EU.

For 0 to 1-inches bgs ISM samples, a 1-inch plug will be used to collect increments. For the 0 to 12 inches bgs ISM samples, a cordless drill with a small-diameter (approximately 0.25-inch) ship auger bit will be used to obtain an approximately cylindrical core from each increment location to a depth of 0 to 12 inches bgs, unless soil conditions require an alternative equivalent method (such as an open-slot push probe). The technique is a modified version of the technique presented in the Hawaii Technical Guidance Manual Notes (HDOH, 2011) and is described as follows:

1. Attach to a cordless drill a 0.25-inch drill bit with a minimum length of 18 inches. Mark the 12-inch depth with paint and/or tape. Verify marking between Eus because there is a high potential for wear when contacting soil.
2. Clear debris from the ground surface at the sampling location.
3. Keeping the drill perpendicular to the ground, advance the bit to the target depth of 18 inches bgs. Soil will collect between the auger flights.
4. Pull the drill straight up and, keeping the bit vertical, transfer the soil directly into the sampling container. Use the drill to spin the bit quickly in the container to release the soil. If necessary, use a gloved hand to remove soil from the bit.
5. The same instrument may be used for all increments within an EU. Decontaminate the drill bit and change gloves between Eus.

Samples will be collected into 1-gallon Ziploc bags dedicated to each replicate. The small hole created during increment collection will be backfilled with surrounding soil to minimize the visibly disturbed area. MS/MSD samples will be collected at a rate of 1 per 20 samples.

In a subset of DUs, ISM samples will be collected from 0 to 1 inches bgs for IVBA analysis (0 to 12 inches bgs in the garden) to assess the bioavailability of lead and arsenic in soil. ISM surface soil samples for IVBA analysis will be sieved in the field using No. 4 or No. 10 sieve screens to verify sufficient sample is provided to the laboratory for analysis. IVBA samples will be sieved to less than 150-µm by the laboratory prior to analysis, in accordance with the laboratory method. Replicates will not be collected for IVBA samples.

IVBA analysis cannot be completed by the environmental laboratory that will conduct the majority of the sample analysis for the Phase III RI (SGS Anchorage). Samples collected for IVBA analysis will be shipped to Pace Analytical for analysis, and therefore, will be collected separately from ISM samples collected for TAL metals and PAH analyses.

5.5.3 DQO 2/3 Surface Soil Field Measurements

The location of EU boundaries will be determined in the field using the interactive electronic field map and GPS. Geologic descriptions of samples, visual/olfactory evidence of contamination, and apparent visual moisture content will be recorded using electronic field forms, in accordance with *SOP-VHB-036: SOP for Geologic Description of Sediments* (includes soil and sediments). Photographs will be taken periodically and uploaded to the electronic field map.

5.5.4 DQO 2/3 Surface Soil Analytical Measurements/Methods

Each ISM sample will be analyzed for the following analytes:

- TAL metals (USEPA Methods 6020B and 1631E) – sample will be sieved to less than 150-µm by the environmental laboratory prior to analysis
- PAHs (USEPA Method 8270 SIM for PAHs)

ISM samples from a subset of Eus will be analyzed for the following analytes:

- IVBA (USEPA method 1340) – sample will be sieved to less than 150-µm by the environmental laboratory prior to analysis

5.6 DQO 6 SURFACE WATER AND SEDIMENT INVESTIGATION

Surface water and sediment samples will be collected from National Creek to answer the following PIQs:

- Do the concentrations of COPCs in National Creek surface water/sediment exceed PALs?
- Where are locations (in the vicinity of the Mill Town) of groundwater seeps, springs, or storm water run-off conveyances that could act as COPC sources to National Creek?
- What concentrations of COPCs in National Creek surface water/sediment are reasonable estimates of exposure point concentrations for human and ecological receptors?

5.6.1 DQO 6 Surface Water and Sediment Sampling Locations

Surface water and sediment samples will be collected from the approximate upstream, midstream, and downstream locations sampled during the Phase II RI. See **Table 1B** for sample location rationale and **Figure 11** for sample locations.

Sampling areas/locations may require field adjustment depending on the presence of flowing surface water. Additionally, sediment sample locations will be selected to target areas of fine-grained sediment deposition to provide the most likely areas of highest COPC concentrations. Several of the COPCs tend to sorb to smaller sized sediment grains. Sample COPC concentrations will be compared to PALs (**Tables 6c and 6d**).

5.6.2 DQO 6 Surface Water and Sediment Sampling Protocol

COPC concentrations in surface water are expected to vary at different river stage/flow conditions. Snow melt and precipitation may dilute COPC concentrations or result in increased concentrations of COPCs due to increased turbidity. Groundwater discharge may also affect concentrations in National Creek. Multiple sampling events are therefore required to evaluate the temporal variability in concentrations of COPCs in National Creek surface water. Sediment concentrations are considered less likely to have temporal variation than surface water; however, several rounds of sediment samples are required to

evaluate COPC concentrations in sediment. Available data regarding variations in stream flow due to seasonal and/or precipitation events will be used to identify when to collect samples during flow conditions that are different from the Phase I and II RI sampling conditions. VHB will also record the location of groundwater seep discharges, springs, or storm water run-off conveyances observed while navigating and sampling National Creek. The ideal conditions for identifying seeps, springs, and storm water run-off conveyances are likely during spring melt and/or following precipitation events.

The Phase III RI field program may consist of one or two mobilizations, depending upon available funding; one in the spring timeframe to target snow melt (June 2024, if possible) and one later in the summer (between July and early September 2024). Co-located samples of surface water and sediment will be collected from National Creek at specified locations during the first mobilization. During the second mobilization, only surface water samples will be collected. Properly calibrated multiparameter probes will be placed directly into National Creek surface water at the sample location where possible. If this is infeasible, surface water will be placed in a clean container, and field water quality parameters will be recorded when the readings stabilize. Surface water samples will be field filtered to allow for the analysis of dissolved metals and DOC. Unfiltered samples will be collected to allow for the analysis of total metals, PAHs, VOCs, sulfate, chloride, alkalinity, sulfide, and hardness. Surface water samples will be collected first, followed by sediment samples to limit turbidity in the water column. Surface water samples will be collected from the middle of the water column (or the top 0-2 inches if the water depth is less than one foot) using a pole-mounted sample container, peristaltic pump, or stainless-steel scoop in accordance with *SOP-VHB-035: SOP for Surface Water Sampling*. Samples will be collected from downstream locations to upstream locations. Sediment samples will be collected from the top 0-6 inches of sediment. Sediment will be collected using a clean stainless steel long-handled scoop. When using a long-handled scoop, the sample will be lifted quickly through the water column to decrease the loss of fines. Sediment samples will be collected in a clean stainless-steel bowl, mixed well, then transferred into new glass sediment jars. Field duplicates will be collected at a rate of 1 per 10 samples. MS/MSD samples will be collected at a rate of 1 per 20 samples.

To support identifying locations of groundwater seep discharges, springs, or storm water run-off conveyances in the vicinity of National Creek, aerial photographs and topographic maps will be consulted and combined with on-Site visual examinations.

5.6.3 DQO 6 Surface Water and Sediment Field Measurements

Surface water and sediment sample locations, groundwater seep discharge, spring, and storm water run-off conveyance locations, and tailings deposit locations will be recorded with GPS. The following surface water field measurements will also be recorded with a multiparameter probe:

- pH
- Conductivity
- Temperature
- Turbidity
- Dissolved oxygen

- ORP

For sediment samples, an estimated sediment grain size will be recorded on the designated field form.

5.6.4 DQO 6 Surface Water and Sediment Analytical Measurements/Methods

Surface water samples will be analyzed for the following analytes:

- Total (unfiltered) and dissolved (filtered) TAL Metals (USEPA Methods 6020 and 1631)
- Hardness (by calculation)
- PAHs (USEPA Method 8270-SIM)
- VOCs (USEPA Method 8260)
- Copper Biotic Ligand Model input parameters (one sample per upstream, midstream, and downstream areas per mobilization):
  - DOC (USEPA Method 9060A-modified)
  - Sulfate and chloride (USEPA Method 300.0)
  - Alkalinity (SM 2320B)
  - Sulfide (SM 4500S D)

Both filtered (dissolved) and unfiltered (total) analyses will be performed on surface water samples analyzed for metals. Dissolved metals and DOC samples will be field filtered using a 0.45-µm dedicated and disposable filter while filling the sample containers.

Sediment samples will be analyzed for the following analytes:

- TAL Metals (USEPA Methods 6020 and 1631)
- PAHs (USEPA Method 8270-SIM)
- VOCs (USEPA Method 8260)
- TOC (USEPA Method 9060 modified)

5.7 DQO 7 GROUNDWATER INVESTIGATION

Groundwater samples will be collected from monitoring wells installed in the vicinity of the Mill Town to answer the following PIQs:

- Do concentrations of COPCs in groundwater in the vicinity of the Mill Town exceed PALs?
- What is the horizontal and vertical extent of groundwater with concentrations of COPCs greater than PALs?
- What is the groundwater flow direction in the vicinity of the Mill Town?

5.7.1 DQO 7 Groundwater Sampling Locations

To determine the horizontal extent of COPCs in groundwater and assess groundwater flow directions across the Site, monitoring wells will be installed at targeted and spatially representative locations near the Mill Town, as presented on **Figure 12.** See rationale for monitoring well locations in **Table 1C**. Groundwater monitoring well locations may require in-field adjustment depending on, among other factors, the presence (or lack thereof) of groundwater in overburden and/or shallow bedrock, refusal encountered by the drill rig, and potential accessibility restrictions. Proposed monitoring well locations were selected to target the following:

- Locations with known surface soil impacts and/or visible tailings based on previously collected data – to understand whether groundwater impacts are present near confirmed source areas;
- Locations near existing groundwater wells;
- Locations that are spatially representative of areas within the vicinity of the Mill Town – to support understanding of groundwater flow direction and horizontal extent of elevated COPCs across the Site;
- Locations accessible by the drill rig;
- Locations where buried utilities or explosives have not been identified; and
- Locations where installed wells are less likely to be damaged (i.e. avoid main roads with vehicle traffic and areas that may be plowed during the winter).

Depths to groundwater, the occurrence of groundwater in overburden and/or shallow bedrock, the thickness of overburden, and subsurface lithology are unknowns at this stage of the RI. As a result, the depths at which monitoring wells will be installed are also unknown and expected to vary across the Site based on location-specific conditions. During the Phase III RI, monitoring well installation will be constrained to overburden or shallow bedrock (defined as approximately 15 feet below the competent bedrock interface).

The number of wells installed during the Phase III RI will depend on the rate of monitoring well installation, which will depend on lithology, depth to groundwater, and other factors. To accommodate these unknowns and provide the best opportunity to collect useful data within the scheduled time the drillers will be on-Site, the locations of proposed monitoring wells are listed numerically on **Figure 12** and in **Table 1C** in the order they will be installed (MW-1 will be installed first, followed by MW-2, etc.). A total of 12 proposed monitoring well locations have been identified, three of which are associated with DQO 9 (**Section 5.9**).

VHB will strive to successfully install monitoring wells at each of the proposed locations during the Phase III program. Factors beyond the control of VHB or VHB's subcontractors, including refusal, depth to groundwater, and/or absence of groundwater in overburden or shallow bedrock, may necessitate adjustments to proposed monitoring well locations. Several factors will determine whether a boring location should be temporarily abandoned in favor of moving on to the next listed boring location, including:

- **Refusal**: if refusal is encountered prior to reaching the groundwater table, the boring location will be adjusted horizontally by approximately 10-15 feet; a localized subsurface condition (e.g. a large cobble) may cause refusal. Another attempt will be made to advance the boring to the

groundwater table in the new location. If refusal is encountered again, drilling may be shifted to the next proposed monitoring well location.

- **Bedrock**: if bedrock is reached before encountering the groundwater table, drilling will continue approximately 18 to 20 feet into bedrock. If groundwater first occurs in shallow bedrock, a monitoring well will be installed in shallow bedrock. If groundwater is not encountered in overburden or shallow bedrock at a proposed well location, a monitoring well may be installed in shallow bedrock based on observed geological conditions and discussion with NPS. If a bedrock well is not installed in this boring, the boring will be grouted and drilling will proceed at the next proposed monitoring well location. Note: if overburden at this location shows evidence of VOC contamination (visual, olfactory, and/or PID), drilling will stop at the bedrock interface to avoid the potential for carrying dense non-aqueous phase liquid (if present) into bedrock during the drilling process.
- **Depth to Groundwater**: if soil borings are advanced in overburden material to depths of 100-feet without encountering bedrock or groundwater, the boring will be grouted and drilling will proceed at the next proposed monitoring well location.

If all proposed monitoring wells are successfully installed ahead of schedule, additional exploratory borings may be advanced at select locations across the Site.

5.7.2 DQO 7 Groundwater Sampling Protocol

Monitoring Well Installation

Borings will be advanced using an air rotary casing advancer system with 4.5-inch diameter outer casing. This type of drill rig achieves borehole advancement by using hydraulic power to rotate a down-the-hole hammer (DTH) which is activated by high pressure air from an air compressor. The DTH rotates and simultaneously crushes the lithology into small pieces, while each hammer percussion of the DTH also pulls the casing down with it. High pressure air is used as a circulating fluid to cool the drill bit, power the DTH, and bring the generated drill cuttings from the bit to the surface. A cyclone is used to capture the air and cuttings that come to the surface. This drilling method may generate dust in the cyclone while drilling through unsaturated material. Water sourced from National Creek (or another clean water source) will be used to suppress dust by pumping/spraying water on cuttings that are funneled through the cyclone. Water sourced from National Creek that is used for dust suppression will not be injected into the subsurface nor come into contact with soils or groundwater that will be sampled (subsurface soils will be collected from several depth intervals during advancement of soil borings as outlined in **Section 5.8**).

Geological observations and logging will be conducted by advancing Modified Penetration Test ("MPT") split-spoons (3-inch diameter) continuously through unconsolidated overburden material. Either a standard down-hole hammer and camera system or NQ3 core tooling will be used to log geology when drilling through bedrock. Geologic descriptions will be recorded on Monitoring Well Log field forms (**Appendix B**) and will include observations of grain size, color (according to the Munsell chart), water content, and texture in accordance with *SOP-VHB-036: SOP for Geologic Description of Sediments.* Bedrock cores will be logged in general accordance with *Midwest GeoSciences Group's Field Guide for Rock Core Logging and Fracture Analysis*. A photoionization detector ("PID") will be used to screen for

potential volatile impacts. After soil cores have been logged and samples are collected, soil will be placed in a drum for waste characterization before disposal (see **Section 5.3**).

During bedrock coring, potable water may need to be injected to cool the drill bit as it is advanced into bedrock. Only potable water will be mixed with bentonite and/or cement used to seal monitoring wells or abandon boreholes. Potable water will be used during drilling. Water will be stored in polyethylene tanks or similar, which will be filled at a location designated by NPS. A sample of the water used during drilling will be collected from the tank; one sample will be collected from each water source. If multiple tanks and pumps are used in series, a single sample will be collected after the water is passed through all of the tanks and pumps.

The approximate depth of the water table will be estimated during drilling by observing moisture content in drill cuttings and/or retrieved cores, and by using an electronic water level indicator. Once drilling has reached the targeted depth, the depth to the bottom of the boring will be measured with a weighted measuring tape. If necessary, filter sand will be used to fill the bottom of the boring until the desired depth of the bottom of the screen is reached. Bentonite chips or pellets may be added beneath the filter sand if the boring extends more than 2 to 3 feet beyond the desired target depth of the well screen bottom. Wells will be screened across the water table and completed with 10-foot-long screens. Shallow wells will be installed with the top of screen no less than 3 feet bgs.

A 2-inch diameter pre-pack PVC well screen with 0.006-inch slots will be threaded to a 2-inch diameter PVC casing and placed in the boring. Screens, casing, and other well construction materials will be new, unused, and kept clean until they are placed in the borehole. Pre-pack well screens will be used to place a uniform sand pack around the well screen. Well screens will not be installed across confining units. Filter sand (10/20 size or equivalent) will be placed in the annular space around the screen to 2 feet above the top of the screen. A minimum of a 2-foot-thick layer of granular bentonite or bentonite chips will be placed above the top of the sand pack and hydrated with potable water if it is above the groundwater table. If there is additional open annular space, a tremie pipe will be used to fill it with a bentonite slurry to 0.5 feet bgs.

A reference mark will be made on the top of the PVC casing for surveying and depth-to-groundwater measurements. A locking well cap will be placed on the well casing. Locks on the well cap and the protective casing will have identical, standard keys supplied by VHB. Copies of the keys will be provided to NPS personnel. Monitoring wells will be completed at the surface with either a flush-mount road box embedded in concrete to prevent damage to the well or a 6-inch steel locking stick-up monument depending on the location and relative traffic.

A well completion diagram that documents relevant material quantities and placement depths will be prepared for each monitoring well installed. Blank monitoring well construction logs are included in **Appendix B**.

Borings that are unsuitable for monitoring well installation (reached refusal or depth limitations before encountering the water table) will be abandoned by pumping a concrete/bentonite slurry from the bottom to the top of the boring using a tremie pipe. Bentonite chips or pellets may be used to fill annular spaces above the water table but must be hydrated immediately with potable water. Excess grout will be disposed of in IDW drums. Abandoned borings will be inspected 24 to 48 hours after slurry placement to check for settling; if settling has occurred, bentonite chips or additional grout will be added to the hole to bring the level within 0.5 feet of the ground surface. After the bentonite and grout have cured and dried, native soil

surrounding the hole will be swept into the hole to make the ground level with the surrounding area. Holes should not present a trip hazard after abandonment.

Monitoring Well Development

Monitoring wells will be developed after installation. Development will be conducted either with a surge block and inertial valve pump, a peristaltic pump, or a whale pump depending on the depth to groundwater. Development is designed to surge and purge the well while pumping at a rate much greater than would be used for sampling. Development will generally be continued until discharge water appears visibly clear quickly after surging and when a minimum of three well volumes of water has been removed. If water does not appear visibly clear after removal of a large volume of water relative to the recharge rate and well volume (e.g., 10 well volumes), development will be discontinued. The well development process will be documented on groundwater sampling or development forms for each well (**Appendix B**).

Water Level Measurements

After monitoring wells have been installed, one synoptic water level round will be conducted to collect data for calculation of horizontal and vertical gradients. Water levels will be measured with an electronic water level meter. The water level meter will be decontaminated between wells. The synoptic round will be performed after the effects of purging, well installation, and development are likely to have subsided. The synoptic water level round will be completed in less than four hours, or sooner if it begins to rain during the activity. Precipitation for 24 hours prior to, and during, the synoptic rounds shall be reported based on data available for the nearest available weather station.

Groundwater Sampling

Monitoring wells will be sampled near the end of the field mobilization to allow the wells to recover from drilling and development, and to allow collected groundwater samples to be delivered to the laboratory within the holding times prescribed by the analytical methods. Wells will be purged and sampled using low-flow methods in accordance with *SOP-VHB-053: SOP for Low Stress Groundwater Sampling of Monitoring Wells and Piezometers*. Because the depth to water is unknown, VHB will bring equipment to support sampling of wells of different depths.

- Depth to water less than 25 feet below top of casing: peristaltic pumps will be used for purging and sampling.
- Depth to water greater than approximately 25 feet below top of casing: bladder pumps will be used for purging and sampling.

The tubing intake will be positioned at the approximate midpoint between the bottom of the screen interval and the measured groundwater level in the wells. The objective of this intake placement is to avoid or reduce formation water cascading down the inside of the screen before sample collection. Field parameters, as described in **Section 5.7.3**, will be measured during low flow sampling to indicate when stable conditions have been achieved and sampling may occur.

Samples for VOC analysis will be collected first, followed by samples for SVOCs, and metals. Groundwater samples will be unfiltered, apart from the sample analyzed for DOC, which will be field-filtered. Sample containers will be sealed and chilled.

Field duplicates will be collected at a rate of 1 per 10 samples and MS/MSD samples will be collected at a rate of 1 per 20. If a listed well does not produce water at a sufficient rate for efficient collection of the QC samples, alternative locations for those samples will be chosen in the field. In addition, a trip blank will be included in each cooler containing samples for VOC analysis, and will be analyzed for VOCs. Trip blanks will be prepared by the laboratory and will accompany sample containers at all times.

5.7.3 DQO 7 Groundwater Field Measurements

Well Development

An electronic water level meter will be used to monitor drawdown during well development. A turbidity meter will be used to check turbidity measurements when the purge water becomes increasingly clear. If feasible, well development will continue until the turbidity reading is less than 10 Nephelometric Turbidity Units ("NTU"). If water does not become clear after removal of a large volume of water relative to the recharge rate and well volume, development will be discontinued, and a note will be made on the sampling forms.

PID Screening

PID screening of the well headspace will be performed immediately upon opening the well for sampling. The result will be recorded on the groundwater sampling field form.

Low Flow Sampling Parameter Monitoring

A multiparameter instrument will be used to measure standard field parameters (pH, temperature, specific conductance, ORP, and dissolved oxygen) during purging. A turbidity meter will be used to collect turbidity measurements. The multiparameter instrument and turbidity meter will be calibrated daily before use and calibration information will be recorded on the multiparameter instrument calibration sheet and turbidity meter calibration sheet, respectively (**Appendix B**). During purging, the field parameter measurements will be recorded on groundwater monitoring well sample collection record forms (**Appendix B**) at a minimum of 5-minute intervals. Samples will be collected after parameters have stabilized, with three consecutive readings within the following limits:

- Turbidity = <1 NTU or +/-10% for values above 1 NTU. Samples will not be filtered
- DO = +/-10%
- Specific conductance =+/-3%
- Temperature = +/-0.2°C
- pH = +/- 0.1 units
- ORP = +/- 10 millivolts

Water Level Measurements

Water levels will be measured before purging and sampling in each well. The measurement will be performed with an electronic water level meter in accordance with VHB's SOP.

5.7.4 DQO 7 Groundwater Analytical Measurements/Methods

Groundwater samples will be analyzed for the following analytes and analytical methods.

- TAL Metals (USEPA Methods 6020 and 1631)
- SVOCs (USEPA method 8270 and 8270-SIM)
- VOCs (USEPA Method 8260D): the full standard analytical laboratory list of VOCs
- Copper Biotic Ligand Model input parameters (for wells located in the vicinity of National Creek and/or in the vicinity of seeps observed during the Phase I RI):
  - DOC (USEPA Method 9060A-modified)
  - Sulfate and chloride (USEPA Method 300.0)
  - Alkalinity (SM 2320B)
  - Sulfide (SM 4500S D)

5.8 DQO 8 SUBSURFACE SOIL INVESTIGATION

Subsurface soil samples will be collected to answer the following PIQs:

- Do concentrations of COPCs in subsurface soil in the vicinity of the Mill Town exceed PALs?
- What is the horizontal and vertical extent of subsurface soil with COPCs concentrations that exceed PALs?

5.8.1 DQO 8 Subsurface Soil Sampling Locations

Subsurface soil samples will be collected from soil borings completed during groundwater monitoring well installation (see **Figure 12**). Samples will be collected from several pre-determined intervals throughout the length of the recovered soil borings, as discussed in **Section 5.8.2**. Subsurface samples will only be collected from overburden soil; cobbles, boulders, bedrock, or other large, consolidated units will not be sampled.

5.8.2 DQO 8 Subsurface Soil Sampling Protocol

Methodologies associated with soil boring advancement and collection of cores are outlined in **Section 5.7**. VHB will screen soil cores for VOCs using a PID at 2-foot intervals. Soil samples will be collected from the core using hand tools. VOCs will be collected first (if applicable), followed by SVOCs and metals.

Subsurface soil samples will be collected at the following depths for the listed analyses at each boring:

- 0 to 6 inches bgs: TAL Metals (USEPA Methods 6020B and 1631E) and SVOCs (USEPA Methods 8270 and 8270-SIM)
  - Where elevated PID readings (e.g., 10 parts per million above background readings or greater) and/or visual/olfactory indications of volatile contaminants, VOCs (USEPA Method 8260) will also be analyzed
- Every 2 feet bgs until the water table is reached: TAL Metals (USEPA Methods 6020B and 1631E) and SVOCs (USEPA Methods 8270 and 8270-SIM)
  - Where elevated PID readings and/or visual/olfactory indications of volatile contaminants, VOCs (USEPA Method 8260) will also be analyzed
- Immediately above the water table: TAL Metals (USEPA Methods 6020B and 1631E), SVOCs (USEPA Methods 8270 and 8270-SIM), and VOCs (USEPA Method 8260)
- Every 4 feet bgs beneath the water table: TAL Metals (USEPA Methods 6020B and 1631E) and SVOCs (USEPA Methods 8270 and 8270-SIM)
  - Where elevated PID readings and/or visual/olfactory indications of volatile contaminants, VOCs (USEPA Method 8260) will also be analyzed

No more than five subsurface soil samples will be sent to the laboratory for analysis for each boring. The field team will select a subset of the samples outlined above to send to the environmental laboratory for analysis; the field team will consider soil descriptions, PID screening results, and sample depths when selecting which samples will be submitted to the environmental laboratory.

5.8.3 DQO 8 Subsurface Soil Field Measurements

Geologic descriptions of samples, visual/olfactory evidence of contamination, PID readings, and apparent visual moisture content will be recorded using electronic field forms, in accordance *with SOP-VHB-036: SOP for Geologic Description of Sediments* (includes soil and sediments). Photographs will be taken periodically and uploaded to the electronic field map.

5.8.4 DQO 8 Subsurface Soil Analytical Measurements/Methods

Subsurface soil samples will be analyzed for:

- TAL Metals (USEPA Methods 6020B and 1631E)
- SVOCs (USEPA Methods 8270 and 8270-SIM)
- VOCs (USEPA Method 8260)

See **Section 5.8.2** for a detailed listing of sampling depths and analyses. Field duplicates will be collected at a rate of 1 per 10 samples and MS/MSD samples will be collected at a rate of 1 per 20 samples. Trip blanks will be prepared by the laboratory and will accompany VOC sample containers.

5.9 DQO 9 BACKGROUND STUDY

Background samples will be collected for several media including surface soil, subsurface soil, surface water, sediment, and groundwater to answer the following PIQs:

- Do background soil COPC concentrations differ between the different geomorphologic environments present at the Site?
- Are Site exposure area concentrations of COPCs in environmental media higher than background?
- What are high-end background threshold concentrations for each COPC that can be used to delineate the site-related contamination extent in environmental media?

5.9.1 DQO 9 Background Study Sampling Locations

Background sample locations were selected to mirror Site geology, geologic process, ecology, and ecologic processes while targeting areas not likely to be impacted by former mining and milling operations. Background surface soil sampling locations were selected so that they were not within 150 feet of a rural road or 300 feet of a building or structure, where possible (consistent with USGS background study methodology). Selected background locations are presented on **Figure 13**.

Background surface soil samples will be collected from the following areas:

- Forested area north of the Mill Town (BG-01) – selected to mirror the geology on the eastern side of the Mill Town.
- Northwest moraine (BG-02) – selected to mirror the moraine geology on the western side of the Mill Town.
- Forested area east of the Mill Town (BG-03) – selected to mirror the geology of the eastern side of the Mill Town.
- West moraine (BG-04) – selected to mirror the moraine geology on the western side of the Mill Town.

Surface water and sediment samples will be collected from the following areas:

- A location approximately 2000 feet upstream of the Mill Town (NCW-BG1 through NCW-BG3).
- A location approximately 1000 feet upstream of the Mill Town (NCW-BG4 through NCW-BG6).

Groundwater and subsurface soil samples will be collected from the following areas (as outlined in DQO 7 and DQO 8):

- MW-05 – presumed hydraulically upgradient and cross-gradient of Mill Town
- MW-11 – presumed hydraulically cross-gradient of Mill Town
- MW-12 – presumed hydraulically cross-gradient of Mill Town

5.9.2 DQO 9 Background Study Sampling Protocol

Surface Soil

Two types of soil samples will be collected during the Phase III RI:

- Discrete surface soil samples – 10 discrete surface soil samples will be collected from 0 to 6 inches bgs from each of the areas identified in **Section 5.9.1**. These samples will be collected from random locations within the DU described below and will provide a background discrete surface soil dataset that can be compared to discrete Site surface soil samples collected during the Phase I and Phase II RIs.
- ISM surface soil samples – one 0.25-acre DU will be established at each area identified in **Section 5.9.1**. ISM surface soil samples will be collected from 0 to 6 inches bgs from each DU in triplicate. These samples will be collected to provide a background ISM surface soil dataset that can be compared to ISM surface soil samples collected during the Phase I RI and ISM samples that will be collected as part of the Phase III RI (outlined in Section 5.5), as well as ISM samples that are likely to be collected in future phases of the RI to evaluate ecological risks. ISM surface soil samples will be composed of 100 increments to mirror the increment density for Site ISM samples collected to date.

Surface Water and Sediment

One or two surface water and sediment sampling events will be conducted during the Phase III RI, based on available project funding: one during the early season mobilization and one during the subsequent mobilization (three surface water and three sediment samples per area per mobilization, 12 surface water and 12 sediment samples will be collected during the Phase III RI). Surface water and sediment background samples will be collected using the sampling protocol outlined in **Section 5.6**.

Groundwater and Subsurface Soil

Groundwater and subsurface soil samples will be collected using the sampling protocols outlined in **Section 5.7** and **Section 5.8**.

5.9.3 DQO 9 Background Study Field Measurements

Surface Soil

Field measurements for soil samples are consistent with the field measurements outlined in **Section 5.5**.

Surface Water and Sediment

Field measurements for surface water and sediment samples are consistent with the field measurements outlined in **Section 5.6**.

Groundwater

Field measurements for groundwater and subsurface soil samples are consistent with the field measurements outlined in **Section 5.7** and **Section 5.8, respectively**.

5.9.4 DQO 9 Background Study Analytical Measurements/Methods

Surface and subsurface soils will be analyzed for the following analytes:

- TAL metals (USEPA methods 6020B, 1631E)
- PAHs (USEPA method 8270-SIM)

Surface water and sediment samples will be analyzed for the following analytes:

- Total (unfiltered) and dissolved (filtered; surface water) TAL Metals (USEPA Methods 6020B and 1631E)
- PAHs (USEPA Method 8270-SIM)
- Hardness (by calculation), surface water only
- Copper Biotic Ligand Model input parameters (one sample per area per mobilization), surface water only:
  - DOC (USEPA Method 9060A-modified)
  - Sulfate and chloride (USEPA Method 300.0)
  - Alkalinity (SM 2320B)
  - Sulfide (SM 4500S D)
- TOC (USEPA Method 9060 modified), sediment only

Groundwater samples will be analyzed for the following analytes:

- Total (unfiltered) TAL Metals (USEPA Methods 6020B and 1631E)
- SVOCs (USEPA method 8270 and 8270-SIM)

6 DATA MANAGEMENT

This section describes how data are managed from the point when it is generated through use and storage. Data generated during this project will be captured electronically or entered by hand into bound field or laboratory logbooks or preprinted forms (Appendix B). Computer generated laboratory data will be managed using the laboratory information management system (LIMS); the LIMS used by subcontracted laboratories are described in their QA/QC documentation.

All project data, communications, and other information will be stored in a format useable to project personnel by VHB for at least 10 years following the investigation. Analytical data provided by the laboratory will be uploaded to EquIS. The Cleanup Lead will maintain and manage hard copies and/or electronic copies of all project related documents. Electronic copies of all information relating to this project are maintained on the VHB project network files, which are backed up once per day. ArcGIS Pro Packages (project files, maps, data, and supplementary elements used) and field and laboratory data (sample location parameters, sample collection methods, field measurement results, laboratory results [including standard laboratory QA/QC data], and, if applicable, the results of laboratory data validation) will be supplied to NPS. Additional information regarding project data management is available in **Tables 8 through 10**.

The list of planned samples (available in **Tables 1A through 2E**) will be compared to the list of samples collected each day to confirm nomenclature accuracy. Completed forms, logbooks, photographs, data packages, and electronic files will be transmitted regularly to the Cleanup Lead. The Cleanup Lead will monitor the progress of sample collection to verify that samples are collected as planned. Periodic desktop and/or telephone audits of field operations will be performed to check that data collection, documentation, and QA/QC procedures are being followed. Data entries created from hand-written notes will be QA checked by another person for accuracy.

The laboratory must maintain and adhere to a formal Quality Management Plan that addresses all data generating aspects of daily operations. All data generation processes will be reviewed and modified as needed to meet project objectives. Laboratory data are managed by the laboratory LIMS beginning with the sample receiving process. The laboratory is required to provide data reports (sample results, QC summary information, and supporting raw data) including electronic data deliverables within the turnaround times specified in **Table 5**. The laboratory will maintain copies of all documents it generates as well as backup files of all electronic data relating to the analysis of samples. All project data will be made available to NPS at the end of the RI.

7 ASSESSMENT AND OVERSIGHT

This section describes the measures that will be employed to ensure that this SAP is implemented properly.

7.1 ASSESSMENT AND CORRECTIVE ACTIONS

The QA Officer and VHB staff who are responsible for data control are specified in the QAPP (**Table 3**). VHB personnel and subcontractors will report to the Cleanup Lead. The Cleanup Lead is responsible for performing or coordinating the quality reviews specified in the QAPP. The Cleanup Lead is also responsible for ensuring that corrective actions are implemented if QA problems are identified during quality reviews.

7.1.1 Field Audit and Response Actions

A desktop (i.e., not in-field) field sampling audit will be performed during the first week of sampling by the Cleanup Lead. The field sampling audit will include the following:

- A brief interview with the field supervisor and/or field staff to assess adherence with SAP requirements.
- A review of all field forms related to the sampling that was performed.
- A review of the COC forms that were submitted with samples.

Results of the field sampling audit are intended to confirm that proper protocols are being followed, or to make improvements to the systems that are in-place. Audit findings will be communicated to the sampling team during the audit if immediate changes are necessary.

7.1.2 Laboratory Audit and Response Actions

An in-person laboratory audit will not be conducted for this project. The data validator will thoroughly review the laboratory SOPs and contact the laboratory prior to implementation of field investigation activities as needed to discuss analytical methods to support the integrity of analytical results.

7.2 QUALITY ASSESSMENT REPORTING

The Field Team Leader will be responsible for preparing daily reports documenting field activities, which will be reviewed by the Contractor Project Manager before being sent to NPS. The Laboratory Project Manager will be responsible for sending an e-mail acknowledging sample login to the Contractor Project Manager within a day of sample login. The Laboratory Project Manager will be responsible for producing

the laboratory data package with EDD to the Contractor Project Manager within the standard laboratory turnaround time after the last sample was received.

7.2.1 Data Verification

Table 9 lists data verification procedures that will be implemented during the investigation.

7.2.2 Data Validation

Table 10 lists data validation procedures that will be used to ensure the data meet the requirements detailed in **Section 4.6**.

7.3 RECONCILIATION WITH DQOS AND DATA USABILITY

The data usability assessment will be led by the Data Validator named in **Table 3**, using procedures described in **Table 10**. VHB and the subcontracted risk assessor will determine how to use data qualified by the data validator to evaluate the fate, transport, and potential risk of the affected contaminant.

8 INVESTIGATION OUTPUTS

This section describes the investigation outputs that this SAP supports. Following completion of all Phase III field work, a Phase III RI Technical Memorandum will be prepared and will include, as appropriate:

- Summaries of data for environmental media and comparison to field background levels and PALs in table and figure formats;
- The estimated extents of surface soil, subsurface soil, groundwater, surface water, and sediment concentrations above field background levels and/or PALs;
- Locations of groundwater seep discharges, springs, storm water run-off conveyances, and tailings deposits along National Creek;
- A summary of the findings;
- Recommendations and data gaps for future phases of the RI investigation; and
- Appendices containing all field data, soil boring and monitoring well logs, analytical reports, validation reports, statistical evaluations and other information collected during the Phase III RI work.

The Phase III RI Technical Memorandum will be submitted in draft, draft final, and final versions to NPS.

9 REFERENCES

Alaska Department of Environmental Conservation (ADEC). 2017. *Guidance on Developing Conceptual Site Models*. January.

ADEC. 2018. *Procedures for Calculating Cleanup Levels*. February 1.

ADEC. 2018. *Guidance for Evaluating Metals at Contaminated Sites*. August.

ADEC. 2022. *Field Sampling Guidance*. January.

ADEC. n.d.a. *Site Report: Powerhouse Fuel Tank Farm*. https://dec.alaska.gov/Applications/SPAR/PublicMVC/CSP/SiteReport/1307. Accessed January 28, 2021.

ADEC. n.d.b. *Site Report: NPS Kennicott Mine*. https://dec.alaska.gov/Applications/SPAR/PublicMVC/CSP/SiteReport/2440. Accessed January 28, 2021.

ADEC. n.d.c. *Site Report: NPS Kennicott Mine Monroe Tank 4(1)*. https://dec.alaska.gov/Applications/SPAR/PublicMVC/CSP/SiteReport/2441. Accessed January 28, 2021.

Alaska Department of Natural Resources. 2016a. *Water Well Log 31843*. August 18.

Alaska Department of Natural Resources. 2016b. *Water Well Log 31844*. August 18.

Amec Foster Wheeler Environment & Infrastructure, Inc. (Amec). 2018. *NPS Preliminary Assessment Report, Wrangell-St. Elias National Park and Preserve, Kennecott Mill Site, EDL# 5AKR3347*. March 29.

EMCON Alaska, Inc. (EMCON). 1992. *Site Investigation Final Report*. America North/EMCON, Inc. September.

Federal Emergency Management Agency (FEMA). 2023. *National Risk Index Copper River Census Area, Alaska*. https://hazards.fema.gov/nri/map. Accessed November 16, 2023.

Fish & Wildlife Service. 2004. *National Wetlands Inventory Mapper*. Retrieved November 7, 2023 from: https://www.fws.gov/wetlands/Data/Mapper.html.

Fish & Wildlife Service. N.d. *Listed Species With Spatial Current Range Believed to or Known to Occur In Alaska*. https://ecos.fws.gov/ecp/report/species-listings-by-state?stateAbbrev=AK&stateName=Alaska&statusCategory=Listed. Accessed November 7, 2023.

Geoengineers. 2006. Memorandum from Jamie Oakley of GeoEngineers to Jon A. Havelock, of Med-Tox Northwest, Subject: *Summary and Recommendations for Kennecott Mine Historical Hydrocarbon Releases*. April 4.

Gilbert, C., White, P., & Worthington, A. 2001. *Cultural landscape Report: Kennecott Mill Town*. National Park Service Wrangell-St. Elias National Park and Preserve, Denver, CO.

Hawaii Department of Health (HDOH). 2011. *Technical Guidance Manual*. https://health.hawaii.gov/heer/tgm/. Accessed January 30, 2024.

Hecht, B. & LaChapelle, E. 1999. *Hydrologic and Hydrogeologic Factors Affecting Aquifer Protection, McCarthy Area, Alaska*. Geotechnical Report for Alaska Department of Environmental Conservation. January.

Intergovernmental Data Quality Task Force (IDQTF). 2005. *Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs*. EPA/505/B-04/900A; DoD DTIC ADA 427785.

Interstate Technology Regulatory Council (ITRC). 2020. *Technical and Regulatory Guidance: Incremental Sampling Methodology Update*. October.

National Park Service (NPS). 1996. *Kennicott Pre-Acquisition Environmental Site Assessment*. December.

NPS. 2014. *Kennecott Operations Plan*.

NPS. 2018. *NPS Protocol for the Selection and Use of Ecological Screening Values for Non-Radiological Analytes*. Revision 2. November.

Trop, J.M., Benowitz, J.A, Kirby, C.S., Brueseke, M. 2021. *Geochronology of the Wrangell Arc: Spatial-Temporal Evolution of Slab-Edge Magmatism along a Flat-Slab, Subduction-Transform Transition, Alaska-Yukon*. Geosphere, 18(1): 19-48. December 22.

United States Climate Data. 2023. *Weather Averages McCarthy, Alaska*. www.usclimatedata.com/climate/mccarthy/alaska/united-states/usak0148. Accessed February 1, 2024.

United States Environmental Protection Agency (USEPA). 2001. *EPA Requirements for Quality Assurance Project Plans*. EPA/240/B-01/003. March.

USEPA. 2002. *Guidance for Quality Assurance Project Plans*. EPA/240/R-02/009. December.

USEPA. 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process*. EPA/240/B-06/001. February.

USEPA. 2021. *Guidance for Sample Collection for In Vitro Bioaccessibility Assay for Arsenic and Lead in Soil and Applications of Relative Bioavailability Data in Human Health Risk Assessment*. January 4.

USEPA. 2023. *Regional Screening Levels (RSLs)*. https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables. November.

USEPA. 2024. *Superfund Residential Lead Sites Handbook*. March.

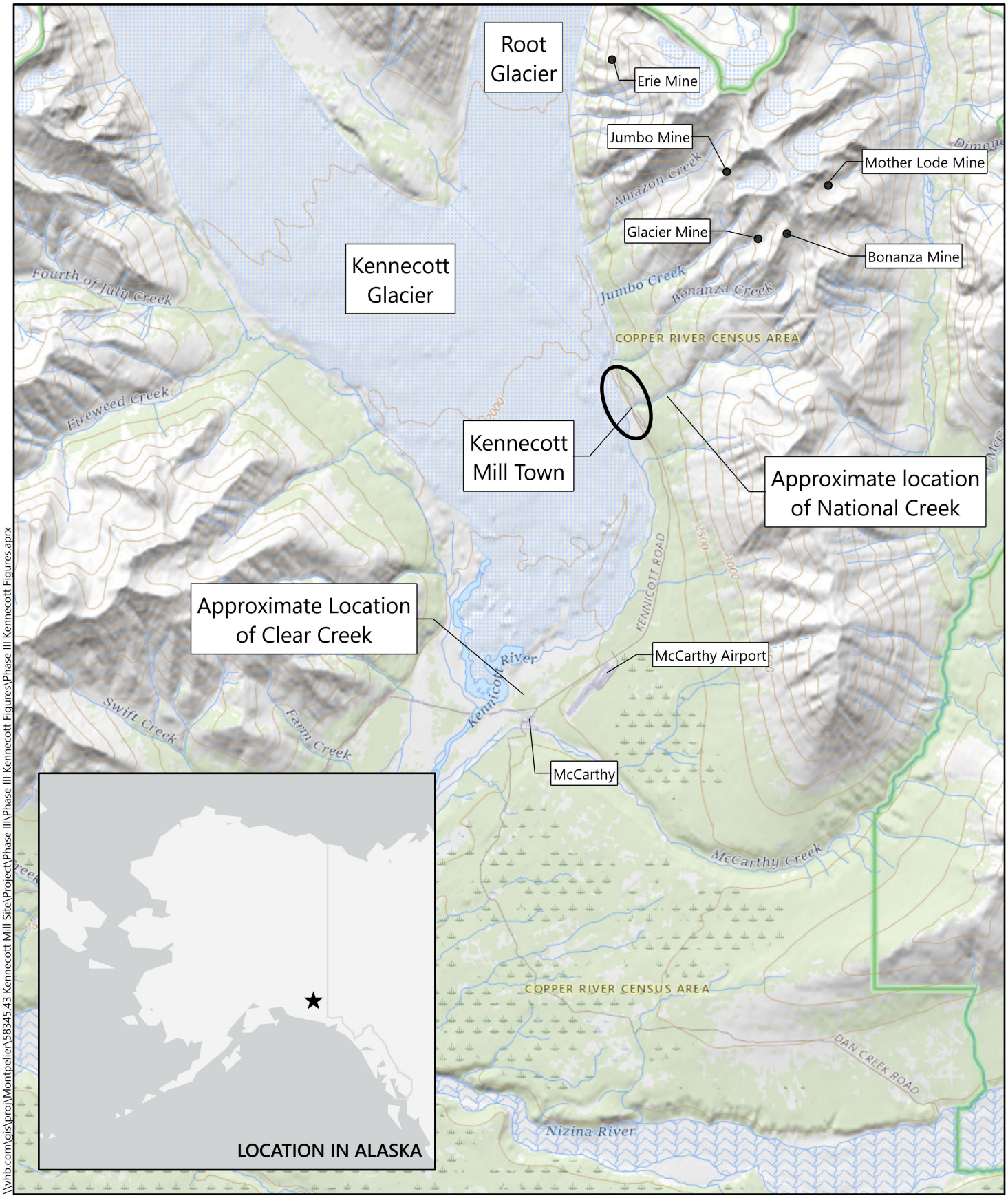
VHB. 2021. *Phase I Remedial Investigation Sampling and Analysis Plan*. Wrangell-St. Elias National Park and Preserve. Kennecott Mines and Mill Town Site. July 30.

Wilson, F.H., Hults, C.P., Mull, C.G, and Karl, S.M, compilers, 2015. *Geologic map of Alaska: U.S. Geological Survey Scientific Investigations Map 3340, pamphlet 196 p., 2 sheets, scale 1:1,584,000*. https://doi.org/10.3133/sim3340. Accessed November 28, 2023.

Winkler, G.R., MacKevett, E.M.,Jr., Plafker, George, Richter, D.H., Rosenkrans, D.S., and Schmoll, H.R. 2000. *A geologic guide to Wrangell–Saint Elias National Park and Preserve, Alaska—A tectonic collage of northbound terranes.* U.S. Geological Survey Professional Paper. 1616, 166 p.

Woodard & Curran (W&C). 2020. *Kennecott Mill Town Exposure Sampling Report*. May.

FIGURES



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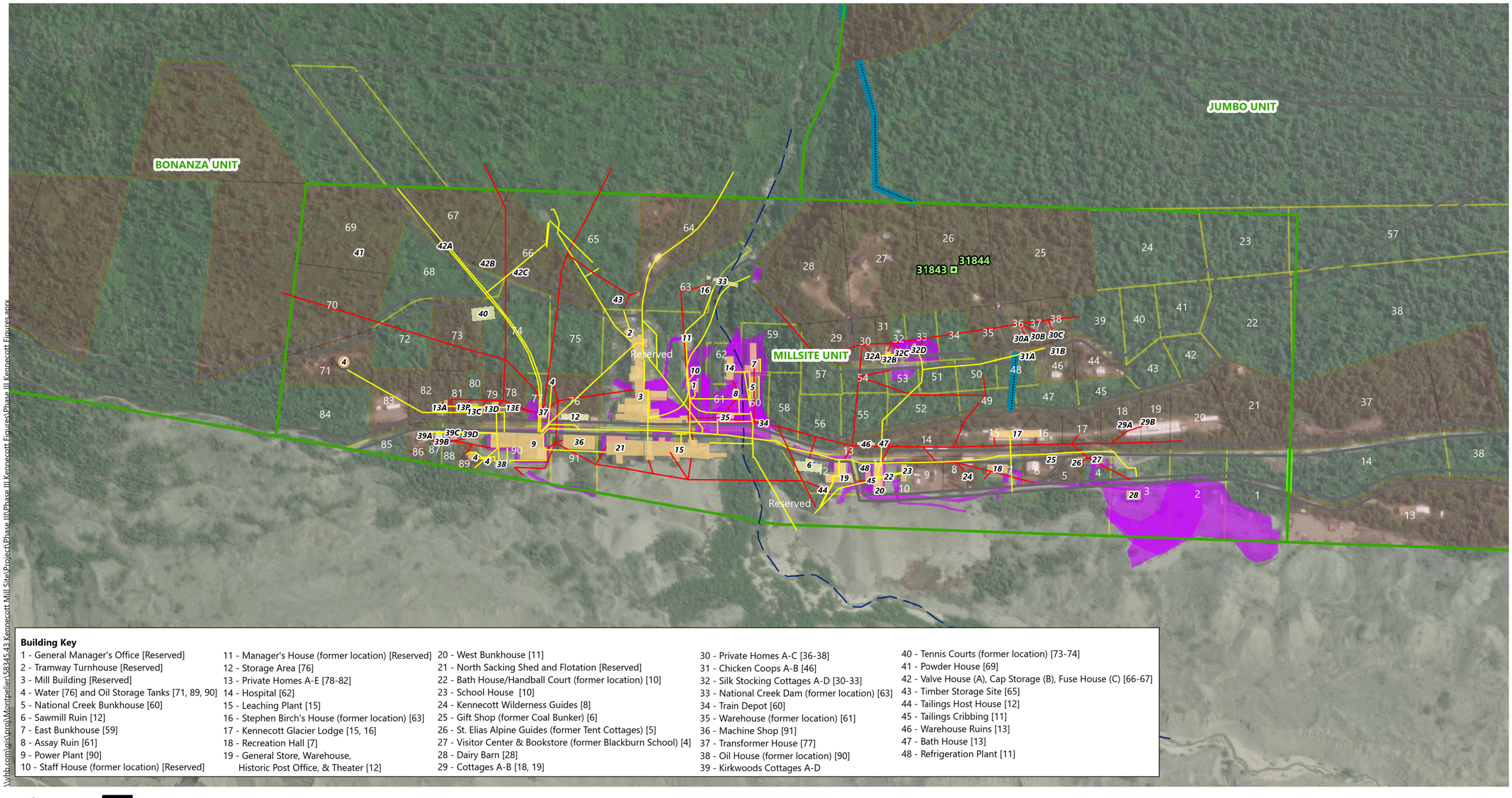
Kennecott Mines and Mill Town Site | Kennecott, AK

Site Locus

Mine Locations

Site Location Map

Sources: VHB, USGS, ESRI



Building Key

1 - General Manager's Office [Reserved]
2 - Tramway Turnhouse [Reserved]
3 - Mill Building [Reserved]
4 - Water [76] and Oil Storage Tanks [71, 89, 90]
5 - National Creek Bunkhouse [60]
6 - Sawmill Ruin [12]
7 - East Bunkhouse [59]
8 - Assay Ruin [61]
9 - Power Plant [90]
10 - Staff House (former location) [Reserved]
11 - Manager's House (former location) [Reserved]
12 - Storage Area [76]
13 - Private Homes A-E [78-82]
14 - Hospital [62]
15 - Leaching Plant [15]
16 - Stephen Birch's House (former location) [63]
17 - Kennecott Glacier Lodge [15, 16]
18 - Recreation Hall [7]
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22 - Bath House/Handball Court (former location) [10]
23 - School House [10]
24 - Kennecott Wilderness Guides [8]
25 - Gift Shop (former Coal Bunker) [6]
26 - St. Elias Alpine Guides (former Tent Cottages) [5]
27 - Visitor Center & Bookstore (former Blackburn School) [4]
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47 - Bath House [13]
48 - Refrigeration Plant [11]

0 100 200 400 Feet

- Building Footprint (See Building Key)
- Former Building Footprint

Land Ownership with Lot Number

- Federal
- Private
- National Creek
- Kennecott Subdivision Boundary (Bonanza, Jumbo, and Millsite Units)
- Underground Utility
- Aboveground Utility
- Disturbed Areas
- Water Supply Well

WRST Kennecott Easements

- Road
- Waterline (3 ft)
- Waterline (5 ft)

Kennecott Mines and Mill Town Site | Kennecott, AK

Site Layout and Land Ownership

Sources:
ESRI Basemap Imagery (2017)
National Park Service Geodatabase layers transmitted to VHB (2020).
NPS 1996 Pre-Acquisition Environmental Report, Figure 2.

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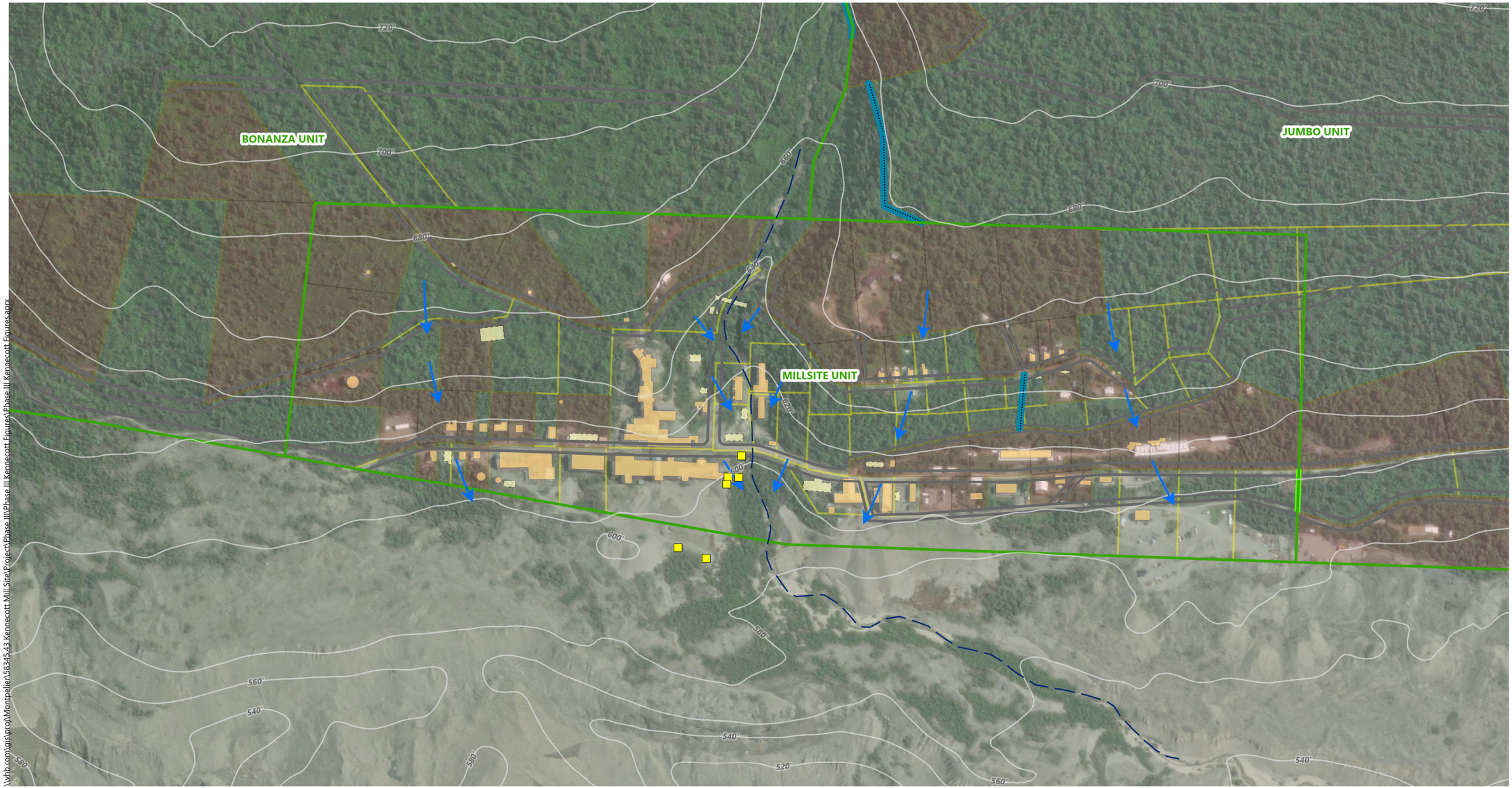

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0 100 200 400 Feet

Building Footprint (See Building Key)

Former Building Footprint

Land Ownership with Lot Number

Federal

Private

National Creek

Kennecott Subdivision Boundary **(Bonanza, Jumbo, and Millsite Units)**

Inferred Surface Water Runoff Pathway

Observed Seep Locations

Contour (20')

WRST Kennecott Easements

Road

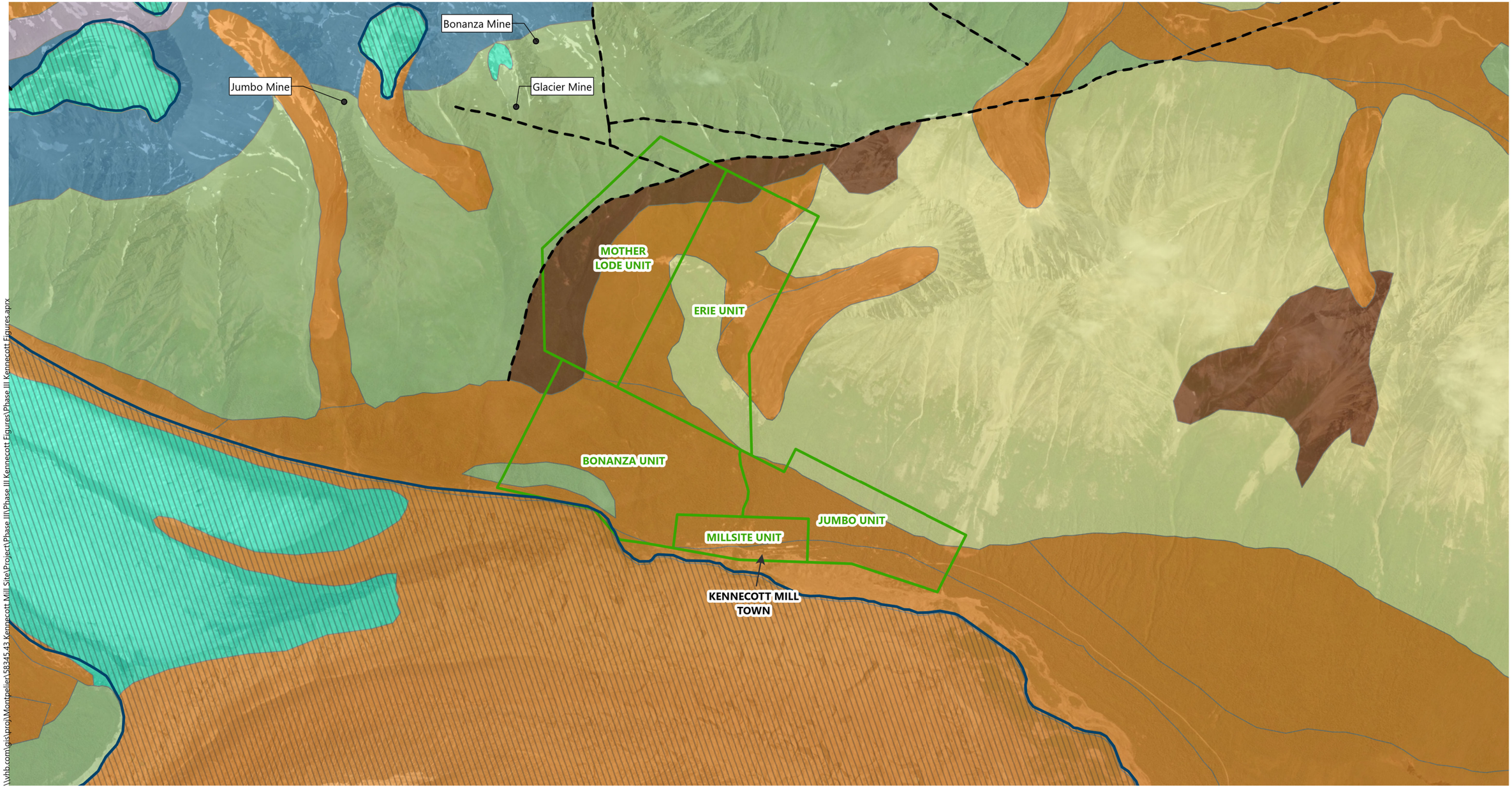
Waterline (3 ft)

Waterline (5 ft)

Kennecott Mines and Mill Town Site | Kennecott, AK

Topography, Drainage, and Surface Water Features

Sources:
ESRI Basemap Imagery (2017)
National Park Service Geodatabase layers transmitted to VHB (2020).
NPS 1996 Pre-Acquisition Environmental Report, Figure 2.



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0 0.5 1 2 Miles

- Kennecott Subdivision Boundary
- Glacier Extent (2013)
- USGS Fault, Approximate

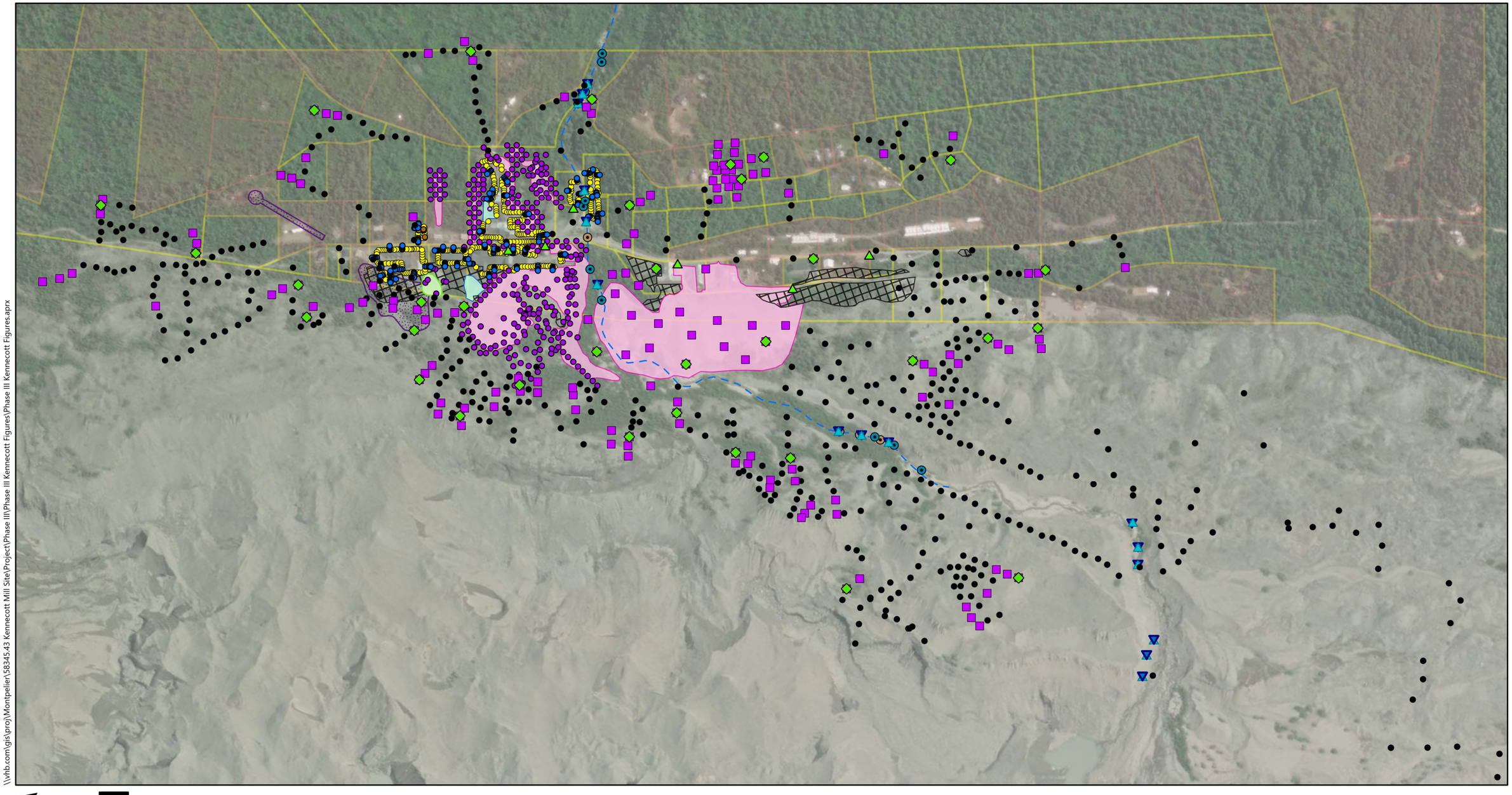
Geologic Unit (per USGS Map of Alaska)

- Chitistone and Nizina Limestones and Kamishak Formation
- Glaciers
- Matanuska Formation and correlative rocks
- McCarthy Formation
- Nikolai and Goon Dip Greenstones and equivalent rocks
- Unconsolidated surficial deposits, undivided
- Wrangell Lava
- Mine Locations

Kennecott Mines and Mill Town Site | Kennecott, AK

Bedrock Geologic Map

Sources:
ESRI Basemap Imagery (2017)
USGS Geologic Map of Alaska
USGS Watershed Boundary Dataset
NPS Randolf Glacier Inventory



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0 250 500 1000 US Feet

- Phase II Lab Analyzed Sample (Discrete)
- Phase II XRF Screening Location (Discrete)
- Phase II Ground Surface XRF Screening Location
- Phase II Sediment Sampling Locations
- Phase II Surface Water Sampling Locations
- Phase I Air Sampling Locations
- Phase I Surface Water/Sediment Sampling Locations
- Phase I Surface Water Sampling Locations
- Phase I XRF Screening Locations
- Phase 1 Soil Sampling Location - Mill Tailings
- Phase 1 Soil Sampling Location - Paint
- Phase 1 Soil Sampling Location - Transformer House
- National Creek

Land Ownership

- Federal Land
- Private Land

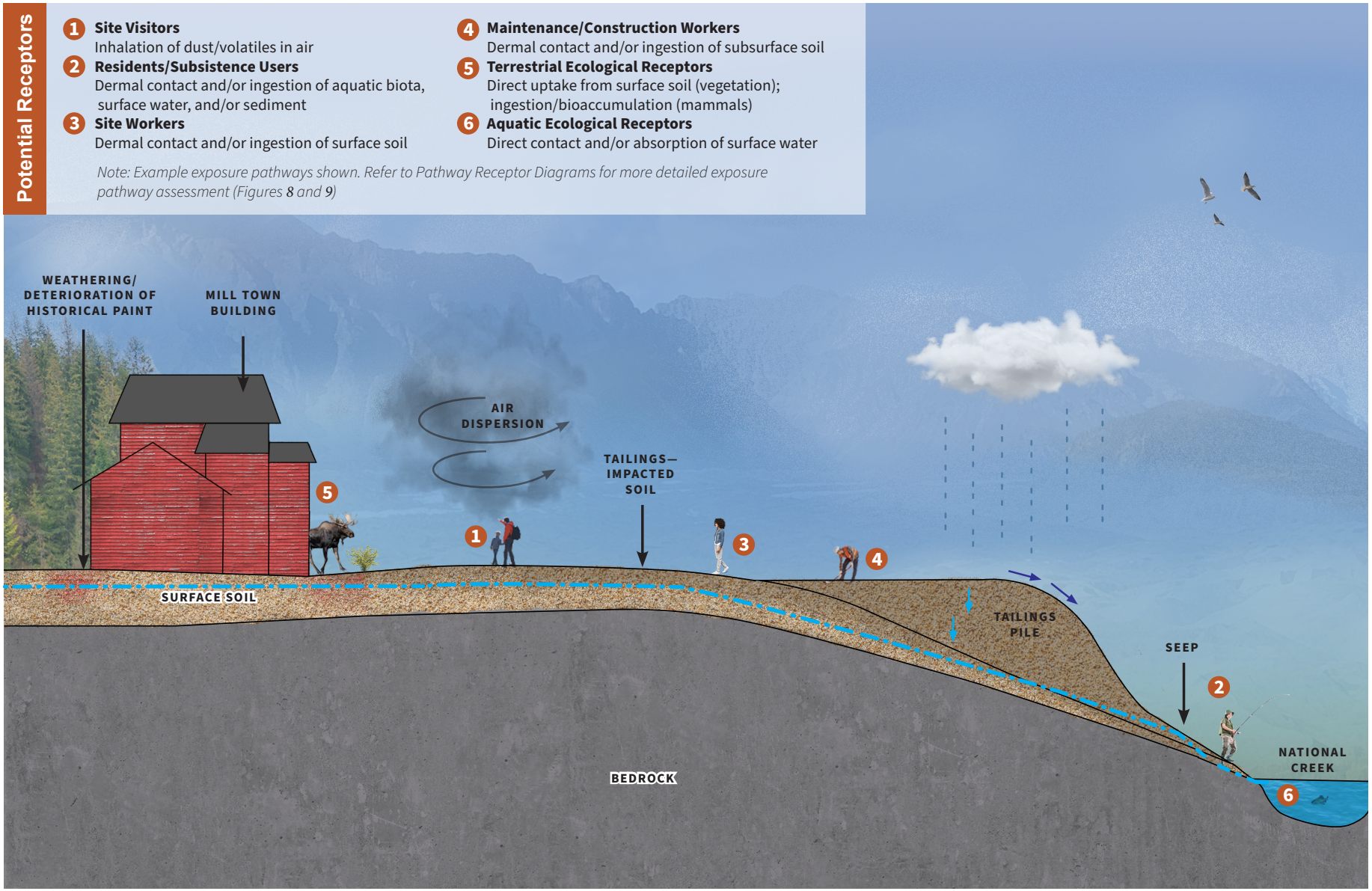
PA/SI Areas of Concern

- Boiler Ash Cap Location
- Dump Location
- Explosive Caps Location (Restricted Area Mapped)
- Fuel Spill Location
- Historically Mapped Tailings Pile Location

Kennecott Mines and Mill Town Site | Kennecott, AK

Phase I and II RI Sample Locations

Sources: VHB, USGS, ESRI

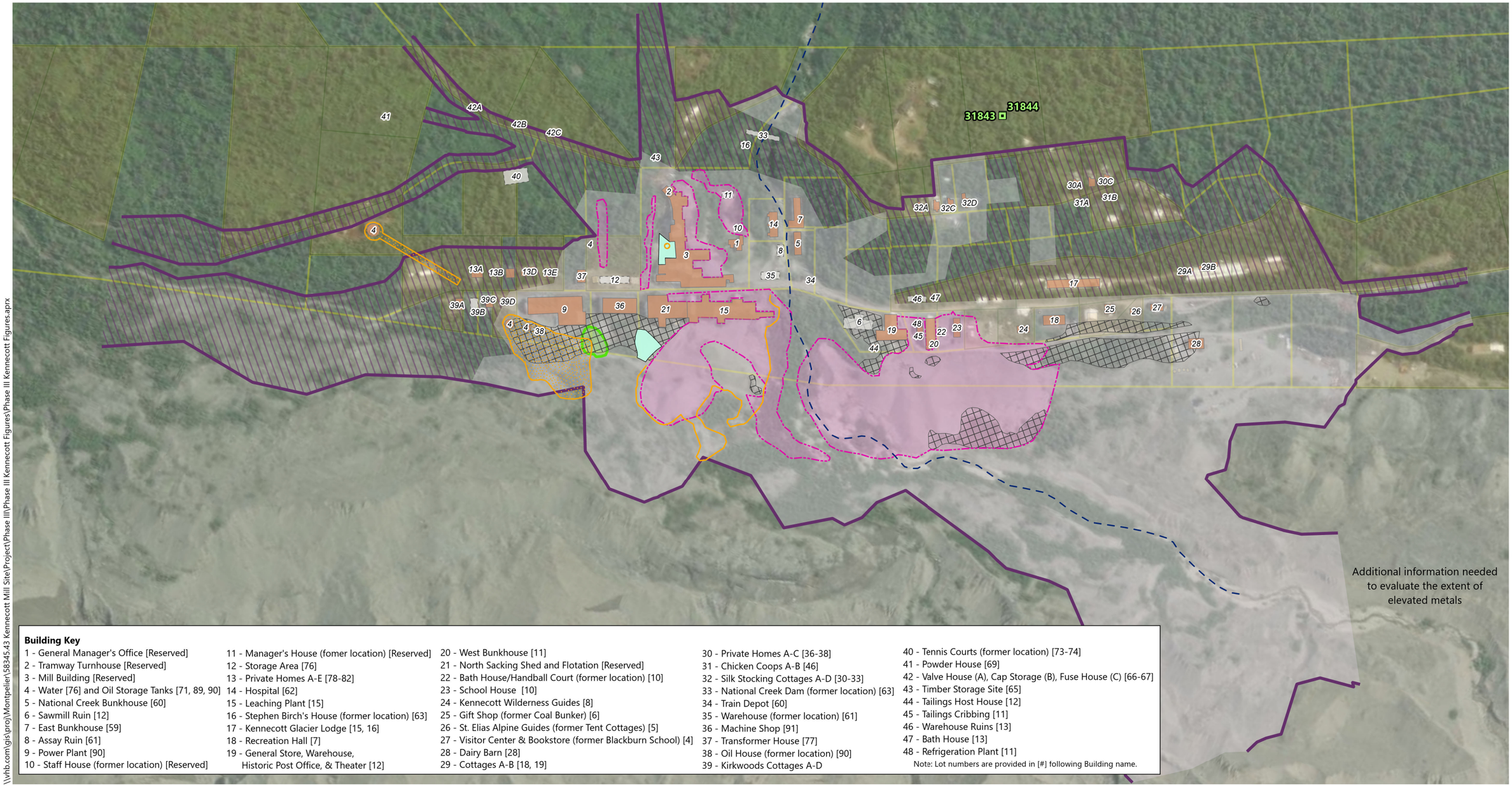


Legend

- **Runoff**
- **Infiltration/percolation**
- **Groundwater Table** (inferred)

Kennecott Mines and Mill Town Site | Kennecott, AK

Conceptual Site Model
Kennecott Mill Town



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47 - Bath House [13]
48 - Refrigeration Plant [11]

Note: Lot numbers are provided in [#] following Building name.

0 500 1000 2000 Feet

Land Ownership
- Federal
- Private

Approximate areas where metals were detected at concentrations greater than preliminary "field" background concentrations
- Elevated metals detected in surface soil during Phase I and/or II RI
- Elevated metals inferred to be present in surface soil based on conceptual understanding of metals distribution

PA Areas of Concern
- Boiler Ash Cap Location
- Dump Location
- Explosive Caps Location (Restricted Area Mapped)
- Fuel Spill Location
- Historically Mapped Tailings Pile Location
- Building Footprint (See Building Key)
- Former Building Footprint
- National Creek
- Observed Edge of Tailings (VHB, 08/2021)
- Water Supply Well

Kennecott Mines and Mill Town Site | Kennecott, AK

Mill Town Areas of Concern

Sources:
ESRI Basemap Imagery (2017)
National Park Service Geodatabase layers transmitted to VHB (2020).
AMEC 2017 Preliminary Assessment, Figure 4.

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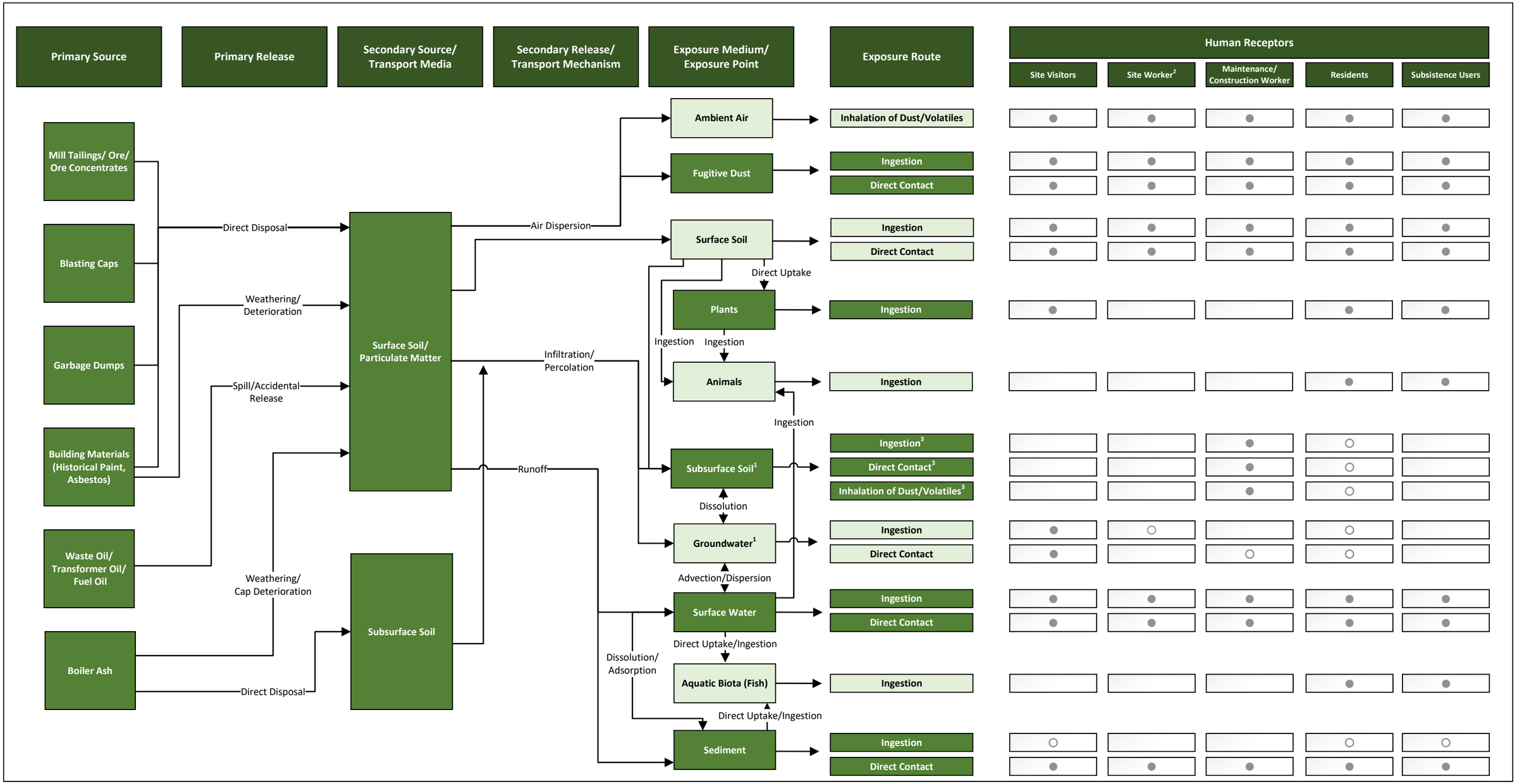


Potential Transport or Exposure Pathway

Potentially Complete Exposure Pathway
(Current and Future)

Complete Exposure Pathway
(Current and Future)

Notes:

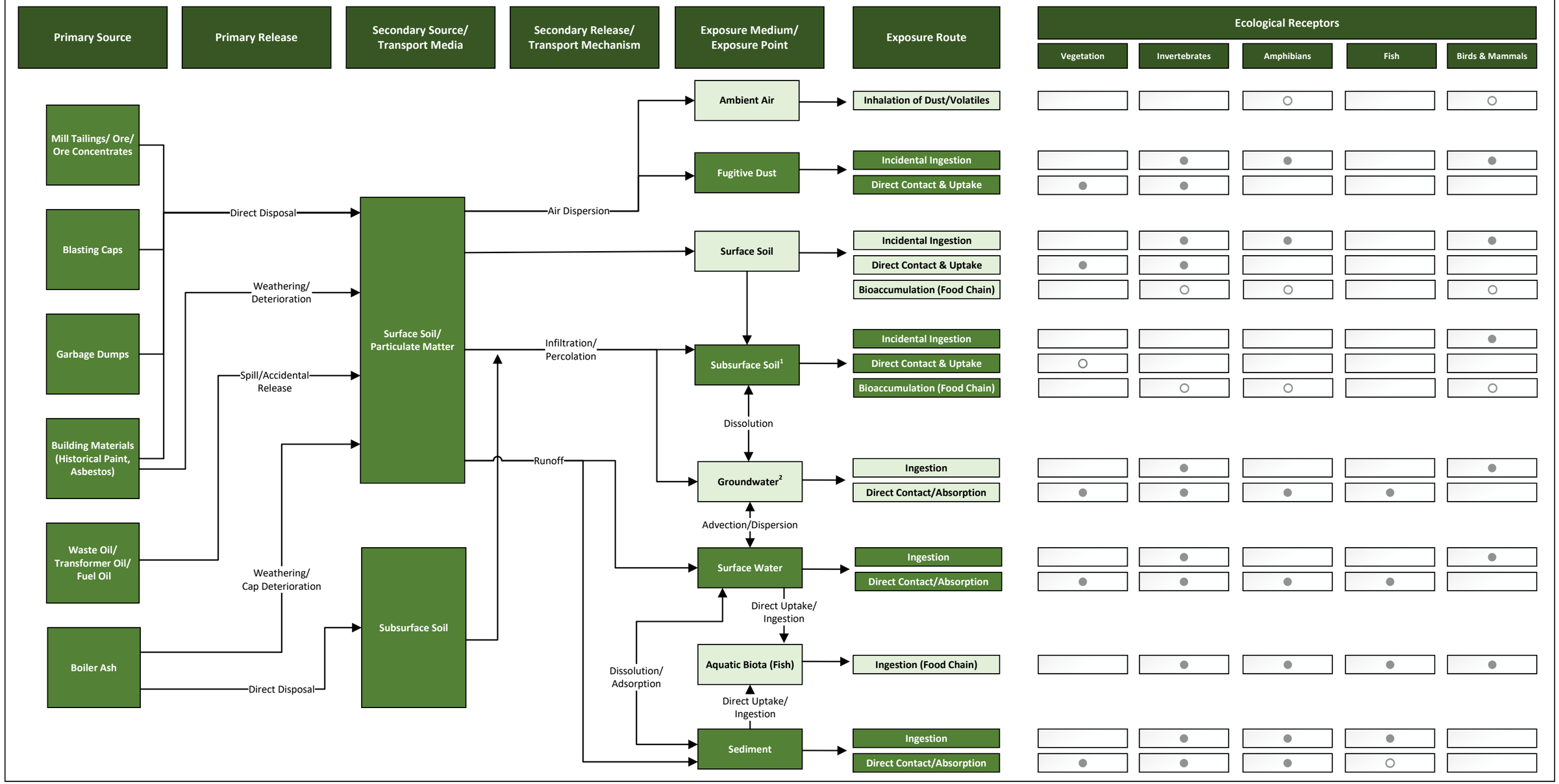
1. Subsurface soil and groundwater may be associated with complete exposure pathways in the future (e.g., if subsurface soils are exposed or if additional groundwater drinking wells are installed).

2. Site Workers may include Park staff (e.g., Park rangers, interpretive staff) and/or private concessions employees.

3. Residents may be exposed to subsurface soils through intrusive activities on private lands.

Kennecott Mines and Mill Town Site | Kennecott, AK

Preliminary Exposure Model for Human Receptors at the Mill Town Site



Potential Transport or Exposure Pathway

Potentially Complete Exposure Pathway
(Current and Future)

Complete Exposure Pathway
(Current and Future)

Notes:

1. Subsurface soil may be associated with complete exposure pathways in the future (e.g., if subsurface soils are exposed).

2. Groundwater will be evaluated as surface water at discharge points of identified seeps.

Kennecott Mines and Mill Town Site | Kennecott, AK

Preliminary Exposure Model for Ecological Receptors at the Mill Town Site

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0 50 100 200 US Feet

National Creek

Primary Land Owner

Federal Land

Private Land

Estimation Units

Kennecott Mines and Mill Town Site | Kennecott, AK

DQO 2/3 Surface Soil Sampling Locations

Sources: VHB, USGS, ESRI

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0 250 500 1000 Feet

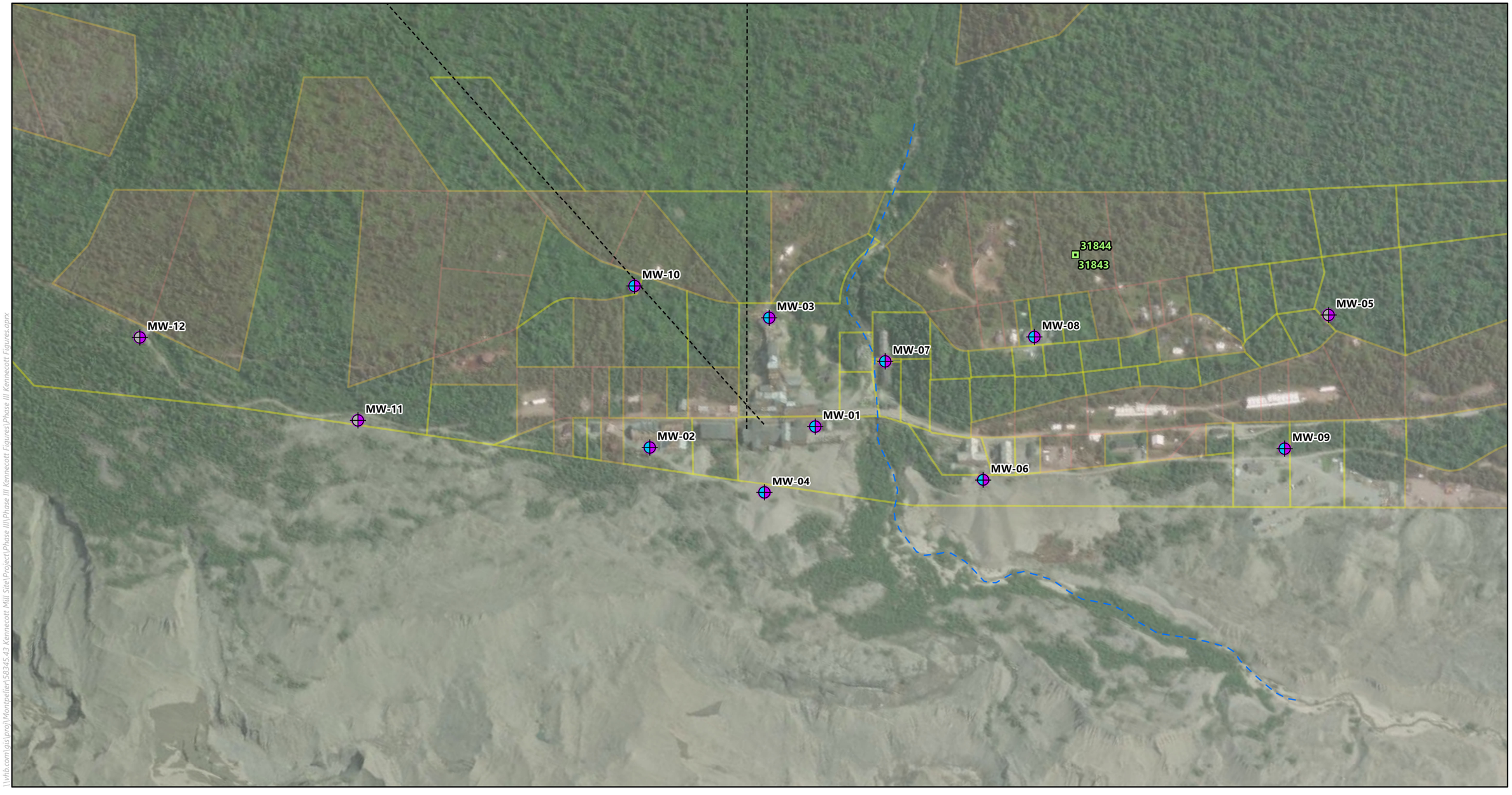
Primary Land Owner
Federal Land
Private Land

Proposed Surface Water and Sediment Samples
National Creek

Kennecott Mines and Mill Town Site | Kennecott, AK

DQO 6
Surface Water and Sediment Sampling Locations

Sources: ESRI, Maxar, VHB, NPS



0 500 1000 2000 US Feet

- Proposed Monitoring Well/Subsurface Soil
- Proposed Monitoring Well/Subsurface Soil (Background)
- Water Supply Well
- Aerial Tramway
- USGS Fault Lines
- National Creek
- Federal Land
- Private Land

Kennecott Mines and Mill Town Site | Kennecott, AK

DQO 7 & 8 Proposed Groundwater Monitoring Well and Subsurface Soil Sampling Locations

Sources: VHB, USGS, ESRI

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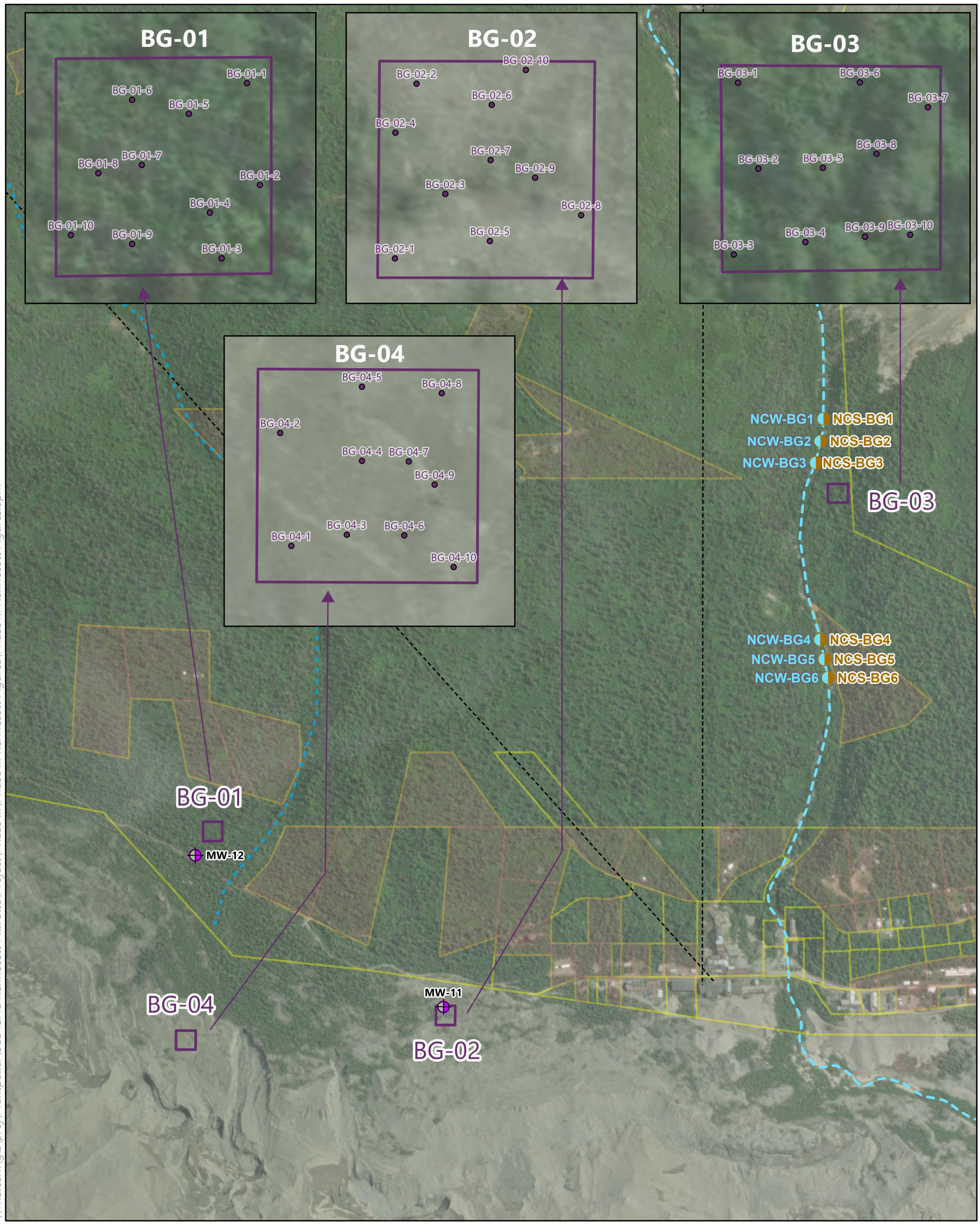

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0 0.13 0.25 0.5 Mile

Kennecott Mines and Mill Town Site | Kennecott, AK

DQO 9 Background Study Sampling Locations

Sources: VHB, USGS, ESRI

- Proposed Surface Water and Sediment Samples
- Proposed Monitoring Well (Background)
- Phase III Background ISM Points
- Phase III Background ISM DUs
- Aerial Tramway
- Bonanza Creek
- National Creek
- Federal Land
- Private Land

TABLES

TABLE 1A DQO 2/3 SURFACE SOIL SAMPLING LOCATIONS DETAILS

Location ID	Sample ID	Location	Reason for Sampling
32C-yard (87pprox.. 0.05-acre EU)	S-32Cyard-0-1A S-32Cyard-0-1B S-3C2yard-0-1C (replicates)	30 increment ISM sample collected from 0-1 inches bgs within the yard of Silk Stocking Cottage 32C	Residential soil may present a direct exposure pathway to persons working, playing, or conducting other recreational activities.0-1 inch interval selected as most relevant interval for human and ecological exposure and most likely to be impacted by COPCs (lead/arsenic-based paint, dust, aerial deposition).
32D-yard (87pprox.. 0.1-acre EU)	S-32Dyard-0-1A S-32Dyard-0-1B S-32Dyard-0-1C (replicates)	45 increment ISM sample collected from 0-1 inches bgs within the yard of Silk Stocking Cottage 32D	
53-yard (87pprox.. 0.05-acre EU)	S-53yard-0-1A S-53yard-0-1B S-53yard-0-1C (replicates)	30 increment ISM sample collected from 0-1 inches bgs within the Lot 53 yard	
32C-dripline (87pprox.. 0.006-acre EU)	S-32Cdrip-0-1A S-32Cdrip-0-1B S-32Cdrip-0-1C (replicates)	30 increment ISM sample collected from 0-1 inches bgs between the building exterior and dripline of Silk Stocking Cottage 32C	Deteriorating lead and arsenic paint from the exterior of buildings may accumulate in soils adjacent to buildings. Additionally, rooftops may collect fine-grained sediments that contain COPCs, which may wash off during storm events and accumulate in soil (USEPA, 2024). 0-1 inch interval selected as most relevant interval for human and ecological exposure and most likely to be impacted by COPCs (lead/arsenic-based paint, dust, aerial deposition).
32D-dripline (87pprox.. 0.006-acre EU)	S-32Ddrip-0-1A S-32Ddrip-0-1B S-32Ddrip-0-1C (replicates)	30 increment ISM sample collected from 0-1 inches bgs between the building exterior and dripline of Silk Stocking Cottage 32D	
32G-dripline (87pprox.. 0.004-acre EU)	S-32Gdrip-0-1A S-32Gdrip-0-1B S-32Gdrip-0-1C (replicates)	30 increment ISM sample collected from 0-1 inches bgs between the building exterior and dripline of Silk Stocking Cottage 32G	
53-garden (87pprox.. 0.01-acre EU)	S-53gard-0-12A S-53gard-0-12B S-53gard-0-12C (replicates)	30 increment ISM sample collected from 0-12 inches bgs within the Lot 53 garden	Garden soils may present a direct exposure pathway to gardening residents (USEPA, 2024). Soil mixing to 12 inches bgs is likely common and human exposure to soils between 0 and 12 inches is possible during gardening.
32C-yard (87pprox.. 0.05-acre EU)	S-32Cyard-0-1D	30 increment ISM sample collected from 0-1 inches bgs within the yard of Silk Stocking Cottage 32C	To assess variability of lead and arsenic IVBA results.
32D-yard (87pprox.. 0.1-acre EU)	S-32Dyard-0-1D	45 increment ISM sample collected from 0-1 inches bgs within the yard of Silk Stocking Cottage 32D	

Location ID	Sample ID	Location	Reason for Sampling
32C-dripline (88pprox.. 0.006-acre EU)	S-32Cdrip-0-1D	30 increment ISM sample collected from 0-1 inches bgs between the building exterior and dripline of Silk Stocking Cottage 32C	
32D-dripline (88pprox.. 0.006-acre EU)	S-32Ddrip-0-1D	30 increment ISM sample collected from 0-1 inches bgs between the building exterior and dripline of Silk Stocking Cottage 32D	
53-garden (88pprox.. 0.01-acre EU)	S-53gard-0-12D	30 increment ISM sample collected from 0-12 inches bgs within the Lot 53 garden	

TABLE 1B DQO 6 SURFACE WATER AND SEDIMENT SAMPLING LOCATIONS DETAILS

Location ID	Sample ID	Location	Reason for Sampling
NCW-U1 NCW/NCS-U2 NCW-U3	NCW-U1-MMDDYY NCW/NCS-U2-MMDDYY NCW-U3-MMDDYY	Upstream of the Mill buildings and main road	To evaluate whether COPCs from Kennecott Mill Town sources are impacting surface water/sediment quality and for comparison to Phase I and II RI results.
NCW-M1 NCW/NCS-M2 NCW-M3	NCW-M1-MMDDYY NCW-M2/NCS-M2-MMDDYY NCW-M3-MMDDYY	Within the vicinity of the Mill Town	To evaluate whether COPCs from Kennecott Mill Town sources are impacting surface water/sediment quality and for comparison to Phase I and II RI results.
NCW-D1 NCW/NCS-D2 NCW-D3 NCW/NCS-D4 NCW/NCS-D5 NCW/NCS-D6 NCW/NCS-D7 NCW/NCS-D8 NCW/NCS-D9	NCW-D1-MMDDYY NCW/NCS-D2-MMDDYY NCW-D3-MMDDYY NCW/NCS-D4-MMDDYY NCW/NCS-D5-MMDDYY NCW/NCS-D6-MMDDYY NCW/NCS-D7-MMDDYY NCW/NCS-D8-MMDDYY NCW/NCS-D9-MMDDYY	Downstream of the Mill Town and large tailings piles	To evaluate whether COPCs from Kennecott Mill Town sources are impacting surface water/sediment quality and for comparison to Phase I and II RI results.

TABLE 1C DQO 7 AND 8 GROUNDWATER AND SUBSURFACE SOIL SAMPLING LOCATIONS DETAILS

Location ID	Sample ID	Location	Reason for Sampling
MW/SB-01	MW-01-MMDDYY/ SB-01-*depth*	West side of Kennecott Road near the Leaching Plant	Tailings observed in this location. Centralized location within Mill Town.
MW/SB-02	MW-02-MMDDYY/ SB-02-*depth*	Behind (west side) the Kennecott Power Plant	Topographically downslope of the Power Plant (likely hydraulically downgradient).
MW/SB-03	MW-03-MMDDYY/ SB-03-*depth*	Behind (east side) the Mill Building	Former operational area, location of former aerial tramway terminus. Tailings observed in this location.
MW/SB-04	MW-04-MMDDYY/ SB-04-*depth*	Within the northern tailings pile to the west of the North Sacking Shed and Flotation Building.	Historical tailings pile. Potential for metals impacts to groundwater/leaching at this location. Topographically downslope groundwater sampling location (likely hydraulically downgradient of former operations area).
MW/SB-05	MW-05-MMDDYY/ SB-05-*depth*	South of the Site in the forested area behind the lodge.	Area suspected to be unimpacted by historical Site activities. Potential for use in DQO 9.
MW/SB-06	MW-06-MMDDYY/ SB-06-*depth*	Within the southern tailings pile to the west of the General Store.	Historical tailings pile. Potential for metals impacts to groundwater/leaching at this location.
MW/SB-07	MW-07-MMDDYY/ SB-07-*depth*	Between the National Creek Bunkhouse and East Bunkhouse.	Represents groundwater conditions adjacent to National Creek. Known surface soil metals impacts from historical paint. Potential for leaching at this location.
MW/SB-08	MW-08-MMDDYY/ SB-08-*depth*	Between Silk Stocking Cottages 32C and 32D.	Known surface soil COPC impacts. Topographically upslope groundwater sampling location (likely hydraulically upgradient).
MW/SB-09	MW-09-MMDDYY/ SB-09-*depth*	Near the southwestern corner of the Recreation Hall.	Adjacent to known historical tailings pile and road. Potential for leaching at this location.
MW/SB-10	MW-10-MMDDYY/ SB-10-*depth*	Northeast of the Site in the forested area. Located near the historic tramline.	Upgradient of the Mill Town; located near the historic tramline. Provides spatial coverage in northeast portion of the Mill Town.
MW/SB-11	MW-11-MMDDYY/ SB-11-*depth*	Northwest of the Site in the moraine area.	Area suspected to be unimpacted by historical Site activities. Potential for use in DQO 9.
MW/SB-12	MW-12-MMDDYY/ SB-12-*depth*	Located within background soil DU (DQO 9) north of the Mill Town.	Area suspected to be unimpacted by historical Site activities. Potential for use in DQO 9.

TABLE 1D DQO 9 BACKGROUND STUDY SAMPLING LOCATIONS DETAILS

Location ID	Sample ID	Location	Reason for Sampling
Soil			
BG-01	ISM Replicates: BG-01A BG-01B BG-01C Discrete Samples: BG-01-1 BG-01-2 BG-01-3 BG-01-4 BG-01-5 BG-01-6 BG-01-7 BG-01-8 BG-01-9 BG-01-10	Background/reference area north of the Site and east of main road in forested area, see **Figure 13**. 100-increment ISM samples collected from 0 to 6 inches bgs in triplicate. Discrete samples collected from 0 to 6 inches bgs within ISM DU boundary.	Forested area on federal land that may not be impacted by historical Site operations. Geomorphologic environment may be comparable to the eastern forested portion of the Mill Town.
BG-02	ISM Replicates: BG-02A BG-02B BG-02C Discrete Samples: BG-02-1 BG-02-2 BG-02-3 BG-02-4 BG-02-5 BG-02-6 BG-02-7 BG-02-8 BG-02-9 BG-02-10	Background/reference area north of the Site and west of main road in moraine area, see **Figure 13.** 100-increment ISM samples collected from 0 to 6 inches bgs in triplicate. Discrete samples collected from 0 to 6 inches bgs within ISM DU boundary.	Moraine area on federal land that may not be impacted by historical Site operations. Geomorphologic environment may be comparable to the western moraine-facing portion of the Mill Town.

Location ID	Sample ID	Location	Reason for Sampling
BG-03	ISM Replicates: BG-03A BG-03B BG-03C Discrete Samples: BG-3-1 BG-3-2 BG-3-3 BG-3-4 BG-3-5 BG-3-6 BG-3-7 BG-3-8 BG-3-9 BG-3-10	Background/reference area east of the Site near National Creek in the forested area, see **Figure 13**. 100-increment ISM samples collected from 0 to 6 inches bgs in triplicate. Discrete samples collected from 0 to 6 inches bgs within ISM DU boundary.	Forested area on federal land that may not be impacted by historical Site operations. Geomorphologic environment that may be comparable to the eastern forested portion of the Mill Town.
BG-04	ISM Replicates: BG-04A BG-04B BG-04C Discrete Samples: BG-04-1 BG-04-2 BG-04-3 BG-04-4 BG-04-5 BG-04-6 BG-04-7 BG-04-8 BG-04-9 BG-04-10	Background/reference area north of the Site and west of main road in moraine area, see **Figure 13.** 100-increment ISM samples collected from 0 to 6 inches bgs in triplicate. Discrete samples collected from 0 to 6 inches bgs within ISM DU boundary.	Moraine area on federal land that may not be impacted by historical Site operations. Geomorphologic environment that may be comparable to the western moraine-facing portion of the Mill Town.
Surface Water & Sediment			
BG1/BG2/BG3	NCW/NCS-BG1 NCW/NCS-BG2 NCW/NCS-BG3	Furthest upstream sampling area	To provide background surface water and sediment quality for comparison to downstream locations and to Phase I and II RI results.
BG4/BG5/BG6	NCW/NCS-BG4 NCW/NCS-BG5 NCW/NCS-BG6	Between the furthest upstream background sampling area and the upstream site sampling area	To provide background surface water and sediment quality for comparison to downstream locations and to Phase I and II RI results.

Location ID	Sample ID	Location	Reason for Sampling
Groundwater & Subsurface Soil			
See **Table 1C** for proposed background monitoring well/soil boring locations.			

TABLE 2A DQO 2/3 SAMPLE TYPES, VOLUMES AND TOTAL NUMBER OF CONTAINERS

SAMPLE ID	MATRIX	DEPTH (units)	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	IVBA
S-32Cdrip-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x[1]	
S-32Cdrip-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cdrip-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cdrip-0-1D	Soil	0-1 in bgs	ISM	1 kilogram			1
S-32Ddrip-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Ddrip-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Ddrip-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Ddrip-0-1D	Soil	0-1 in bgs	ISM	1 kilogram			1
S-32Gdrip-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Gdrip-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Gdrip-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cyard-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cyard-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cyard-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Cyard-0-1D	Soil	0-1 in bgs	ISM	1 kilogram			1
S-32Dyard-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Dyard-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Dyard-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-32Dyard-0-1D	Soil	0-1 in bgs	ISM	1 kilogram			1
S-53yard-0-1A	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-53yard-0-1B	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-53yard-0-1C	Soil	0-1 in bgs	ISM	1 kilogram	1	x	
S-53gard-0-12A	Soil	0-12 in bgs	ISM	1 kilogram	1	x	
S-53gard-0-12B	Soil	0-12 in bgs	ISM	1 kilogram	1	x	
S-53gard-0-12C	Soil	0-12 in bgs	ISM	1 kilogram	1	x	
S-32Cdrip-0-1B-MS	Soil	0-1 in bgs	ISM	*Take from parent*	x	x	
S-32Cdrip-0-1B-MSD	Soil	0-1 in bgs	ISM	*Take from parent*	x	x	
Total number of solid containers:					21	0	4
EB-S-MMDDYY	Water (lab-provided)	-	EB	250 mL	1	2	
Total number of aqueous containers:					1	2	0

[1]An “x” signifies a sample analysis without a dedicated sample container.

TABLE 2B DQO 6 SAMPLE TYPES, VOLUMES AND TOTAL NUMBER OF CONTAINERS

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	VOCs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
NCW-U1-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x[1]					
NCW-U1-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-U2-MMDDYY	Surface Water	Grab	1.75 L	2	2	3	x		1	1	1	
NCW-U2-MMDDYY-F	Surface Water	Grab	750 mL	2				1				
NCW-U3-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-U3-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-M1-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-M1-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-M2-MMDDYY	Surface Water	Grab	1.75 L	2	2	3	x		1	1	1	
NCW-M2-MMDDYY-F	Surface Water	Grab	750 mL	2				1				
NCW-M3-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-M3-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-D1-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D1-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-D2-MMDDYY	Surface Water	Grab	1.75 L	2	2	3	x		1	1	1	
NCW-D2-MMDDYY-F	Surface Water	Grab	750 mL	2				1				
NCW-D3-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D3-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-D4-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D4-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-D5-MMDDYY	Surface Water	Grab	1.75 L	2	2	3	x		1	1	1	
NCW-D5-MMDDYY-F	Surface Water	Grab	750 mL	2				1				
NCW-D6-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D6-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-D7-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D7-MMDDYY-F	Surface Water	Grab	750 mL	2								

[1] An “x” signifies a sample analysis without a dedicated sample container.

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	VOCs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
NCW-D8-MMDDYY	Surface Water	Grab	1.75 L	2	2	3	x		1	1	1	
NCW-D8-MMDDYY-F	Surface Water	Grab	750 mL	2				1				
NCW-D9-MMDDYY	Surface Water	Grab	1.12 L	2	2	3	x					
NCW-D9-MMDDYY-F	Surface Water	Grab	750 mL	2								
NCW-L1-MMDDYY	Surface Water	Duplicate	1.75 L	2	2	3	x		1	1	1	
NCW-L1-MMDDYY-F	Surface Water	Duplicate	750 mL	2				1				
NCW-L2-MMDDYY	Surface Water	Duplicate	1.12 L	2	2	3	x					
NCW-L2-MMDDYY-F	Surface Water	Duplicate	750 mL	2								
NCW-XX-MMDDYY-MS	Surface Water	MS	1.75 L	2	2	3	x		1	1	1	
NCW-XX-MMDDYY-MS-F	Surface Water	MS	750 mL	2				1				
NCW-XX-MMDDYY-MSD	Surface Water	MSD	1.75 L	2	2	3	x		1	1	1	
NCW-XX-MMDDYY-MSD-F	Surface Water	MSD	750 mL	2				1				
EB-NCW-MMDDYY	Water (lab-provided)	EB	1.12 L	2	2	3	x					
EB-NCW-MMDDYY-F	Water (lab-provided)	EB	500 mL	2								
TB-01-MMDDYY	Water (lab-provided)	Surface Water Trip Blank	80 mL			2						
TB-02-MMDDYY	Water (lab-provided)	Surface Water Trip Blank	80 mL			2						
EB-NCS-MMDDYY	Water (lab-provided)	EB	1.12 L	2	2	3						
TB-03-MMDDYY	Methanol (lab-provided)	Sediment Trip Blank	80 mL			2						
TB-04-MMDDYY	Methanol (lab-provided)	Sediment Trip Blank	80 mL			2						
Total number of aqueous containers (per sampling event)				82	42	71	0	8	8	8	8	0
Total number of aqueous containers (for Phase III RI)				164	84	213	0	16	16	16	16	0
NCS-U2-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-M2-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D2-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D4-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D5-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D6-MMDDYY	Sediment	Grab	12 oz	1	x	1						x

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	VOCs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
NCS-D7-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D8-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-D9-MMDDYY	Sediment	Grab	12 oz	1	x	1						x
NCS-L1-MMDDYY	Sediment	Duplicate	12 oz	1	x	1						x
NCS-XX-MMDDYY-MS	Sediment	MS	*Take from parent*	x	x	x						
NCS-XX-MMDDYY-MSD	Sediment	MSD	*Take from parent*	x	x	x						
Total number of solid containers:				10	0	10	0	0	0	0	0	0

TABLE 2C DQO 7 SAMPLE TYPES, VOLUMES AND TOTAL NUMBER OF CONTAINERS

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	SVOCs	VOCs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide
MW-01-MMDDYY	Groundwater	Grab	3.75 L	2	2	2	3	x[1]		1	1	1
MW-01-MMDDYY-F	Groundwater	Grab	750 mL	2					1			
MW-02-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-02-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-03-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-03-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-04-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-04-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-05-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-05-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-06-MMDDYY	Groundwater	Grab	3.75 L	2	2	2	3	x		1	1	1
MW-06-MMDDYY-F	Groundwater	Grab	750 mL	2					1			
MW-07-MMDDYY	Groundwater	Grab	3.75 L	2	2	2	3	x		1	1	1
MW-07-MMDDYY-F	Groundwater	Grab	750 mL	2					1			
MW-08-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-08-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-09-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-09-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-10-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-10-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-11-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-11-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-12-MMDDYY	Groundwater	Grab	3.12 L	2	2	2	3	x				
MW-12-MMDDYY-F	Groundwater	Grab	750 mL	2								
MW-50-MMDDYY	Groundwater	Duplicate	3.75 L	2	2	2	3	x		1	1	1
MW-50-MMDDYY-F	Groundwater	Duplicate	750 mL	2					1			

[1] An “x” signifies a sample analysis without a dedicated sample container.

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	SVOCs	VOCs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide
MW-51-MMDDYY	Groundwater	Duplicate	3.12 L	2	2	2	3	x				
MW-51-MMDDYY-F	Groundwater	Duplicate	750 mL	2								
MW-01-MMDDYY-MS	Groundwater	MS	3.75 L	2	2	2	3	x		1	1	1
MW-01-MMDDYY-F-MS	Groundwater	MS	750 mL	2					1			
MW-01-MMDDYY-MSD	Groundwater	MSD	3.75 L	2	2	2	3	x		1	1	1
MW-01-MMDDYY-F-MSD	Groundwater	MSD	750 mL	2					1			
EB-GW-MMDDYY	Water (lab-provided)	EB	3.12 L	2	2	2	3	x				
DW-XX-MMDDYY	Drilling Water	Grab	3.12 L	2	2	2	3					
TB-05-MMDDYY	Water (lab-provided)	Trip Blank	80 mL				2					
TB-06-MMDDYY	Water (lab-provided)	Trip Blank	80 mL				2					
Total number of aqueous containers (to samples all wells – additional containers needed to sample wells installed during first mobilization, if more than one mobilization occurs)				68	36	36	58	0	6	6	6	6

TABLE 2D DQO 8 SAMPLE TYPES, VOLUMES AND TOTAL NUMBER OF CONTAINERS

SAMPLE ID	MATRIX	DEPTH	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	SVOCs	VOCs
SB-01-depth	Soil	TBD	Grab	12 oz	1	x[1]	x	1
SB-01-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-01-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-01-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-01-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-02-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-02-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-02-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-02-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-02-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-03-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-03-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-03-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-03-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-03-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-04-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-04-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-04-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-04-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-04-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-05-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-05-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-05-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-05-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-05-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-06-depth	Soil	TBD	Grab	12 oz	1	x	x	1

[1] An “x” signifies a sample analysis without a dedicated sample container.

SAMPLE ID	MATRIX	DEPTH	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	SVOCs	VOCs
SB-06-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-06-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-06-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-06-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-07-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-07-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-07-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-07-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-07-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-08-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-08-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-08-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-08-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-08-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-09-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-09-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-09-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-09-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-09-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-10-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-10-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-10-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-10-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-10-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-11-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-11-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-11-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-11-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-11-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-12-depth	Soil	TBD	Grab	12 oz	1	x	x	1

SAMPLE ID	MATRIX	DEPTH	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	SVOCs	VOCs
SB-12-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-12-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-12-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-12-depth	Soil	TBD	Grab	12 oz	1	x	x	1
SB-50-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-51-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-52-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-53-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-54-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-55-depth	Soil	TBD	Duplicate	12 oz	1	x	x	1
SB-XX-depth-MS	Soil	TBD	MS	4 oz	x	x	x	1
SB-XX-depth-MSD	Soil	TBD	MSD	4 oz	x	x	x	1
SB-XX-depth-MS	Soil	TBD	MS	4 oz	x	x	x	1
SB-XX-depth-MSD	Soil	TBD	MSD	4 oz	x	x	x	1
SB-XX-depth-MS	Soil	TBD	MS	4 oz	x	x	x	1
SB-XX-depth-MSD	Soil	TBD	MSD	4 oz	x	x	x	1
Total number of solid containers					66	0	0	72
EB-SB-MMDDYY	Water (lab-provided)	-	EB	3.12 L	2	2	2	3
EB-SB-MMDDYY	Water (lab-provided)	-	EB	3.12 L	2	2	2	3
EB-SB-MMDDYY	Water (lab-provided)	-	EB	3.12 L	2	2	2	3
TB-07-MMDDYY	Methanol (lab-provided)	-	Trip Blank	80 mL				2
TB-08-MMDDYY	Methanol (lab-provided)	-	Trip Blank	80 mL				2
Total number of aqueous containers					6	6	6	13

TABLE 2E DQO 9 SAMPLE TYPES, VOLUMES AND TOTAL NUMBER OF CONTAINERS

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
NCW-B1-MMDDYY	Surface Water	Grab	1 L	2	2	x[1]					
NCW-B1-MMDDYY-F	Surface Water	Grab	500 mL	2							
NCW-B2-MMDDYY	Surface Water	Grab	1 L	2	2	x		1	1	1	
NCW-B2-MMDDYY-F	Surface Water	Grab	500 mL	2			1				
NCW-B3-MMDDYY	Surface Water	Grab	1 L	2	2	x					
NCW-B3-MMDDYY-F	Surface Water	Grab	500 mL	2							
NCW-B4-MMDDYY	Surface Water	Grab	1 L	2	2	x					
NCW-B4-MMDDYY-F	Surface Water	Grab	500 mL	2							
NCW-B5-MMDDYY	Surface Water	Grab	1 L	2	2	x		1	1	1	
NCW-B5-MMDDYY-F	Surface Water	Grab	500 mL	2			1				
NCW-B6-MMDDYY	Surface Water	Grab	1 L	2	2	x					
NCW-B6-MMDDYY-F	Surface Water	Grab	500 mL	2							
NCW-K1-MMDDYY	Surface Water	Duplicate	1 L	2	2	x		1	1	1	
NCW-K1-MMDDYY-F	Surface Water	Duplicate	500 mL	2			1				
NCW-XX-MMDDYY-MS	Surface Water	MS	1 L	2	2	x		1	1	1	
NCW-XX-MMDDYY-F-MS	Surface Water	MS	500 mL	2			1				
NCW-XX-MMDDYY-MSD	Surface Water	MSD	1 L	2	2	x		1	1	1	
NCW-XX-MMDDYY-F-MSD	Surface Water	MSD	500 mL	2			1				
EB-BSW-MMDDYY	Water (lab-provided)	EB	1 L	2	2						
EB-BSW-MMDDYY-F	Water (lab-provided)	EB	500 mL	2							
EB-Bsed-MMDDYY	Water (lab-provided)	EB	1 L	2	2						
EB-BGS-MMDDYY	Water (lab-provided)	EB	1 L	2	2						
EB-BGS-MMDDYY	Water (lab-provided)	EB	1 L	2	2						
EB-BGS-MMDDYY	Water (lab-provided)	EB	1 L	2	2						
Total number of aqueous containers				48	28	0	5	5	5	5	0

[1] An “x” signifies a sample analysis without a dedicated sample container.

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
NCS-B1-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-B2-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-B3-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-B4-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-B5-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-B6-MMDDYY	Sediment	Grab	8 oz	1	x						x
NCS-K1-MMDDYY	Sediment	Duplicate	8 oz	1	x						x
NCS-XX-MMDDYY-MS	Sediment	MS	*Take from parent*	x	x						x
NCS-XX-MMDDYY-MSD	Sediment	MSD	*Take from parent*	x	x						x
BG-01-1	Soil	Discrete	8 oz	1	x						
BG-01-2	Soil	Discrete	8 oz	1	x						
BG-01-3	Soil	Discrete	8 oz	1	x						
BG-01-4	Soil	Discrete	8 oz	1	x						
BG-01-5	Soil	Discrete	8 oz	1	x						
BG-01-6	Soil	Discrete	8 oz	1	x						
BG-01-7	Soil	Discrete	8 oz	1	x						
BG-01-8	Soil	Discrete	8 oz	1	x						
BG-01-9	Soil	Discrete	8 oz	1	x						
BG-01-10	Soil	Discrete	8 oz	1	x						
BG-01A	Soil	ISM	1 kilogram	1	x						
BG-01B	Soil	ISM	1 kilogram	1	x						
BG-01C	Soil	ISM	1 kilogram	1	x						
BG-02-1	Soil	Discrete	8 oz	1	x						
BG-02-2	Soil	Discrete	8 oz	1	x						
BG-02-3	Soil	Discrete	8 oz	1	x						
BG-02-4	Soil	Discrete	8 oz	1	x						
BG-02-5	Soil	Discrete	8 oz	1	x						
BG-02-6	Soil	Discrete	8 oz	1	x						
BG-02-7	Soil	Discrete	8 oz	1	x						

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
BG-02-8	Soil	Discrete	8 oz	1	x						
BG-02-9	Soil	Discrete	8 oz	1	x						
BG-02-10	Soil	Discrete	8 oz	1	x						
BG-02A	Soil	ISM	1 kilogram	1	x						
BG-02B	Soil	ISM	1 kilogram	1	x						
BG-02C	Soil	ISM	1 kilogram	1	x						
BG-03-1	Soil	Discrete	8 oz	1	x						
BG-03-2	Soil	Discrete	8 oz	1	x						
BG-03-3	Soil	Discrete	8 oz	1	x						
BG-03-4	Soil	Discrete	8 oz	1	x						
BG-03-5	Soil	Discrete	8 oz	1	x						
BG-03-6	Soil	Discrete	8 oz	1	x						
BG-03-7	Soil	Discrete	8 oz	1	x						
BG-03-8	Soil	Discrete	8 oz	1	x						
BG-03-9	Soil	Discrete	8 oz	1	x						
BG-03-10	Soil	Discrete	8 oz	1	x						
BG-03A	Soil	ISM	1 kilogram	1	x						
BG-03B	Soil	ISM	1 kilogram	1	x						
BG-03C	Soil	ISM	1 kilogram	1	x						
BG-04-1	Soil	Discrete	8 oz	1	x						
BG-04-2	Soil	Discrete	8 oz	1	x						
BG-04-3	Soil	Discrete	8 oz	1	x						
BG-04-4	Soil	Discrete	8 oz	1	x						
BG-04-5	Soil	Discrete	8 oz	1	x						
BG-04-6	Soil	Discrete	8 oz	1	x						
BG-04-7	Soil	Discrete	8 oz	1	x						
BG-04-8	Soil	Discrete	8 oz	1	x						
BG-04-9	Soil	Discrete	8 oz	1	x						
BG-04-10	Soil	Discrete	8 oz	1	x						
BG-04A	Soil	ISM	1 kilogram	1	x						

SAMPLE ID	MATRIX	TYPE	REQUIRED VOLUME	TAL Metals	PAHs	Hardness	DOC	Sulfate & Chloride	Alkalinity	Sulfide	TOC
BG-04B	Soil	ISM	1 kilogram	1	x						
BG-04C	Soil	ISM	1 kilogram	1	x						
BG-20-1	Soil	Duplicate	8 oz	1	x						
BG-20-2	Soil	Duplicate	8 oz	1	x						
BG-20-3	Soil	Duplicate	8 oz	1	x						
BG-20-4	Soil	Duplicate	8 oz	1	x						
BG-XX-X-MS	Soil	MS	*Take from parent*	x	x						
BG-XX-X-MSD	Soil	MSD	*Take from parent*	x	x						
BG-XX-X-MS	Soil	MS	*Take from parent*	x	x						
BG-XX-X-MSD	Soil	MSD	*Take from parent*	x	x						
BG-XXX-MS	Soil	MS	*Take from parent*	x	x						
BG-XXX-MSD	Soil	MSD	*Take from parent*	x	x						
Total number of solid containers:				63	0	0	0	0	0	0	0

TABLE 3 PERSONNEL ROLES AND RESPONSIBILITIES

ORGANIZATION	PROJECT TITLE, NAME, AND CONTACT INFORMATION	RESPONSIBILITIES
NPS	*CERCLA Project Coordinator, NPS Contaminated Site Project Manager* Sarah Venator Sarah_Venator@nps.gov	Review and approve the SAP before implementation. Approve changes to SAP before implementation. Approve corrective actions for field work and/or laboratory work before implementation if actions will affect the data quality or usability. Review and approve the Phase III RI Technical Memorandum.
Department of the Interior (DOI)	*Legal Lead* Nathalie Doherty nathalie.doherty@sol.doi.gov	Provide an opinion on changes to the SAP, as necessary. Review and approve the Phase III RI Technical Memorandum
NPS Contractor (VHB)	*Cleanup Lead* Jonathan Ordway jordway@vhb.com	Discuss the need for corrective actions for field work and/or laboratory work with the NPS Contaminated Site Project Manager and Legal Lead, as appropriate. Provide technical support and oversight during preparation of the draft and final Phase III RI Technical Memorandum.
NPS Contractor (VHB)	*Contractor Project Manager* Brie Hicknell bhicknell@vhb.com	Point of contact with NPS and Environmental Compliance and Cleanup Division (ECCD). Manage all project phases. Prepare SAP, QAPP, and Phase III RI Technical Memorandum. Review daily reports and submit to NPS and ECCD. Determine the need for corrective action for laboratory issues in conjunction with the Laboratory Project Manager and Data Validator, as appropriate.
NPS Contractor (VHB)	*Field Team Leader* Adam Robtoy arobtoy@vhb.com	Lead the field work and document daily activities in field notes and a daily progress report. Notify Contractor Project Manager and Technical Lead when unforeseen circumstances require work stoppage and/or immediate corrective actions. Verify completeness of and file all field notes, daily progress reports, COCs, shipping documents, corrective action forms, laboratory reports, and other project-related correspondence electronically on the Contractor server. Carry out corrective actions as directed by the Contractor Project Manager.
SGS North America, Inc.	*Laboratory Project Manager* Justin Nelson Justin.Nelson@sgs.com	Main point of contact at laboratory for all sample container ordering, shipping, sample receiving/logins, and COC questions. Notify the Contractor Project Manager of the need for corrective action for analytical issues.
Environmental Data Quality, Inc.	*Chemistry QA Officer/Data Validator* Shawne M. Rodgers edqi@environmentaldataqualityinc.com	Review field COC and sample logins, discuss the need for field or laboratory corrective action with the Contractor Project Manager.

ORGANIZATION	PROJECT TITLE, NAME, AND CONTACT INFORMATION	RESPONSIBILITIES
		Perform data validation, prepare the validation summary report, and approve the release of analytical data.

TABLE 4 STANDARD OPERATING PROCEDURES

TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION	MATRIX/ANALYTICAL GROUP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK?
Field SOPs				
SOP-VHB-007 Chain-of-Custody Records, v.6, 01/2022	VHB	N/A	N/A	No
SOP-VHB-18 Deviation from Protocols or SOPs and for Notation, Correction and Documentation of Unforeseen Circumstance, v.3, 01/2022	VHB	N/A	N/A	No
SOP-VHB-026 Soil Sampling for Organic and Inorganic Compounds, v.4, 01/2022	VHB	Soil	N/A	No
SOP-VHB-027 Decontamination of Field Equipment, v.5, 1/2022	VHB	N/A	N/A	No
SOP-VHB-034 Use of Field Log Books, v.4, 1/2022	VHB	N/A	N/A	No
SOP-VHB-055 Standard Operating Procedures for Calibration and Use of YSI Model 556 MPS, Revised 01/2022	VHB	Water	Multi-parameter YSI Model 556 MPS	No
SOP-VHB -028 Standard Operating Procedure for Sediment and Sludge Sampling, Revised 01/2022	VHB	Sediment	N/A	No
SOP-VHB-035 Standard Operating Procedure for Surface Water Sampling, Revised 01/2022	VHB	Water	N/A	No

TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION	MATRIX/ANALYTICAL GROUP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK?
SOP-VHB-036 Standard Operating Procedure for Geologic Description of Sediments, Revised 01/2022.	VHB	Sediment	N/A	No
USEPA Method 1669 Sampling Ambient Water for Trace Metals at USEPA Water Quality Criteria Levels, 07/1996.	USEPA	Water	N/A	No
Field Guide for Rock Core Logging and Fracture Analysis	Midwest GeoSciences Group	Rock	N/A	No
Laboratory SOPs				
ISM Prep Processing				
149r08: Multi-Incremental Soil Processing for Semi-volatile & Metal Analysis, 09/2021.	SGS North America, Inc. – Alaska Division.	Soil	N/A	No
Metals				
361r18: Acid Digestion of Soils, Sludges and Sediments for ICP-MS, Method SW846 3050B, 05/2021.	SGS North America, Inc. – Alaska Division.	Solid/TAL Metals	N/A	No
375r07: Total Recoverable Metals Low Level Preparation, Method 200.2, 04/2022	SGS North America, Inc. – Alaska Division.	Aqueous/TAL Metals	N/A	No
342r28: Determination of Metals by ICP-MS, Method SW6020B, 04/2023.	SGS North America, Inc. – Alaska Division.	Solid/TAL Metals	Inductively coupled plasma-mass spectrometer (ICP-MS)	No
345r12: Digestion of Aqueous Matrices for ICP-MS Metals Analyses, Method SW846 3010A, 04/2022.	SGS North America, Inc. – Alaska Division.	Aqueous/TAL Metals	N/A	No
354r15: Mercury in Water and Soil by Oxidation, Purge & Trap, and CVAFS, Method 1631E, 08/2023	SGS North America, Inc. – Alaska Division.	Aqueous and Solid/Mercury	PS Analytical Merlin System mercury analyzer	No
385r10: Determination of Metals in Drinking Water and Wastewater by ICP-MS (Low Level Method), Method 200.8, 09/2023	SGS North America, Inc. – Alaska Division.	Aqueous/TAL Metals	ICP-MS	No

TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION	MATRIX/ANALYTICAL GROUP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK?
Polyaromatic Hydrocarbons (PAHs)				
727r14: Polynuclear Aromatic Hydrocarbons by GCMS-SIM, Method 8270E-SIM, 625M-SIM, 02/2024	SGS North America, Inc. – Alaska Division.	Aqueous, Solid/PAHs	Gas Chromatograph/Mass Spectrometer (GC/MS)	No
784r02: Solid-Phase Extraction, Method SW846 3535A, 09/2021.	SGS North America, Inc. – Alaska Division.	Aqueous/PAHs	N/A	No
SVOCs				
761r24: Sonication Extraction of Semi-Volatile Compounds in Soil, Method SW846 3550C, 05/2023.	SGS North America, Inc. – Alaska Division.	Solid/SVOCs, PAHs	N/A	No
759r22: Continuous Liquid-Liquid Extraction for Semi-Volatile Compounds, Method SW846 3520C, 04/2023	SGS North America, Inc. – Alaska Division.	Aqueous/SVOCs	N/A	No
721r17: Extractable Semi-Volatile Compounds by GCMS, Method SW846 8270D and 625, 02/2022.	SGS North America, Inc. – Alaska Division.	Aqueous, Solid/SVOCs	GC/MS	No
VOCs				
767r13: Purge and Trap Extraction, Non-aqueous, Method SW846 5035A, 09/2021	SGS North America, Inc. – Alaska Division.	Solid/VOCs	N/A	No
764r13: Purge and Trap Aqueous, Method SW846 8000B & 5030, 09/2021	SGS North America, Inc. – Alaska Division.	Aqueous/VOCs	N/A	No
783r06: Purgeable Organic Compounds Analysis by GC/MS and GC/MS-SIM, Method 8260D and 824, 09/2021	SGS North America, Inc. – Alaska Division.	Solid, Aqueous/VOCs	GC/MS	No
TOC/DOC				
320r14: Total Organic Carbon in Soil, Method SW 9060A, 12/2021.	SGS North America, Inc. – Alaska Division.	Solid/TOC	TOC combustion analyzer	No

TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION	MATRIX/ANALYTICAL GROUP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK?
321r17: Total Organic Carbon and Dissolved Organic Carbon in Water, Method SM5310B/SW9060A, 12/2020	SGS North America, Inc. – Alaska Division.	Aqueous/DOC	Shimadzu TOC-V CSN with autosampler	No
Biotic Ligand Model Parameters				
394r010: Alkalinity, Acidity, pH, Free CO_2 & Conductivity by Auto-titrator, Method SM2320B, SM2310B, SM4500H-B, 4500-CO_2, SM2510A and B, 04/2023	SGS North America, Inc. – Alaska Division.	Aqueous/Alkalinity	N/A	No
368r14: Sulfide in Water – Colorimetric, EPA 376.2; SM4500S^{2-}, Hach Method 8131, 11/2022	SGS North America, Inc. – Alaska Division.	Aqueous/Sulfide	N/A	No
318r28: Determination of Inorganic Anions by Ion Chromatography, USEPA Method 300.0, SW9056A, SW5050, 09/2023	SGS North America, Inc. – Alaska Division.	Aqueous/Chloride & Sulfate	Ion chromatograph	No
IVBA				
ENV-SOP-SHRT-0162v00: In Vitro Bioaccessibility Assay for Metals in Soil by EPA Method 1340, 05/01/2023.	Pace Analytical Services	Soil/IVBA	N/A	No
ENV-SOP-MTJL-0221: Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8). 07/25/2022.	Pace Analytical Services	Soil/Metals in IVBA samples	ICP/MS	No

TABLE 5 SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

DQO	ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ LABORATORY SOP[1]	CONTAINER(S) (NUMBER, SIZE & TYPE PER SAMPLE)	PRESERVATION	HOLDING TIME (days)	DATA PACKAGE TURNAROUND (business days)
9	TAL Metals, PAHs	Soil - discrete	6020B, 8270E-SIM/361r18, 342r28, 354r15, 727r14, 761r24	1 x 8-oz glass jar w/ Teflon lid	≤ 6 degrees Celsius (°C)	Metals except Mercury: 180 Mercury: 28 PAHs: 14-extraction; 40-analysis	10
2/9	TAL Metals, PAHs	Soil – ISM	6020B, 8270E-SIM/361r18, 342r28, 354r15, 727r14, 761r24, 149r08	1 gallon Ziploc bag	≤ 6 °C	Metals except Mercury: 180 Mercury: 28 PAH extraction: 14 days, analysis: 40 days	10
2	IVBA	Soil - discrete	1340/ENV-SOP-SHRT-0162v00, ENV-SOP-MTJL-0221	1 gallon Ziploc bag	None	6 months	10
6/7/9	TAL Metals (except Mercury), hardness	Surface Water/ Groundwater	200.8 Low Level, SM 2340B/385r10	250-mL HDPE	HNO_3; pH <2	Metals: 180 Hardness: 180	10
6/7/9	Mercury	Surface Water/ Groundwater	1631E/354r15	250-mL fluoropolymer	HCl; pH < 2	90	10
6/7/9	Dissolved TAL Metals (except Mercury), hardness	Surface Water	200.8 Low Level, SM 2340B/385r10	250-mL HDPE	Field-filtered; HNO_3; pH <2	Metals: 180 Hardness: 180	10
6/7/9	Dissolved Mercury	Surface Water	1631E/354r15	250-mL fluoropolymer	Field-filtered; HCl; pH < 2	90	10
6/7/9	PAHs	Surface Water/ Groundwater	8270E-SIM/727r14, 784r02	2 x 250-mL amber glass	≤ 6 °C	7-extraction; 40-analysis	10
7	SVOCs	Groundwater	8270D/759r22, 721r17	2 x 1-L amber glass	≤ 6 °C	7-extraction; 40-analysis	10
6/7	VOCs	Surface Water/ Groundwater	8260D/783r06, 764r13	3 x 40 mL VOA vials	HCl; pH < 2; ≤ 6°C	14	10

DQO	ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ LABORATORY SOP[1]	CONTAINER(S) (NUMBER, SIZE & TYPE PER SAMPLE)	PRESERVATION	HOLDING TIME (days)	DATA PACKAGE TURNAROUND (business days)
					(no headspace)		
6/7	DOC	Surface Water/ Groundwater	5310B/9060A/320r14	250 mL amber glass	Field-filtered; HCl; pH < 2; ≤ 6°C	28	10
6/7	Sulfate, chloride	Surface Water/ Groundwater	300.0/318r28	125-mL HPDE	≤ 6 °C	28	10
6/7	Alkalinity	Surface Water/ Groundwater	2320B/394r010	250-mL HDPE	≤ 6 °C	14	10
6/7	Sulfide	Surface Water/ Groundwater	4500-S^{2-}-D/368r14	250-mL HPDE	$ZnAc_2$ & NaOH, pH > 9; ≤ 6°C	7	10
6/9	TAL Metals, PAHs, TOC	Sediment	6020B, 1631E, 8270, 8270-SIM, 9060A/361r18, 342r28, 354r15, 727r14, 761r24, 320r14	1 x 8-oz glass jar w/ Teflon lid	≤ 6 °C	PAHs: 14-extraction; 40-analysis; Metals except Mercury: 180; Mercury & TOC: 28	10
6/8/9	VOCs[2]	Sediment/Soil[2]	8260D/783r06, 767r13	1 pre-weighed 4 oz. jar + 1 x 40 mL VOA vial containing 25 mL MeOH	MeOH; ≤ 6°C	14	10
8	TAL Metals, PAHs, SVOCs,	Soil	6020B, 1631E, 8270D, 8270E-SIM/361r18, 342r28, 354r15, 727r14, 761r24, 721r17,	1 x 8-oz glass jar w/ Teflon lid	≤ 6 °C	SVOCs: 14-extraction; 40-analysis; Metals except Mercury: 180; Mercury: 28	10

Notes:

[1] Laboratory SOPs are listed in Table 4.

[2] For each VOC soil/sediment sample, the laboratory will provide a pre-weighed straight-sided 4 oz. jar plus one 40 mL vial containing 25 mL of MeOH that has been spiked with the VOC surrogates. VHB should place between 50 to 75 grams of sediment into the 4-oz jar and then all of the MeOH from the 40 mL vial should be poured over the sample so that the sediment is completely covered by solvent. The sealed jar is ready for shipment to the laboratory.

Laboratory Information: *SGS North America, Inc. Alaska Division. 200 W Potter Dr, Anchorage, AK 99518*

Contact: *Justin Nelson, Justin.Nelson@sgs.com, 907-206-1339*

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
Metals (6020)									
Aluminum	7429-90-5	7700	110000	50	None	None	**50**	7	20
Antimony	7440-36-0	3.1	47	0.25	41	4.6	**0.25**	**0.31**	**1**
Arsenic	7440-38-2	0.68	3	0.25	8.8	0.2	**0.2**	**0.31**	**1**
Barium	7440-39-3	1,500	22,000	17	20000	2100	**17**	0.094	0.3
Beryllium	7440-41-7	16	230	2.4	200	260	**2.4**	0.031	0.1
Cadmium	7440-43-9	0.71	10	0.27	92	9.1	**0.27**	0.062	0.2
Calcium	7440-70-2	None	None	None	None	None	**None**	15	50
Chromium (total)	7440-47-3	None	None	0.34	None	None	**0.34**	*0.31*	*1*
Cobalt	7440-48-4	2.3	35	13	None	None	**2.3**	0.15	0.5
Copper	7440-50-8	310	4,700	14	4,100	370	**14**	0.18	0.6
Iron	7439-89-6	5500	82000	None	None	None	**5500**	15	50
Lead	7439-92-1	100	800	0.94	400	None	**0.94**	0.062	0.2
Magnesium	7439-95-4	None	None	None	None	None	**None**	15	50
Manganese	7439-96-5	180	2600	220	2700	370	**180**	0.062	0.2
Nickel	7440-02-0	140	1800	10	2000	340	**10**	0.062	0.2
Potassium	7440-09-7	None	None	None	None	None	**None**	31	100
Selenium	7782-49-2	39	580	0.33	510	7	**0.33**	**0.62**	**2**
Silver	7440-22-4	39	580	2	510	11	**2**	0.15	0.5
Sodium	7440-23-5	None	None	None	None	None	**None**	31	100
Thallium	7440-28-0	0.078	1.2	0.027	1	0.19	**0.027**	**0.062**	**0.2**
Vanadium	7440-62-2	39	580	0.71	510	1100	**0.71**	**1.5**	**5**
Zinc	7440-66-6	2300	35000	6.6	30000	4900	**6.6**	0.78	2.5
Mercury (1631)									
Mercury	7439-97-6	1.1	4.6	0.013	3.1	0.36	**0.013**	0.000078	0.00025

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
Non-PAH SVOCs (8270)									
2,2'-oxybis (1-chloropropane)	108-60-1	310	4700	None	None	None	**310**	0.00063	0.005
2,4,5-Trichlorophenol	95-95-4	630	8200	4	8200	28	**4**	0.0008	0.005
2,4,6-Trichlorophenol	88-06-2	6.3	82	10	82	0.092	**0.092**	0.00058	0.005
2,4-Dichlorophenol	120-83-2	19	250	None	250	0.21	**0.21**	0.00057	0.005
2,4-Dimethylphenol	105-67-9	130	1600	0.01	1600	3.2	**0.01**	0.0013	0.005
2,4-Dinitrophenol	51-28-5	13	160	20	160	0.34	**0.34**	0.005	0.025
2,4-Dinitrotoluene	121-14-2	1.7	7.4	6	23	0.024	**0.024**	0.0005	0.005
2,6-Dinitrotoluene	606-20-2	0.36	1.5	4	4.7	0.005	**0.005**	0.00065	0.005
2-Chloronaphthalene	91-58-7	480	6000	None	6200	26	**26**	0.0005	0.005
2-Chlorophenol	95-57-8	39	580	0.39	510	0.71	**0.39**	0.00061	0.005
2-Methylphenol	95-48-7	320	4100	0.67	4100	6.2	**0.67**	0.0006	0.005
2-Nitroaniline	88-74-4	63	800	5.3	None	None	**5.3**	0.0012	0.005
2-Nitrophenol	88-75-5	None	None	7	None	None	**7**	0.00054	0.005
3&4-Methylphenol	108-39-4 & 106-44-5	130	1600	None	4100	6.1	**6.1**	0.00082	0.005
3,3´-Dichlorobenzidine	91-94-1	1.2	5.1	None	16	0.056	**0.056**	0.0012	0.005
3-Nitroaniline	99-09-2	None	None	None	None	None	**None**	0.00058	0.005
4,6-Dinitro-2-methylphenol	534-52-1	0.51	6.6	None	None	None	**0.51**	0.002	0.01
4-Bromophenyl phenyl ether	101-55-3	None	None	None	None	None	**None**	0.00052	0.005
4-Chloro-3-methylphenol	59-50-7	630	8200	None	None	None	**630**	0.00057	0.005
4-Chloroaniline	106-47-8	2.7	11	1	35	0.015	**0.015**	0.0013	0.005
4-Chlorophenyl phenyl ether	7005-72-3	None	None	None	None	None	**None**	0.0005	0.005
4-Nitroaniline	100-01-6	25	110	None	None	None	**25**	0.0014	0.005
4-Nitrophenol	100-02-7	None	None	7	None	None	**7**	0.005	0.025
bis(-2-chloroethoxy)Methane	111-91-1	19	250	None	None	None	**19**	0.0005	0.005

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
bis(-2-chloroethyl)Ether	111-44-4	0.23	1	None	2.8	0.00042	**0.00042**	**0.00058**	**0.005**
bis(2-ethylhexyl)Phthalate	117-81-7	39	160	0.02	500	88	**0.02**	0.001	0.01
Butylbenzylphthalate	85-68-7	290	1200	90	3700	16	**16**	0.001	0.005
Carbazole	86-74-8	None	None	79	None	None	**79**	0.0007	0.005
Dibenzofuran	132-64-9	7.8	120	6.1	95	0.97	**0.97**	0.0005	0.005
Diethyl phthalate	84-66-2	5100	66000	100	66000	60	**60**	0.001	0.01
Dimethyl phthalate	131-11-3	None	None	10	66000	48	**10**	0.001	0.005
Di-n-butyl phthalate	84-74-2	630	8200	0.011	8200	16	**0.011**	0.002	0.01
Di-n-octyl phthalate	117-84-0	63	820	0.91	820	370	**0.91**	0.001	0.005
Hexachlorobenzene	118-74-1	0.078	0.96	0.079	2	0.0082	**0.0082**	0.00051	0.005
Hexachlorobutadiene	87-68-3	1.2	5.3	None	3.3	0.02	**0.02**	0.00051	0.005
Hexachlorocyclopentadiene	77-47-4	0.18	0.75	10	1.4	0.0093	**0.0093**	0.001	0.005
Hexachloroethane	67-72-1	1.8	8	None	17	0.018	**0.018**	0.00059	0.005
Isophorone	78-59-1	570	2400	None	7400	2.7	**2.7**	0.0005	0.005
Nitrobenzene	98-95-3	5.1	22	2.2	43	0.0079	**0.0079**	0.0005	0.005
n-Nitroso-di-n-propylamine	621-64-7	0.078	0.33	None	1	0.00068	**0.00068**	*0.0005*	*0.005*
N-Nitrosodiphenylamine	86-30-6	110	470	20	1400	4.6	**4.6**	0.00054	0.005
Pentachlorophenol	87-86-5	1	4	0.36	13	0.0043	**0.0043**	**0.005**	**0.025**
Phenol	108-95-2	1900	25000	0.79	25000	29	**0.79**	0.0005	0.005
PAHs (8270-SIM)									
1-Methylnaphthalene	90-12-0	18	73	None	68	0.41	**0.41**	0.00625	0.025
2-Methylnaphthalene	91-57-6	24	300	16	310	1.3	**1.3**	0.00625	0.025
Acenaphthene	83-32-9	360	4500	0.25	4600	37	**0.25**	0.00625	0.025
Acenaphthylene	208-96-8	None	None	120	2300	18	**18**	0.00625	0.025
Anthracene	120-12-7	1800	23000	6.8	23000	390	**6.8**	0.00625	0.025
Benzo(a)anthracene	56-55-3	1.1	21	0.73	14	0.7	**0.7**	0.00625	0.025
Benzo(a)pyrene	50-32-8	0.11	2.1	2	1.5	1.9	**0.11**	0.00625	0.025
Benzo(b)fluoranthene	205-99-2	1.1	21	18	15	20	**1.1**	0.00625	0.025
Benzo(g,h,i)perylene	191-24-2	None	None	25	2300	15000	**25**	0.00625	0.025
Benzo(k)fluoranthene	207-08-9	11	210	71	150	190	**11**	0.00625	0.025
Chrysene	218-01-9	110	2100	3.1	1500	600	**3.1**	0.00625	0.025
Dibenzo(a,h)anthracene	53-70-3	0.11	2.1	14	1.5	6.3	**0.11**	0.00625	0.025
Fluoranthene	206-44-0	240	3000	10	3100	590	**10**	0.00625	0.025

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
Fluorene	86-73-7	240	3000	3.7	3100	36	**3.7**	0.00625	0.025
Indeno(1,2,3-cd)pyrene	193-39-5	1.1	21	71	15	65	**1.1**	0.00625	0.025
Naphthalene	91-20-3	2	8.6	1	29	0.038	**0.038**	0.005	0.020
Phenanthrene	85-01-8	None	None	5.5	2300	39	**5.5**	0.00625	0.025
Pyrene	129-00-0	180	2300	10	2300	87	**10**	0.00625	0.025
VOCs (8260)									
1,1,1,2-Tetrachloroethane	630-20-6	2	8.8	None	21	0.022	**0.022**	0.0062	0.02
1,1,1-Trichloroethane	71-55-6	810	3600	260	360	32	**32**	0.0078	0.025
1,1,2,2-Tetrachloroethane	79-34-5	0.6	2.7	None	6.1	0.003	**0.003**	0.00062	0.002
1,1,2-Trichloroethane	79-00-5	0.15	0.63	None	1.6	0.0014	**0.0014**	0.0005	0.001
1,1-Dichloroethane	75-34-3	3.6	16	210	46	0.092	**0.092**	0.0078	0.025
1,1-Dichloroethene	75-35-4	23	100	11	330	1.2	**1.2**	0.0078	0.025
1,1-Dichloropropene	563-58-6	None	None	None	None	None	**None**	0.0078	0.025
1,2,3-Trichlorobenzene	87-61-6	6.3	93	20	81	0.15	**0.15**	0.03	0.1
1,2,3-Trichloropropane	96-18-4	0.0051	0.11	None	0.066	0.000031	**0.000031**	**0.00062**	**0.002**
1,2,4-Trichlorobenzene	120-82-1	5.8	26	0.27	45	0.082	**0.082**	0.0078	0.025
1,2,4-Trimethylbenzene	95-63-6	30	180	None	43	0.61	**0.61**	0.03	0.1
1,2-Dibromo-3-chloropropane	96-12-8	0.0053	0.064	None	None	None	**0.0053**	**0.031**	**0.1**
1,2-Dibromoethane	106-93-4	0.036	0.16	None	0.42	0.00024	**0.00024**	*0.00075*	*0.0015*
1,2-Dichlorobenzene	95-50-1	180	930	0.92	78	2.4	**0.92**	0.0078	0.025
1,2-Dichloroethane	107-06-2	0.46	2	0.85	5.5	0.0055	**0.0055**	0.0007	0.002
1,2-Dichloropropane	78-87-5	1.6	6.6	700	17	0.03	**0.03**	0.005	0.01
1,3,5-Trimethylbenzene	108-67-8	27	150	None	37	0.66	**0.66**	0.0078	0.025
1,3-Dichlorobenzene	541-73-1	None	None	0.74	62	2.3	**0.74**	0.0078	0.025
1,3-Dichloropropane	142-28-9	160	2300	None	None	None	**160**	0.0031	0.01
1,4-Dichlorobenzene	106-46-7	2.6	11	0.89	21	0.037	**0.037**	0.0078	0.025
2,2-Dichloropropane	594-20-7	None	None	None	None	None	**None**	0.0078	0.025
2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	2700	19000	350	23000	15	**15**	0.078	0.025
2-Chlorotoluene	95-49-8	160	2300	None	None	None	**160**	0.0078	0.25
2-Hexanone	591-78-6	20	130	0.36	270	0.11	**0.11**	0.06	0.025
4-Chlorotoluene	106-43-4	160	2300	None	None	None	**160**	0.01	0.12
4-Isopropyltoluene	99-87-6	None	None	None	None	None	**None**	0.04	0.08
4-Methyl-2-pentanone (MIBK)	108-10-1	3300	14000	9.7	2200	18	**9.7**	0.078	0.25

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
Acetone	67-64-1	7000	110000	1.2	81000	38	**1.2**	0.11	0.25
Benzene	71-43-2	1.2	5.1	24	11	0.022	**0.022**	0.0039	0.0125
Bromobenzene	108-86-1	29	180	None	160	0.36	**0.36**	0.0078	0.025
Bromochloromethane	74-97-5	15	63	None	None	None	**15**	0.0078	0.025
Bromodichloromethane	75-27-4	0.29	1.3	None	3.6	0.0043	**0.0043**	0.00062	0.002
Bromoform	75-25-2	19	86	None	240	0.1	**0.1**	0.0078	0.025
Bromomethane	74-83-9	0.68	3	None	10	0.024	**0.024**	0.008	0.02
Carbon disulfide	75-15-0	77	350	0.81	500	2.9	**0.81**	0.031	0.1
Carbon tetrachloride	56-23-5	0.65	2.9	58.6	9.1	0.021	**0.021**	0.0039	0.0125
Chlorobenzene	108-90-7	28	130	2.4	180	0.46	**0.46**	0.0078	0.025
Chloroethane	75-00-3	540	2300	None	1400	72	**72**	0.062	0.2
Chloroform	67-66-3	0.32	1.4	8	4	0.0071	**0.0071**	0.003	0.006
Chloromethane	74-87-3	11	46	None	170	0.61	**0.61**	0.0078	0.025
cis-1,2-Dichloroethene	156-59-2	6.3	37	89.6	200	0.12	**0.12**	0.0078	0.025
cis-1,3-Dichloropropene	10061-01-5	None	None	None	None	None	**None**	0.0039	0.0125
Dibromochloromethane	124-48-1	8.3	39	None	110	0.0027	**0.0027**	0.0015	0.005
Dibromomethane	74-95-3	2.4	9.9	None	31	0.025	**0.025**	0.0078	0.025
Dichlorodifluoromethane	75-71-8	8.7	37	None	150	3.9	**3.9**	0.03	0.1
Ethylbenzene	100-41-4	5.8	25	None	49	0.13	**0.13**	0.0078	0.025
Isopropylbenzene	98-82-8	190	990	None	54	5.6	**5.6**	0.0078	0.025
m&p-Xylene	179601-23-1	58^	250^	4.162	None	None	**4.162**	0.015	0.05
Methyl tert-butyl ether (MTBE)	1634-04-4	47	210	None	670	0.4	**0.4**	0.031	0.1
Methylene chloride	75-09-2	35	320	2.6	None	None	**2.6**	0.031	0.1
n-Butylbenzene	104-51-8	390	5800	None	20	23	**20**	0.0078	0.025
n-Propylbenzene	103-65-1	380	2400	None	52	9.1	**9.1**	0.0078	0.025
o-Xylene	95-47-6	64	280	4.162	None	None	**4.162**	0.0078	0.025
sec-Butylbenzene	135-98-8	780	12000	None	28	42	**28**	0.0078	0.025
Styrene	100-42-5	600	3500	1.2	180	10	**1.2**	0.0078	0.025
tert-Butylbenzene	98-06-6	780	12000	None	None	None	**780**	0.0078	0.025
Tetrachloroethene	127-18-4	8.1	39	0.18	68	0.19	**0.18**	0.0039	0.0125
Toluene	108-88-3	490	4700	23	200	6.7	**6.7**	0.0078	0.025
trans-1,2-Dichloroethene	156-60-5	7	30	89.6	960	1.3	**1.3**	0.0078	0.01
trans-1,3-Dichloropropene	10061-02-6	None	None	None	None	None	**None**	0.0039	0.0125

Table 6A - Soil Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)					Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Soil ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]	Alaska Migration to Groundwater Soil Cleanup Levels[5]		MDLs	QLs
Trichloroethene	79-01-6	0.41	1.9	1.387	None	None	**0.41**	0.0032	0.05
Trichlorofluoromethane	75-69-4	2300	35000	52	980	41	**41**	0.015	0.0008
Vinyl chloride	75-01-4	0.059	1.7	0.12	None	0.0008	**0.0008**	0.00025	0.0008

Sources:

[1] EPA Residential Soil Regional Screening Level (TR 1E-06 and HQ 0.1), November 2023

[2] EPA Industrial Soil Regional Screening Level (TR 1E-06 and HQ 0.1), November 2023

[3] NPS Ecological Screening Level (ESV) Lowest SLERA COPEC Selection Value for Soil, November 2018

[4] Alaska State Soil Cleanup Levels (Under 40-inch Zone), October 2023

[5] Alaska State Soil Cleanup Levels (Migration to Groundwater), October 2023

Notes:

Black cell indicates MDL and QL are greater than the Project Action Limit.

Italicized text in grey cell indicates QL is greater than, but MDL is less than the Project Action Level.

MDL = Method detection limit

QL = Quantitation limit

mg/kg = milligram per kilogram

Table 6B - Groundwater Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
Metals (200.8 low level)														
Aluminum	7429-90-5	None	2000	None	5	None	None	5000	750	87	None	**5**	0.62	2
Antimony	7440-36-0	6	0.78	5.6	30	6	None	None	None	None	14	**0.78**	0.015	0.05
Arsenic	7440-38-2	10	0.052	0.018	3.1	10	50	100	340	150	None	**0.018**	**0.2**	**0.8**
Barium	7440-39-3	2,000	380	1,000	3.9	2000	None	None	None	None	None	**3.9**	0.04	0.25
Beryllium	7440-41-7	4	2.5	None	0.66	4	None	100	None	None	None	**0.66**	0.025	0.05
Cadmium	7440-43-9	5	0.18	None	0.07	5	10	10	*	*	None	**0.07**	0.015	0.05
Calcium	7440-70-2	None	None	None	116,000	None	None	None	None	None	None	**116000**	15	50
Chromium (total)	7440-47-3	100	None	None	None	100	None	100	None	None	None	**100**	0.15	0.5
Cobalt	7440-48-4	None	0.6	None	3	None	None	50	None	None	None	**0.6**	0.01	0.025
Copper	7440-50-8	1300	80	1300	0.23	None	None	200	*	*	1300	**0.23**	*0.2*	*0.5*
Iron	7439-89-6	None	1,400	None	158	None	None	5000	None	1000	None	**158**	6.2	20
Lead	7439-92-1	15	15	None	0.92	None	50	5,000	*	*	None	**0.92**	0.031	0.1
Magnesium	7439-95-4	None	None	None	82,000	None	None	None	None	None	None	**82000**	6.2	20
Manganese	7439-96-5	None	43	50	112	300	None	200	None	None	None	**43**	0.031	0.1
Nickel	7440-02-0	None	39	610	5	None	None	200	*	*	610	**5**	0.15	0.62
Potassium	7440-09-7	None	None	None	53,000	None	None	None	None	None	None	**53000**	15	50
Selenium	7782-49-2	50	10	170	1	50	10	20	None	5	170	**1**	0.31	1
Silver	7440-22-4	None	9.4	None	0.067	None	None	None	*	None	None	**0.067**	0.0062	0.02
Sodium	7440-23-5	None	None	None	680,000	None	None	None	None	None	None	**680000**	31	100
Thallium	7440-28-0	2	0.02	0.24	0.03	2	None	None	None	None	1.7	**0.02**	0.0062	0.02
Vanadium	7440-62-2	None	8.6	None	19	None	None	100	None	None	None	**8.6**	0.31	1
Zinc	7440-66-6	None	600	7400	30	None	None	2000	*	*	9100	**30**	3.1	10
Mercury (1631E)														
Mercury	7439-97-6	2	0.063	None	0.026	2	None	None	2.4	0.012	0.05	**0.012**	0.0005	0.001

Table 6B - Groundwater Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
Non-PAH SVOCs (8270E)														
2,2'-oxybis (1-chloropropane)	108-60-1	None	71	200	None	None	None	None	None	None	None	**71**	0.74	5
2,4,5-Trichlorophenol	95-95-4	None	120	300	18	None	None	None	None	None	2600	**18**	0.74	5
2,4,6-Trichlorophenol	88-06-2	None	1.2	1.5	18	None	None	None	None	None	None	**1.2**	*0.75*	*5*
2,4-Dichlorophenol	120-83-2	None	4.6	10	0.2	None	None	None	None	None	93	**0.2**	**0.84**	**5**
2,4-Dimethylphenol	105-67-9	None	36	100	None	None	None	None	None	None	540	**36**	0.74	5
2,4-Dinitrophenol	51-28-5	None	3.9	10	None	None	None	None	None	None	70	**3.9**	**5**	**25**
2,4-Dinitrotoluene	121-14-2	None	0.24	0.049	65	None	None	None	None	None	None	**0.049**	**0.81**	**5**
2,6-Dinitrotoluene	606-20-2	None	0.049	None	230	None	None	None	None	None	None	**0.049**	**0.71**	**5**
2-Chloronaphthalene	91-58-7	None	75	800	None	None	None	None	None	None	1700	**75**	0.5	5
2-Chlorophenol	95-57-8	None	9.1	30	7	None	None	None	None	None	120	**7**	0.63	5
2-Methylphenol	95-48-7	None	93	None	13	None	None	None	None	None	None	**13**	0.85	5
2-Nitroaniline	88-74-4	None	19	None	None	None	None	None	None	None	None	**19**	1.8	5
2-Nitrophenol	88-75-5	None	None	None	60	None	None	None	None	None	None	**60**	0.85	5
3&4-Methylphenol	108-39-4 & 106-44-5	None	37	None	None	None	None	None	None	None	None	**37**	0.98	5
3,3´-Dichlorobenzidine	91-94-1	None	0.13	0.049	None	None	None	None	None	None	None	**0.049**	**0.64**	**5**
3-Nitroaniline	99-09-2	None	None	None	None	None	None	None	None	None	None	**None**	0.88	5
4,6-Dinitro-2-methylphenol	534-52-1	None	0.15	2	None	None	None	None	None	None	13.4	**0.15**	**2**	**10**
4-Bromophenyl phenyl ether	101-55-3	None	None	None	1.5	None	None	None	None	None	None	**1.5**	*0.85*	*5*
4-Chloro-3-methylphenol	59-50-7	None	140	500	None	None	None	None	None	None	None	**140**	0.59	5
4-Chloroaniline	106-47-8	None	0.37	None	None	None	None	None	None	None	None	**0.37**	**0.63**	**5**
4-Chlorophenyl phenyl ether	7005-72-3	None	None	None	None	None	None	None	None	None	None	**None**	0.54	5
4-Nitroaniline	100-01-6	None	3.8	None	None	None	None	None	None	None	None	**3.8**	*1.2*	*5*
4-Nitrophenol	100-02-7	None	None	None	60	None	None	None	None	None	None	**60**	5	25
bis(-2-chloroethoxy)Methane	111-91-1	None	5.9	None	None	None	None	None	None	None	None	**5.9**	0.81	5
bis(-2-chloroethyl)Ether	111-44-4	None	0.014	0.03	None	None	None	None	None	None	None	**0.014**	**0.73**	**5**
bis(2-ethylhexyl)Phthalate	117-81-7	6	5.6	0.32	3	6	None	None	None	None	None	**0.32**	**1**	**5**
Butylbenzylphthalate	85-68-7	None	16	0.1	19	None	None	None	None	None	3000	**0.1**	**1**	**5**
Carbazole	86-74-8	None	None	None	None	None	None	None	None	None	None	**None**	0.6	5
Dibenzofuran	132-64-9	None	0.79	None	3.7	None	None	None	None	None	None	**0.79**	*0.6*	*5*
Diethyl phthalate	84-66-2	None	1500	600	210	None	None	None	None	None	23000	**210**	1	5
Dimethyl phthalate	131-11-3	None	None	2000	3	None	None	None	None	None	313000	**3**	*1*	*5*
Di-n-butyl phthalate	84-74-2	None	90	20	19	None	None	None	None	None	2700	**19**	1	5
Di-n-octyl phthalate	117-84-0	None	20	None	3	None	None	None	None	None	None	**3**	*1*	*5*
Hexachlorobenzene	118-74-1	1	0.0098	0.000079	None	1	None	None	None	None	None	**0.000079**	**0.69**	**5**
Hexachlorobutadiene	87-68-3	None	0.14	0.01	1.3	None	None	None	None	None	None	**0.01**	**0.5**	**5**
Hexachlorocyclopentadiene	77-47-4	50	0.041	4	None	50	None	None	None	None	240	**0.041**	**1.8**	**5**
Hexachloroethane	67-72-1	None	0.33	0.1	12	None	None	None	None	None	None	**0.1**	**1.6**	**5**
Isophorone	78-59-1	None	78	34	None	None	None	None	None	None	None	**34**	0.78	5
Nitrobenzene	98-95-3	None	0.14	10	550	None	None	None	None	None	17	**0.14**	**0.93**	**5**
n-Nitroso-di-n-propylamine	621-64-7	None	0.011	0.005	None	None	None	None	None	None	None	**0.005**	**0.67**	**5**
N-Nitrosodiphenylamine	86-30-6	None	12	3.3	40	None	None	None	None	None	None	**3.3**	*0.81*	*5*
Pentachlorophenol	87-86-5	1	0.041	0.03	0.5	1	None	None	None	None	None	**0.03**	**5**	**25**
Phenol	108-95-2	None	580	4000	4	None	None	None	None	None	21000	**4**	*0.5*	*5*

Table 6B - Groundwater Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
PAHs (8270D-SIM)														
1-Methylnaphthalene	90-12-0	None	1.1	None	None	None	None	None	None	None	None	**1.1**	0.0037	0.0125
2-Methylnaphthalene	91-57-6	None	3.6	None	330	None	None	None	None	None	None	**3.6**	0.0037	0.0125
Acenaphthene	83-32-9	None	53	70	5.8	None	None	None	None	None	1200	**5.8**	0.0037	0.0125
Acenaphthylene	208-96-8	None	None	None	4800	None	None	None	None	None	None	**4800**	0.0037	0.0125
Anthracene	120-12-7	None	180	300	0.012	None	None	None	None	None	9600	**0.012**	*0.0037*	*0.0125*
Benzo(a)anthracene	56-55-3	None	0.03	0.0012	0.018	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Benzo(a)pyrene	50-32-8	0.2	0.025	0.00012	0.014	0.2	None	None	None	None	None	**0.00012**	**0.0015**	**0.005**
Benzo(b)fluoranthene	205-99-2	None	0.25	0.0012	9	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Benzo(g,h,i)perylene	191-24-2	None	None	None	7.6	None	None	None	None	None	None	**7.6**	0.0037	0.0125
Benzo(k)fluoranthene	207-08-9	None	2.5	0.012	0.0041	None	None	None	None	None	None	**0.0041**	*0.0037*	*0.0125*
Chrysene	218-01-9	None	25	0.12	0.0018	None	None	None	None	None	None	**0.0018**	**0.0037**	**0.0125**
Dibenzo(a,h)anthracene	53-70-3	None	0.025	0.00012	0.0034	None	None	None	None	None	None	**0.00012**	**0.0015**	**0.005**
Fluoranthene	206-44-0	None	80	20	0.04	None	None	None	None	None	300	**0.04**	0.0037	0.0125
Fluorene	86-73-7	None	29	50	3	None	None	None	None	None	1300	**3**	0.0037	0.0125
Indeno(1,2,3-cd)pyrene	193-39-5	None	0.25	0.0012	4.3	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Naphthalene	91-20-3	None	0.12	None	1.1	None	None	None	None	None	None	**0.12**	0.0078	0.025
Phenanthrene	85-01-8	None	None	None	0.4	None	None	None	None	None	None	**0.4**	0.00775	0.025
Pyrene	129-00-0	None	12	20	0.025	None	None	None	None	None	960	**0.025**	0.0037	0.0125
VOCs (8260D)														
1,1,1,2-Tetrachloroethane	630-20-6	None	0.57	None	None	None	None	None	None	None	None	**0.57**	0.15	0.5
1,1,1-Trichloroethane	71-55-6	200	800	10000	11	200	None	None	None	None	None	**11**	0.31	1
1,1,2,2-Tetrachloroethane	79-34-5	None	0.076	0.2	610	None	None	None	None	None	None	**0.076**	**0.15**	**0.5**
1,1,2-Trichloroethane	79-00-5	5	0.041	0.55	1200	5	None	None	None	None	None	**0.041**	**0.12**	**0.4**
1,1-Dichloroethane	75-34-3	None	2.8	None	47	None	None	None	None	None	None	**2.8**	0.31	1
1,1-Dichloroethene	75-35-4	7	28	300	25	7	None	None	None	None	None	**7**	0.31	1
1,1-Dichloropropene	563-58-6	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
1,2,3-Trichlorobenzene	87-61-6	None	0.7	None	8	None	None	None	None	None	None	**0.7**	*0.31*	*1*
1,2,3-Trichloropropane	96-18-4	None	0.00075	None	None	None	None	None	None	None	None	**0.00075**	**0.31**	**1**
1,2,4-Trichlorobenzene	120-82-1	70	0.4	0.071	24	70	None	None	None	None	260	**0.071**	**0.31**	**1**
1,2,4-Trimethylbenzene	95-63-6	None	5.6	None	None	None	None	None	None	None	None	**5.6**	0.31	1
1,2-Dibromo-3-chloropropane	96-12-8	0.2	0.00033	None	None	None	None	None	None	None	None	**0.00033**	**3.1**	**10**
1,2-Dibromoethane	106-93-4	0.05	0.0075	None	None	0.05	None	None	None	None	None	**0.0075**	**3.1**	**10**
1,2-Dichlorobenzene	95-50-1	600	30	1000	0.7	600	None	None	None	None	2700	**0.7**	0.018	0.075
1,2-Dichloroethane	107-06-2	5	0.17	9.9	100	5	None	None	None	None	None	**0.17**	**0.31**	**1**
1,2-Dichloropropane	78-87-5	5	0.82	0.9	None	5	None	None	None	None	None	**0.82**	0.2	0.5
1,3,5-Trimethylbenzene	108-67-8	None	6	None	None	None	None	None	None	None	None	**6**	0.31	1
1,3-Dichlorobenzene	541-73-1	None	None	7	71	None	None	None	None	None	400	**7**	0.31	1
1,3-Dichloropropane	142-28-9	None	37	None	None	None	None	None	None	None	None	**37**	0.31	1
1,4-Dichlorobenzene	106-46-7	75	0.48	300	15	75	None	None	None	None	400	**0.48**	*0.15*	*0.5*
2,2-Dichloropropane	594-20-7	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	None	560	None	7200	None	None	None	None	None	None	**560**	0.15	0.5
2-Chlorotoluene	95-49-8	None	24	None	None	None	None	None	None	None	None	**24**	0.31	1
2-Hexanone	591-78-6	None	3.8	None	99	None	None	None	None	None	None	**3.8**	*3.1*	*10*
4-Chlorotoluene	106-43-4	None	25	None	None	None	None	None	None	None	None	**25**	0.31	1
4-Isopropyltoluene	99-87-6	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
4-Methyl-2-pentanone (MIBK)	108-10-1	None	630	None	170	None	None	None	None	None	None	**170**	3.1	10
Acetone	67-64-1	None	1800	None	1500	None	None	None	None	None	None	**1500**	3.1	10
Benzene	71-43-2	5	0.46	0.58	46	5	None	None	None	None	None	**0.46**	0.12	0.4
Bromobenzene	108-86-1	None	6.2	None	None	None	None	None	None	None	None	**6.2**	0.31	1
Bromochloromethane	74-97-5	None	8.3	None	None	None	None	None	None	None	None	**8.3**	0.31	1
Bromodichloromethane	75-27-4	80	0.13	0.95	None	None	None	None	None	None	None	**0.13**	**0.15**	**0.5**

Table 6B - Groundwater Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
Bromoform	75-25-2	80	3.3	7	320	None	None	None	None	None	None	**3.3**	0.31	1
Bromomethane	74-83-9	None	0.75	100	1300	None	None	None	None	None	48.0	**0.75**	**3**	**6**
Carbon disulfide	75-15-0	None	81	None	0.92	None	None	None	None	None	None	**0.92**	**3.1**	**10**
Carbon tetrachloride	56-23-5	5	0.46	0.4	9.8	5	None	None	None	None	None	**0.4**	*0.31*	*1*
Chlorobenzene	108-90-7	100	7.8	100	1.3	None	None	None	None	None	680	**1.3**	0.15	0.5
Chloroethane	75-00-3	None	830	None	None	None	None	None	None	None	None	**830**	0.31	1
Chloroform	67-66-3	80	0.22	60	1.8	None	None	None	None	None	None	**0.22**	**0.31**	**1**
Chloromethane	74-87-3	None	19	None	None	None	None	None	None	None	None	**19**	0.31	1
cis-1,2-Dichloroethene	156-59-2	70	2.5	None	590	70	None	None	None	None	None	**2.5**	0.31	1
cis-1,3-Dichloropropene	10061-01-5	None	None	None	0.055	None	None	None	None	None	None	**0.055**	**0.31**	**1**
Dibromochloromethane	124-48-1	80	0.87	0.8	None	None	None	None	None	None	None	**0.8**	0.15	0.5
Dibromomethane	74-95-3	None	0.83	None	None	None	None	None	None	None	None	**0.83**	*0.31*	*1*
Dichlorodifluoromethane	75-71-8	None	20	None	None	None	None	None	None	None	None	**20**	0.31	1
Ethylbenzene	100-41-4	700	1.5	68	7.3	700	None	None	None	None	3100	**1.5**	0.31	1
Isopropylbenzene	98-82-8	None	45	None	None	None	None	None	None	None	None	**45**	0.31	1
m&p-Xylene	179601-23-1	10,000^	19^	None	1.8	10,000^	None	None	None	None	None	**1.8**	*0.62*	*2*
Methyl tert-butyl ether (MTBE)	1634-04-4	None	14	None	10000	None	None	None	None	None	None	**14**	3.1	10
Methylene chloride	75-09-2	5	11	20	98.1	5	None	None	None	None	None	**5**	*3.1*	*10*
n-Butylbenzene	104-51-8	None	100	None	None	None	None	None	None	None	None	**100**	0.31	1
n-Propylbenzene	103-65-1	None	66	None	None	None	None	None	None	None	None	**66**	0.31	1
o-Xylene	95-47-6	None	19	None	13	None	None	None	None	None	None	**13**	0.31	1
sec-Butylbenzene	135-98-8	None	200	None	None	None	None	None	None	None	None	**200**	0.31	1
Styrene	100-42-5	100	120	None	72	100	None	None	None	None	None	**72**	0.31	1
tert-Butylbenzene	98-06-6	None	69	None	None	None	None	None	None	None	None	**69**	0.31	1
Tetrachloroethene	127-18-4	5	4.1	10	50	5	None	None	None	None	None	**4.1**	0.31	1
Toluene	108-88-3	1000	110	57	2	1000	None	None	None	None	6800	**2**	0.31	1
trans-1,2-Dichloroethene	156-60-5	100	6.8	100	590	100	None	None	None	None	700	**6.8**	0.31	1
trans-1,3-Dichloropropene	10061-02-6	None	None	None	0.055	None	None	None	None	None	None	**0.055**	**0.31**	**1**
Trichloroethene	79-01-6	5	0.28	0.6	21	5	None	None	None	None	None	**0.28**	**0.31**	**1**
Trichlorofluoromethane	75-69-4	None	520	None	None	None	None	None	None	None	None	**520**	0.31	1
Vinyl chloride	75-01-4	2	0.019	0.022	None	2	None	None	None	None	None	**0.019**	**0.05**	**0.15**

Sources:

[1] EPA Maximum Contaminant Level Regional Screening Level (RSL) (TR=1E-06 and HQ=0.1), November 2023

[2] EPA Tap Water Regional Screening Level (RSL) for (TR=1E-06 and HQ=0.1), November 2023

[3] National Recommended Water Quality Criteria - Human Health for consumption of water and organisms

[4] NPS Lowest SLERA COPEC Ecological Screening Level for Surface Water, November 2018

[5] State of Alaska Department of Environmental Conservation Drinking Water, 2022

[6] State of Alaska Department of Environmental Conservation Stockwater, 2022

[7] State of Alaska Department of Environmental Conservation Irrigation Water, 2022

[8] State of Alaska Department of Environmental Conservation Aquatic Life for Freshwater (Acute), 2022

[9] State of Alaska Department of Environmental Conservation Aquatic Life for Freshwater (Chronic), 2022

[10] State of Alaska Department of Environmental Conservation Human Health for Consumption of Water and Organisms, 2022

^ Xylenes screening level reported for m&p-xylene

*Hardness-dependent

Notes:

Black cell indicates MDL and QL are greater than the Project Action Limit.

Italicized text in grey cell indicates QL is greater than, but MDL is less than the Project Action Level.

MDL = Method detection limit

QL = Quantitation limit

µg/L = micrograms per liter

Table 6C - Surface Water Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
Metals (200.8 low level)														
Aluminum	7429-90-5	None	2000	None	5	None	None	5000	750	87	None	**5**	0.62	2
Antimony	7440-36-0	6	0.78	5.6	30	6	None	None	None	None	14	**0.78**	0.015	0.05
Arsenic	7440-38-2	10	0.052	0.018	3.1	10	50	100	340	150	None	**0.018**	**0.2**	**0.8**
Barium	7440-39-3	2,000	380	1,000	3.9	2000	None	None	None	None	None	**3.9**	0.04	0.25
Beryllium	7440-41-7	4	2.5	None	0.66	4	None	100	None	None	None	**0.66**	0.025	0.05
Cadmium	7440-43-9	5	0.18	None	0.07	5	10	10	*	*	None	**0.07**	0.015	0.05
Calcium	7440-70-2	None	None	None	116,000	None	None	None	None	None	None	**116000**	15	50
Chromium (total)	7440-47-3	100	None	None	None	100	None	100	None	None	None	**100**	0.15	0.5
Cobalt	7440-48-4	None	0.6	None	3	None	None	50	None	None	None	**0.6**	0.01	0.025
Copper	7440-50-8	1300	80	1300	0.23	None	None	200	*	*	1300	**0.23**	*0.2*	*0.5*
Iron	7439-89-6	None	1,400	None	158	None	None	5000	None	1000	None	**158**	6.2	20
Lead	7439-92-1	15	15	None	0.92	None	50	5,000	*	*	None	**0.92**	0.031	0.1
Magnesium	7439-95-4	None	None	None	82,000	None	None	None	None	None	None	**82000**	6.2	20
Manganese	7439-96-5	None	43	50	112	300	None	200	None	None	None	**43**	0.031	0.1
Nickel	7440-02-0	None	39	610	5	None	None	200	*	*	610	**5**	0.15	0.62
Potassium	7440-09-7	None	None	None	53,000	None	None	None	None	None	None	**53000**	15	50
Selenium	7782-49-2	50	10	170	1	50	10	20	None	5	170	**1**	0.31	1
Silver	7440-22-4	None	9.4	None	0.067	None	None	None	*	None	None	**0.067**	0.0062	0.02
Sodium	7440-23-5	None	None	None	680,000	None	None	None	None	None	None	**680000**	31	100
Thallium	7440-28-0	2	0.02	0.24	0.03	2	None	None	None	None	1.7	**0.02**	0.0062	0.02
Vanadium	7440-62-2	None	8.6	None	19	None	None	100	None	None	None	**8.6**	0.31	1
Zinc	7440-66-6	None	600	7400	30	None	None	2000	*	*	9100	**30**	3.1	10
Mercury (1631E)														
Mercury	7439-97-6	2	0.063	None	0.026	2	None	None	2.4	0.012	0.05	**0.012**	0.0005	0.001
Non-PAH SVOCs (8270E)														
2,2'-oxybis (1-chloropropane)	108-60-1	None	71	200	None	None	None	None	None	None	None	**71**	0.74	5
2,4,5-Trichlorophenol	95-95-4	None	120	300	18	None	None	None	None	None	2600	**18**	0.74	5
2,4,6-Trichlorophenol	88-06-2	None	1.2	1.5	18	None	None	None	None	None	None	**1.2**	*0.75*	*5*
2,4-Dichlorophenol	120-83-2	None	4.6	10	0.2	None	None	None	None	None	93	**0.2**	**0.84**	**5**
2,4-Dimethylphenol	105-67-9	None	36	100	None	None	None	None	None	None	540	**36**	0.74	5
2,4-Dinitrophenol	51-28-5	None	3.9	10	None	None	None	None	None	None	70	**3.9**	**5**	**25**
2,4-Dinitrotoluene	121-14-2	None	0.24	0.049	65	None	None	None	None	None	None	**0.049**	**0.81**	**5**
2,6-Dinitrotoluene	606-20-2	None	0.049	None	230	None	None	None	None	None	None	**0.049**	**0.71**	**5**
2-Chloronaphthalene	91-58-7	None	75	800	None	None	None	None	None	None	1700	**75**	0.5	5
2-Chlorophenol	95-57-8	None	9.1	30	7	None	None	None	None	None	120	**7**	0.63	5
2-Methylphenol	95-48-7	None	93	None	13	None	None	None	None	None	None	**13**	0.85	5
2-Nitroaniline	88-74-4	None	19	None	None	None	None	None	None	None	None	**19**	1.8	5
2-Nitrophenol	88-75-5	None	None	None	60	None	None	None	None	None	None	**60**	0.85	5

Table 6C - Surface Water Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
3&4-Methylphenol	108-39-4 & 106-44-5	None	37	None	None	None	None	None	None	None	None	**37**	0.98	5
3,3´-Dichlorobenzidine	91-94-1	None	0.13	0.049	None	None	None	None	None	None	None	**0.049**	**0.64**	**5**
3-Nitroaniline	99-09-2	None	None	None	None	None	None	None	None	None	None	**None**	0.88	5
4,6-Dinitro-2-methylphenol	534-52-1	None	0.15	2	None	None	None	None	None	None	13.4	**0.15**	**2**	**10**
4-Bromophenyl phenyl ether	101-55-3	None	None	None	1.5	None	None	None	None	None	None	**1.5**	*0.85*	*5*
4-Chloro-3-methylphenol	59-50-7	None	140	500	None	None	None	None	None	None	None	**140**	0.59	5
4-Chloroaniline	106-47-8	None	0.37	None	None	None	None	None	None	None	None	**0.37**	**0.63**	**5**
4-Chlorophenyl phenyl ether	7005-72-3	None	None	None	None	None	None	None	None	None	None	**None**	0.54	5
4-Nitroaniline	100-01-6	None	3.8	None	None	None	None	None	None	None	None	**3.8**	*1.2*	*5*
4-Nitrophenol	100-02-7	None	None	None	60	None	None	None	None	None	None	**60**	5	25
bis(-2-chloroethoxy)Methane	111-91-1	None	5.9	None	None	None	None	None	None	None	None	**5.9**	0.81	5
bis(-2-chloroethyl)Ether	111-44-4	None	0.014	0.03	None	None	None	None	None	None	None	**0.014**	**0.73**	**5**
bis(2-ethylhexyl)Phthalate	117-81-7	6	5.6	0.32	3	6	None	None	None	None	None	**0.32**	**1**	**5**
Butylbenzylphthalate	85-68-7	None	16	0.1	19	None	None	None	None	None	3000	**0.1**	**1**	**5**
Carbazole	86-74-8	None	None	None	None	None	None	None	None	None	None	**None**	0.6	5
Dibenzofuran	132-64-9	None	0.79	None	3.7	None	None	None	None	None	None	**0.79**	*0.6*	*5*
Diethyl phthalate	84-66-2	None	1500	600	210	None	None	None	None	None	23000	**210**	1	5
Dimethyl phthalate	131-11-3	None	None	2000	3	None	None	None	None	None	313000	**3**	*1*	*5*
Di-n-butyl phthalate	84-74-2	None	90	20	19	None	None	None	None	None	2700	**19**	1	5
Di-n-octyl phthalate	117-84-0	None	20	None	3	None	None	None	None	None	None	**3**	*1*	*5*
Hexachlorobenzene	118-74-1	1	0.0098	0.000079	None	1	None	None	None	None	None	**0.000079**	**0.69**	**5**
Hexachlorobutadiene	87-68-3	None	0.14	0.01	1.3	None	None	None	None	None	None	**0.01**	**0.5**	**5**
Hexachlorocyclopentadiene	77-47-4	50	0.041	4	None	50	None	None	None	None	240	**0.041**	**1.8**	**5**
Hexachloroethane	67-72-1	None	0.33	0.1	12	None	None	None	None	None	None	**0.1**	**1.6**	**5**
Isophorone	78-59-1	None	78	34	None	None	None	None	None	None	None	**34**	0.78	5
Nitrobenzene	98-95-3	None	0.14	10	550	None	None	None	None	None	17	**0.14**	**0.93**	**5**
n-Nitroso-di-n-propylamine	621-64-7	None	0.011	0.005	None	None	None	None	None	None	None	**0.005**	**0.67**	**5**
N-Nitrosodiphenylamine	86-30-6	None	12	3.3	40	None	None	None	None	None	None	**3.3**	*0.81*	*5*
Pentachlorophenol	87-86-5	1	0.041	0.03	0.5	1	None	None	None	None	None	**0.03**	**5**	**25**
Phenol	108-95-2	None	580	4000	4	None	None	None	None	None	21000	**4**	*0.5*	*5*
PAHs (8270D-SIM)														
1-Methylnaphthalene	90-12-0	None	1.1	None	None	None	None	None	None	None	None	**1.1**	0.0037	0.0125
2-Methylnaphthalene	91-57-6	None	3.6	None	330	None	None	None	None	None	None	**3.6**	0.0037	0.0125
Acenaphthene	83-32-9	None	53	70	5.8	None	None	None	None	None	1200	**5.8**	0.0037	0.0125
Acenaphthylene	208-96-8	None	None	None	4800	None	None	None	None	None	None	**4800**	0.0037	0.0125
Anthracene	120-12-7	None	180	300	0.012	None	None	None	None	None	9600	**0.012**	*0.0037*	*0.0125*
Benzo(a)anthracene	56-55-3	None	0.03	0.0012	0.018	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Benzo(a)pyrene	50-32-8	0.2	0.025	0.00012	0.014	0.2	None	None	None	None	None	**0.00012**	**0.0015**	**0.005**
Benzo(b)fluoranthene	205-99-2	None	0.25	0.0012	9	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Benzo(g,h,i)perylene	191-24-2	None	None	None	7.6	None	None	None	None	None	None	**7.6**	0.0037	0.0125
Benzo(k)fluoranthene	207-08-9	None	2.5	0.012	0.0041	None	None	None	None	None	None	**0.0041**	*0.0037*	*0.0125*
Chrysene	218-01-9	None	25	0.12	0.0018	None	None	None	None	None	None	**0.0018**	**0.0037**	**0.0125**
Dibenzo(a,h)anthracene	53-70-3	None	0.025	0.00012	0.0034	None	None	None	None	None	None	**0.00012**	**0.0015**	**0.005**
Fluoranthene	206-44-0	None	80	20	0.04	None	None	None	None	None	300	**0.04**	0.0037	0.0125
Fluorene	86-73-7	None	29	50	3	None	None	None	None	None	1300	**3**	0.0037	0.0125
Indeno(1,2,3-cd)pyrene	193-39-5	None	0.25	0.0012	4.3	None	None	None	None	None	None	**0.0012**	**0.0037**	**0.0125**
Naphthalene	91-20-3	None	0.12	None	1.1	None	None	None	None	None	None	**0.12**	0.0078	0.025
Phenanthrene	85-01-8	None	None	None	0.4	None	None	None	None	None	None	**0.4**	0.00775	0.025
Pyrene	129-00-0	None	12	20	0.025	None	None	None	None	None	960	**0.025**	0.0037	0.0125

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VOCs (8260D)														
1,1,1,2-Tetrachloroethane	630-20-6	None	0.57	None	None	None	None	None	None	None	None	**0.57**	0.15	0.5
1,1,1-Trichloroethane	71-55-6	200	800	10000	11	200	None	None	None	None	None	**11**	0.31	1
1,1,2,2-Tetrachloroethane	79-34-5	None	0.076	0.2	610	None	None	None	None	None	None	**0.076**	**0.15**	**0.5**
1,1,2-Trichloroethane	79-00-5	5	0.041	0.55	1200	5	None	None	None	None	None	**0.041**	**0.12**	**0.4**
1,1-Dichloroethane	75-34-3	None	2.8	None	47	None	None	None	None	None	None	**2.8**	0.31	1
1,1-Dichloroethene	75-35-4	7	28	300	25	7	None	None	None	None	None	**7**	0.31	1
1,1-Dichloropropene	563-58-6	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
1,2,3-Trichlorobenzene	87-61-6	None	0.7	None	8	None	None	None	None	None	None	**0.7**	*0.31*	*1*
1,2,3-Trichloropropane	96-18-4	None	0.00075	None	None	None	None	None	None	None	None	**0.00075**	**0.31**	**1**
1,2,4-Trichlorobenzene	120-82-1	70	0.4	0.071	24	70	None	None	None	None	260	**0.071**	**0.31**	**1**
1,2,4-Trimethylbenzene	95-63-6	None	5.6	None	None	None	None	None	None	None	None	**5.6**	0.31	1
1,2-Dibromo-3-chloropropane	96-12-8	0.2	0.00033	None	None	None	None	None	None	None	None	**0.00033**	**3.1**	**10**
1,2-Dibromoethane	106-93-4	0.05	0.0075	None	None	0.05	None	None	None	None	None	**0.0075**	**3.1**	**10**
1,2-Dichlorobenzene	95-50-1	600	30	1000	0.7	600	None	None	None	None	2700	**0.7**	0.018	0.075
1,2-Dichloroethane	107-06-2	5	0.17	9.9	100	5	None	None	None	None	None	**0.17**	**0.31**	**1**
1,2-Dichloropropane	78-87-5	5	0.82	0.9	None	5	None	None	None	None	None	**0.82**	0.2	0.5
1,3,5-Trimethylbenzene	108-67-8	None	6	None	None	None	None	None	None	None	None	**6**	0.31	1
1,3-Dichlorobenzene	541-73-1	None	None	7	71	None	None	None	None	None	400	**7**	0.31	1
1,3-Dichloropropane	142-28-9	None	37	None	None	None	None	None	None	None	None	**37**	0.31	1
1,4-Dichlorobenzene	106-46-7	75	0.48	300	15	75	None	None	None	None	400	**0.48**	*0.15*	*0.5*
2,2-Dichloropropane	594-20-7	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	None	560	None	7200	None	None	None	None	None	None	**560**	0.15	0.5
2-Chlorotoluene	95-49-8	None	24	None	None	None	None	None	None	None	None	**24**	0.31	1
2-Hexanone	591-78-6	None	3.8	None	99	None	None	None	None	None	None	**3.8**	*3.1*	*10*
4-Chlorotoluene	106-43-4	None	25	None	None	None	None	None	None	None	None	**25**	0.31	1
4-Isopropyltoluene	99-87-6	None	None	None	None	None	None	None	None	None	None	**None**	0.31	1
4-Methyl-2-pentanone (MIBK)	108-10-1	None	630	None	170	None	None	None	None	None	None	**170**	3.1	10
Acetone	67-64-1	None	1800	None	1500	None	None	None	None	None	None	**1500**	3.1	10
Benzene	71-43-2	5	0.46	0.58	46	5	None	None	None	None	None	**0.46**	0.12	0.4
Bromobenzene	108-86-1	None	6.2	None	None	None	None	None	None	None	None	**6.2**	0.31	1
Bromochloromethane	74-97-5	None	8.3	None	None	None	None	None	None	None	None	**8.3**	0.31	1
Bromodichloromethane	75-27-4	80	0.13	0.95	None	None	None	None	None	None	None	**0.13**	**0.15**	**0.5**
Bromoform	75-25-2	80	3.3	7	320	None	None	None	None	None	None	**3.3**	0.31	1
Bromomethane	74-83-9	None	0.75	100	1300	None	None	None	None	None	48.0	**0.75**	**3**	**6**
Carbon disulfide	75-15-0	None	81	None	0.92	None	None	None	None	None	None	**0.92**	**3.1**	**10**
Carbon tetrachloride	56-23-5	5	0.46	0.4	9.8	5	None	None	None	None	None	**0.4**	*0.31*	*1*
Chlorobenzene	108-90-7	100	7.8	100	1.3	None	None	None	None	None	680	**1.3**	0.15	0.5
Chloroethane	75-00-3	None	830	None	None	None	None	None	None	None	None	**830**	0.31	1
Chloroform	67-66-3	80	0.22	60	1.8	None	None	None	None	None	None	**0.22**	**0.31**	**1**
Chloromethane	74-87-3	None	19	None	None	None	None	None	None	None	None	**19**	0.31	1
cis-1,2-Dichloroethene	156-59-2	70	2.5	None	590	70	None	None	None	None	None	**2.5**	0.31	1
cis-1,3-Dichloropropene	10061-01-5	None	None	None	0.055	None	None	None	None	None	None	**0.055**	**0.31**	**1**
Dibromochloromethane	124-48-1	80	0.87	0.8	None	None	None	None	None	None	None	**0.8**	0.15	0.5
Dibromomethane	74-95-3	None	0.83	None	None	None	None	None	None	None	None	**0.83**	*0.31*	*1*
Dichlorodifluoromethane	75-71-8	None	20	None	None	None	None	None	None	None	None	**20**	0.31	1
Ethylbenzene	100-41-4	700	1.5	68	7.3	700	None	None	None	None	3100	**1.5**	0.31	1
Isopropylbenzene	98-82-8	None	45	None	None	None	None	None	None	None	None	**45**	0.31	1
m&p-Xylene	179601-23-1	10,000^	19^	None	1.8	10,000^	None	None	None	None	None	**1.8**	*0.62*	*2*

Table 6C - Surface Water Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (µg/L)										Project Action Level (µg/L)	Achievable Laboratory Limits (µg/L)	
		Federal Maximum Contaminant Level[1]	Federal Tap Water Screening Level[2]	NRWQC for Human Health[3]	NPS Lowest Freshwater COPEC Ecological Screening Value[4]	Alaska Drinking Water[5]	Alaska Stockwater[6]	Alaska Irrigation[7]	Alaska Aquatic Life for Freshwater (Acute)[8]	Alaska Aquatic Life for Freshwater (Chronic)[9]	Alaska Human Health for Consumption of Water & Organisms[10]		MDLs	QLs
Methyl tert-butyl ether (MTBE)	1634-04-4	None	14	None	10000	None	None	None	None	None	None	**14**	3.1	10
Methylene chloride	75-09-2	5	11	20	98.1	5	None	None	None	None	None	**5**	*3.1*	*10*
n-Butylbenzene	104-51-8	None	100	None	None	None	None	None	None	None	None	**100**	0.31	1
n-Propylbenzene	103-65-1	None	66	None	None	None	None	None	None	None	None	**66**	0.31	1
o-Xylene	95-47-6	None	19	None	13	None	None	None	None	None	None	**13**	0.31	1
sec-Butylbenzene	135-98-8	None	200	None	None	None	None	None	None	None	None	**200**	0.31	1
Styrene	100-42-5	100	120	None	72	100	None	None	None	None	None	**72**	0.31	1
tert-Butylbenzene	98-06-6	None	69	None	None	None	None	None	None	None	None	**69**	0.31	1
Tetrachloroethene	127-18-4	5	4.1	10	50	5	None	None	None	None	None	**4.1**	0.31	1
Toluene	108-88-3	1000	110	57	2	1000	None	None	None	None	6800	**2**	0.31	1
trans-1,2-Dichloroethene	156-60-5	100	6.8	100	590	100	None	None	None	None	700	**6.8**	0.31	1
trans-1,3-Dichloropropene	10061-02-6	None	None	None	0.055	None	None	None	None	None	None	**0.055**	**0.31**	**1**
Trichloroethene	79-01-6	5	0.28	0.6	21	5	None	None	None	None	None	**0.28**	**0.31**	**1**
Trichlorofluoromethane	75-69-4	None	520	None	None	None	None	None	None	None	None	**520**	0.31	1
Vinyl chloride	75-01-4	2	0.019	0.022	None	2	None	None	None	None	None	**0.019**	**0.05**	**0.15**

Sources:

[1]EPA Maximum Contaminant Level Regional Screening Level (RSL) (TR=1E-06 and HQ=0.1), November 2021

[2]EPA Tap Water Regional Screening Level (RSL) for (TR=1E-06 and HQ=0.1), November 2021

[3]National Recommended Water Quality Criteria - Human Health for consumption of water and organisms

[4]NPS Lowest SLERA COPEC Ecological Screening Level for Surface Water, November 2018

[5]State of Alaska Department of Environmental Conservation Drinking Water, 2022

[6]State of Alaska Department of Environmental Conservation Stockwater, 2022

[7]State of Alaska Department of Environmental Conservation Irrigation Water, 2022

[8]State of Alaska Department of Environmental Conservation Aquatic Life for Freshwater (Acute), 2022

[9]State of Alaska Department of Environmental Conservation Aquatic Life for Freshwater (Chronic), 2022

[10]State of Alaska Department of Environmental Conservation Human Health for Consumption of Water and Organisms, 2022

^ Xylenes screening level reported for m&p-xylene

*Hardness-dependent

Notes:

Black cell indicates MDL and QL are greater than the Project Action Limit.

Italicized text in grey cell indicates QL is greater than, but MDL is less than the Project Action Level.

MDL = Method detection limit

QL = Quantitation limit

µg/L = micrograms per liter

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
Metals (6020B)								
Aluminum	7429-90-5	7700	110000	25000	None	**7700**	7	20
Antimony	7440-36-0	3.1	47	None	41	**3.1**	0.31	1
Arsenic	7440-38-2	0.68	3	9.7	8.8	**0.68**	*0.31*	*1*
Barium	7440-39-3	1,500	22,000	150	20000	**150**	0.094	0.3
Beryllium	7440-41-7	16	230	None	200	**16**	0.031	0.1
Cadmium	7440-43-9	0.71	10	0.58	92	**0.58**	0.062	0.2
Calcium	7440-70-2	None	None	None	None	**None**	15	50
Chromium (total)	7440-47-3	None	None	36	None	**36**	0.31	1
Cobalt	7440-48-4	2.3	35	None	None	**2.3**	0.15	0.5
Copper	7440-50-8	310	4,700	28	4,100	**28**	0.18	0.6
Iron	7439-89-6	5500	82000	20000	None	**5500**	15	50
Lead	7439-92-1	100	800	35	400	**35**	0.062	0.2
Magnesium	7439-95-4	None	None	None	None	**None**	15	50
Manganese	7439-96-5	180	2600	460	2700	**180**	0.062	0.2
Mercury > 0.3 mg/kg[5]	7439-97-6	1.1	4.6	0.18	3.1	**0.18**	*0.1*	*0.3*
Nickel	7440-02-0	140	1800	20	2000	**20**	0.062	0.2
Potassium	7440-09-7	None	None	None	None	**None**	31	100
Selenium	7782-49-2	39	580	0.72	510	**0.72**	*0.62*	*2*
Silver	7440-22-4	39	580	0.5	510	**0.5**	0.15	0.5
Sodium	7440-23-5	None	None	None	None	**None**	31	100
Thallium	7440-28-0	0.078	1.2	None	1	**0.078**	*0.062*	*0.2*
Vanadium	7440-62-2	39	580	None	510	**39**	1.5	5
Zinc	7440-66-6	2300	35000	98	30000	**98**	0.78	2.5
Mercury (1631E)								
Mercury	7439-97-6	1.1	4.6	0.18	3.1	**0.18**	0.000078	0.00025

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
Non-PAH SVOCs (8270E)								
2,2'-oxybis (1-chloropropane)	108-60-1	310	4700	None	None	**310**	0.00063	0.005
2,4,5-Trichlorophenol	95-95-4	630	8200	0.29	8200	**0.29**	0.0008	0.005
2,4,6-Trichlorophenol	88-06-2	6.3	82	None	82	**6.3**	0.00058	0.005
2,4-Dichlorophenol	120-83-2	19	250	None	250	**19**	0.00057	0.005
2,4-Dimethylphenol	105-67-9	130	1600	None	1600	**130**	0.0013	0.005
2,4-Dinitrophenol	51-28-5	13	160	None	160	**13**	0.005	0.025
2,4-Dinitrotoluene	121-14-2	1.7	7.4	0.29	23	**0.29**	0.0005	0.005
2,6-Dinitrotoluene	606-20-2	0.36	1.5	None	4.7	**0.36**	0.00065	0.005
2-Chloronaphthalene	91-58-7	480	6000	None	6200	**480**	0.0005	0.005
2-Chlorophenol	95-57-8	39	580	0.027	510	**0.027**	0.00061	0.005
2-Methylphenol	95-48-7	320	4100	0.012	4100	**0.012**	0.0006	0.005
2-Nitroaniline	88-74-4	63	800	None	None	**63**	0.0012	0.005
2-Nitrophenol	88-75-5	None	None	None	None	**None**	0.00054	0.005
3&4-Methylphenol	108-39-4 & 106-44-5	130	1600	None	4100	**130**	0.005	0.025
3,3´-Dichlorobenzidine	91-94-1	1.2	5.1	None	16	**1.2**	0.00082	0.005
3-Nitroaniline	99-09-2	None	None	None	None	**None**	0.0012	0.005
4,6-Dinitro-2-methylphenol	534-52-1	0.51	6.6	None	None	**0.51**	0.00058	0.005
4-Bromophenyl phenyl ether	101-55-3	None	None	0.26	None	**0.26**	0.002	0.01
4-Chloro-3-methylphenol	59-50-7	630	8200	None	None	**630**	0.00052	0.005
4-Chloroaniline	106-47-8	2.7	11	None	35	**2.7**	0.00057	0.005
4-Chlorophenyl phenyl ether	7005-72-3	None	None	None	None	**None**	0.0013	0.005
4-Methylphenol	106-44-5	25	110	None	None	**25**	0.0005	0.005
4-Nitroaniline	100-01-6	None	None	None	None	**None**	0.0014	0.005

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
bis(-2-chloroethoxy)Methane	111-91-1	19	250	None	None	**19**	0.0005	0.005
bis(-2-chloroethyl)Ether	111-44-4	0.23	1	None	2.8	**0.23**	0.00058	0.005
bis(2-ethylhexyl)Phthalate	117-81-7	39	160	453	500	**39**	0.001	0.01
Butylbenzylphthalate	85-68-7	290	1200	0.1	3700	**0.1**	0.001	0.005
Carbazole	86-74-8	None	None	None	None	**None**	0.0007	0.005
Dibenzofuran	132-64-9	7.8	120	0.3	95	**0.3**	0.0005	0.005
Diethyl phthalate	84-66-2	5100	66000	0.6	66000	**0.6**	0.001	0.01
Dimethyl phthalate	131-11-3	None	None	None	66000	**66000**	0.001	0.005
Di-n-butyl phthalate	84-74-2	630	8200	0.011	8200	**0.011**	0.002	0.01
Di-n-octyl phthalate	117-84-0	63	820	17	820	**17**	0.001	0.005
Hexachlorobenzene	118-74-1	0.078	0.96	None	2	**0.078**	0.00051	0.005
Hexachlorobutadiene	87-68-3	1.2	5.3	0.7	3.3	**0.6981**	0.00051	0.005
Hexachlorocyclopentadiene	77-47-4	0.18	0.75	None	1.4	**0.18**	0.001	0.005
Hexachloroethane	67-72-1	1.8	8	0.21	17	**0.2136**	0.00059	0.005
Isophorone	78-59-1	570	2400	None	7400	**570**	0.0005	0.005
Nitrobenzene	98-95-3	5.1	22	None	43	**5.1**	0.0005	0.005
n-Nitroso-di-n-propylamine	621-64-7	0.078	0.33	None	1	**0.078**	0.0005	0.005
N-Nitrosodiphenylamine	86-30-6	110	470	0.52	1400	**0.52**	0.00054	0.005
Pentachlorophenol	87-86-5	1	4	0.01	13	**0.01**	*0.005*	*0.025*
Phenol	108-95-2	1900	25000	0.0012	25000	**0.0012**	*0.0005*	*0.005*
PAHs (8270D-SIM)								
1-Methylnaphthalene	90-12-0	18	73	None	68	**18**	0.00625	0.025
2-Methylnaphthalene	91-57-6	24	300	0.076	310	**0.076**	0.00625	0.025
Acenaphthene	83-32-9	360	4500	0.076	4600	**0.076**	0.00625	0.025
Acenaphthylene	208-96-8	None	None	0.076	2300	**0.076**	0.00625	0.025

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
Anthracene	120-12-7	1800	23000	0.01	23000	**0.01**	*0.00625*	*0.025*
Benzo(a)anthracene	56-55-3	1.1	21	0.015	14	**0.015**	*0.00625*	*0.025*
Benzo(a)pyrene	50-32-8	0.11	2.1	0.032	1.5	**0.032**	0.00625	0.025
Benzo(b)fluoranthene	205-99-2	1.1	21	0.19	15	**0.19**	0.00625	0.025
Benzo(g,h,i)perylene	191-24-2	None	None	0.016	2300	**0.016**	*0.00625*	*0.025*
Benzo(k)fluoranthene	207-08-9	11	210	0.24	150	**0.24**	0.00625	0.025
Chrysene	218-01-9	110	2100	0.026	1500	**0.026**	0.00625	0.025
Dibenzo(a,h)anthracene	53-70-3	0.11	2.1	0.033	1.5	**0.033**	0.00625	0.025
Fluoranthene	206-44-0	240	3000	0.031	3100	**0.031**	0.00625	0.025
Fluorene	86-73-7	240	3000	0.01	3100	**0.01**	*0.00625*	*0.025*
Indeno(1,2,3-cd)pyrene	193-39-5	1.1	21	0.017	15	**0.017**	*0.00625*	*0.025*
Naphthalene	91-20-3	2	8.6	0.014	29	**0.014**	*0.005*	*0.02*
Phenanthrene	85-01-8	None	None	0.019	2300	**0.019**	*0.00625*	*0.025*
Pyrene	129-00-0	180	2300	0.044	2300	**0.044**	0.00625	0.025
VOCs (8260D)								
1,1,1,2-Tetrachloroethane	630-20-6	2	8.8	None	21	**2**	0.0062	0.02
1,1,1-Trichloroethane	71-55-6	810	3600	0.012	360	**0.012**	*0.0078*	*0.025*
1,1,2,2-Tetrachloroethane	79-34-5	0.6	2.7	0.57	6.1	**0.56913**	0.00062	0.002
1,1,2-Trichloroethane	79-00-5	0.15	0.63	0.6	1.6	**0.15**	0.0005	0.001
1,1-Dichloroethane	75-34-3	3.6	16	0.0082	46	**0.0082**	*0.0078*	*0.025*
1,1-Dichloroethene	75-35-4	23	100	0.015	330	**0.015**	*0.0078*	*0.025*
1,1-Dichloropropene	563-58-6	None	None	None	None	**None**	0.0078	0.025
1,2,3-Trichlorobenzene	87-61-6	6.3	93	0.14	81	**0.14**	0.03	0.1
1,2,3-Trichloropropane	96-18-4	0.0051	0.11	None	0.066	**0.0051**	0.00062	0.002
1,2,4-Trichlorobenzene[l]	120-82-1	5.8	26	0.011	45	**0.011**	*0.0078*	*0.025*

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
1,2,4-Trimethylbenzene	95-63-6	30	180	None	43	**30**	0.03	0.1
1,2-Dibromo-3-chloropropane	96-12-8	0.0053	0.064	None	None	**0.0053**	**0.031**	**0.1**
1,2-Dibromoethane	106-93-4	0.036	0.16	None	0.42	**0.036**	0.00075	0.0015
1,2-Dichlorobenzene[l]	95-50-1	180	930	0.0043	78	**0.0043**	**0.0078**	**0.025**
1,2-Dichloroethane	107-06-2	0.46	2	0.017	5.5	**0.0174**	0.0007	0.002
1,2-Dichloropropane	78-87-5	1.6	6.6	None	17	**1.6**	0.005	0.01
1,3,5-Trimethylbenzene	108-67-8	27	150	None	37	**27**	0.0078	0.025
1,3-Dichlorobenzene[l]	541-73-1	None	None	0.44	62	**0.44**	0.0078	0.025
1,3-Dichloropropane	142-28-9	160	2300	None	None	**160**	0.0031	0.01
1,4-Dichlorobenzene[l]	106-46-7	2.6	11	0.03	21	**0.03**	0.0078	0.025
2,2-Dichloropropane	594-20-7	None	None	None	None	**None**	0.0078	0.025
2-Butanone (Methyl ethyl ketone) (MEK)	78-93-3	2700	19000	0.92	23000	**0.92**	0.078	0.025
2-Chlorotoluene	95-49-8	160	2300	None	None	**160**	0.0078	0.25
2-Hexanone	591-78-6	20	130	0.013	270	**0.013**	**0.06**	**0.025**
4-Chlorotoluene	106-43-4	160	2300	None	None	**160**	0.01	0.12
4-Isopropyltoluene	99-87-6	None	None	None	None	**None**	0.04	0.08
4-Methyl-2-pentanone (MIBK)	108-10-1	3300	14000	0.032	2200	**0.032**	**0.078**	**0.25**
Acetone	67-64-1	7000	110000	0.065	81000	**0.065**	**0.11**	**0.25**
Benzene	71-43-2	1.2	5.1	0.01	11	**0.01**	*0.0039*	*0.0125*
Bromobenzene	108-86-1	29	180	None	160	**29**	0.0078	0.025
Bromochloromethane	74-97-5	15	63	None	None	**15**	0.0078	0.025
Bromodichloromethane	75-27-4	0.29	1.3	None	3.6	**0.29**	0.00062	0.002
Bromoform	75-25-2	19	86	0.28	240	**0.27872**	0.0078	0.025
Bromomethane	74-83-9	0.68	3	None	10	**0.68**	0.008	0.02
Carbon disulfide	75-15-0	77	350	0.00042	500	**0.00042**	**0.031**	**0.1**
Carbon tetrachloride	56-23-5	0.65	2.9	0.017	9.1	**0.017**	0.0039	0.0125
Chlorobenzene	108-90-7	28	130	0.0028	180	**0.0028**	**0.0078**	**0.025**
Chloroethane	75-00-3	540	2300	None	1400	**540**	0.062	0.2

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
Chloroform	67-66-3	0.32	1.4	0.00072	4	**0.00072**	**0.003**	**0.006**
Chloromethane	74-87-3	11	46	None	170	**11**	0.0078	0.025
cis-1,2-Dichloroethene	156-59-2	6.3	37	None	200	**6.3**	0.0078	0.025
cis-1,3-Dichloropropene	10061-01-5	None	None	2.5E-05	None	**2.5E-05**	**0.0039**	**0.0125**
Dibromochloromethane	124-48-1	8.3	39	None	110	**8.3**	0.0015	0.005
Dibromomethane	74-95-3	2.4	9.9	None	31	**2.4**	0.0078	0.025
Dichlorodifluoromethane	75-71-8	8.7	37	None	150	**8.7**	0.03	0.1
Ethylbenzene	100-41-4	5.8	25	0.026	49	**0.026**	0.0078	0.025
Isopropylbenzene	98-82-8	190	990	None	54	**54**	0.0078	0.025
m&p-Xylene	179601-23-1	58^	250^	0.007	None	**0.007**	**0.015**	**0.05**
Methyl tert-butyl ether (MTBE)	1634-04-4	47	210	None	670	**47**	0.031	0.1
Methylene chloride	75-09-2	35	320	0.011	None	**0.011**	**0.031**	**0.1**
n-Butylbenzene	104-51-8	390	5800	None	20	**20**	0.0078	0.025
n-Propylbenzene	103-65-1	380	2400	None	52	**52**	0.0078	0.025
o-Xylene	95-47-6	64	280	0.047	None	**0.047**	0.0078	0.025
sec-Butylbenzene	135-98-8	780	12000	None	28	**28**	0.0078	0.025
Styrene	100-42-5	600	3500	0.56	180	**0.56**	0.0078	0.025
tert-Butylbenzene	98-06-6	780	12000	None	None	**780**	0.0078	0.025
Tetrachloroethene	127-18-4	8.1	39	0.002	68	**0.002**	**0.0039**	**0.0125**
Toluene	108-88-3	490	4700	0.0036	200	**0.0036**	**0.0078**	**0.025**
trans-1,2-Dichloroethene	156-60-5	7	30	0.31	960	**0.31**	0.0078	0.01
trans-1,3-Dichloropropene	10061-02-6	None	None	2.5E-05	None	**2.5E-05**	**0.0039**	**0.0125**
Trichloroethene	79-01-6	0.41	1.9	0.035	None	**0.03486**	*0.0032*	*0.05*
Trichlorofluoromethane	75-69-4	2300	35000	None	980	**980**	0.015	0.0008
Vinyl chloride	75-01-4	0.059	1.7	None	None	**0.059**	0.00025	0.0008

Table 6D - Sediment Screening Levels and Laboratory Specific Detection/Reporting Limits

Analyte	CAS Number	Screening Levels (mg/kg)				Project Action Level (mg/kg)	Achievable Laboratory Limits (mg/kg)	
		Residential RSL[1]	Industrial RSL[2]	NPS Sediment ESV[3]	Alaska Under 40 Inch Zone Soil Cleanup Levels [4]		MDLs	QLs
TOC (9060A mod)								
TOC %		None	None	None	None	**0.05**	0.015	0.05

Sources:

[1] EPA Residential Soil Regional Screening Level (TR 1E-06 and HQ 0.1), November 2023

[2] EPA Industrial Soil Regional Screening Level (TR 1E-06 and HQ 0.1), November 2021

[3] NPS Ecological Screening Level (ESV) Lowest SLERA COPEC Selection Value for Sediment, November 2018

[4] Alaska State Soil Cleanup Levels (Under 40-inch Zone), January 2022

[5] Mercury will be analyzed by 6020B if detected > 0.3 mg/kg in soil/sediment; if non-detect by 6020B, mercury will be analyzed by 1631E.

[^] Xylenes screening level reported for m&p-xylene

Notes:

Black cell indicates MDL and QL are greater than the Project Action Limit.

Italicized text in grey cell indicates MDL is less than the Project Action Level but the QL is greater than the Project Action Level.

MDL = Method detection limit

QL = Quantitation limit

mg/kg = milligram per kilogram

TABLE 7 LABORATORY QUALITY CONTROL AND DATA VALIDATION CRITERIA

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Matrix: All	**Analytical Group: Metals**			**SOP: 361r18, 375r07, 342r28, 345r12, 354r15, 385r10, ENV-SOP-MTJL-0221**	
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 field duplicate per 10 discrete samples	Not applicable	Review sampling procedures	RPD ≤ 30% for aqueous samples; RPD ≤ 50% for solid samples
Equipment Blank	Measure of potential field contamination	1 per 20 samples per sampling methodology	Not applicable	Review decontamination procedures	< QL
Method Blank (MB)	Analyte-free matrix that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	No analytes detected > QL	Investigate source of contamination. Samples associated with the contaminated blank shall be reanalyzed or may be reported as qualified if sample concentrations are > 10x blank level	< QL
Internal Standards (ICP-MS only)	Added to each sample extract prior to analysis and used for quantitation of targets and to ensure accuracy of analysis.	Every sample and QC	Recovery 70%- 130% of the intensity of the cal. Blank (SOP limit)	5-fold dilution; reanalyze	Recovery within 70-130%

[1] Data validation (DV) criteria are based on the USEPA *National Functional Guidelines for Inorganic Superfund Methods Data Review*, Office of Superfund Remediation and Technology Innovation (OSRTI), USEPA, OLEM 8240.0-51, EPA-540-R-20-005, November 2020 (NFG). For validation purposes, it is assumed the laboratory will perform calibration, maintenance and corrective actions per the specified method and laboratory method-specific SOPs. Professional judgement may be used during data validation, as allowed by the NFG, to take alternate DV actions based on cumulative QA/QC issues.

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures. LCS associated with Sediments may be a Standard Reference Material (SRM) from a verified source.	1 per prep batch of up to 20 samples	Recovery limits 80%-120% or vendor SRM limits	Reanalyze and if acceptable, continue. If unacceptable, re-digest & reanalyze LCS with associated field samples. Analytes in the LCS that fail high and are non-detect in the samples can be reported.	Aqueous: Recovery within 80-120% Solid: vendor SRM control limits
LCSD	Matrix-matched blank duplicate matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures. LCS associated with Sediments may be a Standard Reference Material (SRM) from a verified source.	1 per prep batch of up to 20 samples (only required if no MSD or lab duplicate)	RPD ≤ 20%	Flag data	RPD ≤ 20%
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 set per prep batch of up to 20 samples (MSD not required if laboratory duplicate performed)	Recovery limits 75%-125%; Aqueous: RPD ≤ 20%; Solid: RPD ≤35%	Flag data	Recovery within 75%-125%; Aqueous: RPD ≤ 20%; Solid: RPD ≤ 35%

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
ISM Replicates A,B,C	Replicate ISM samples will be collected with a total of approximately 1 kilogram soil each replicate to assess overall precision and representativeness of ISM sample results.	3 replicates to be collected / analyzed in each DU	None	No lab corrective action unless reanalysis requested by project manager. Potential data usability issue.	%RSD ≤ 35%
Matrix: All	**Analytical Group: Metals - Mercury**			**SOP: 354r15**	
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 field duplicate per 10 discrete samples	Not applicable	Review sampling procedures	Aqueous: RPD ≤ 30% Solid: RPD ≤ 50%
Equipment Blank	Measure of potential field contamination	1 per 20 samples per sampling methodology	Not applicable	Review decontamination procedures	< QL
Method Blank (MB)	Analyte-free matrix that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	No analytes detected > QL	Investigate source of contamination. Samples associated with the contaminated blank shall be re-prepared & reanalyzed or may be reported as qualified if sample concentrations are > 10x blank level	< QL

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Recovery limits 75% - 125%	Reanalyze and if acceptable, continue. If unacceptable, re-digest & reanalyze LCS with associated field samples. Analytes in the LCS that fail high and are non-detect in the samples can be reported.	Aqueous: Recovery within 75 - 125% Solid: Vendor certified / SRM limits
LCSD	Matrix-matched blank duplicate matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples (only required if no MSD or lab duplicate)	RPD ≤ 20%	Flag data	RPD ≤ 20%
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 set per prep batch of up to 20 samples (MSD not required if laboratory duplicate performed)	Recovery limits 71% - 125% (SOP limits); Aqueous: RPD ≤20% Solid RPD ≤ 35%	Flag data	Recovery within 75% - 125%; Aqueous: RPD ≤ 20% Solid RPD ≤ 35%
ISM Replicates A,B,C	Replicate ISM samples will be collected with a total of approximately 1 kilogram soil each replicate to assess overall precision and representativeness of ISM sample results.	3 replicates to be collected / analyzed in each DU	%RSD ≤ 35%	No lab corrective action unless reanalysis requested by project manager. Potential data usability issue.	%RSD ≤ 35%

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Matrix: All	**Analytical Group: SVOCs/PAHs**			**SOP: 727r14**	
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 field duplicate per 10 discrete samples	Not applicable	Review sampling procedures	RPD ≤ 30% for aqueous samples; RPD ≤ 50% for solid samples
Equipment Blank	Measure of potential field contamination	1 per 20 samples per sampling methodology	Not applicable	Review decontamination procedures	< QL
Method Blank (MB)	Analyte-free matrix that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	No analytes detected > QL or > 10% the amount measured in any sample (8270); No analytes detected > ½ QL or > 10% the amount measured in any sample (8270-SIM) (SOP limits)	Re-extract and/or reanalyze entire prep batch if samples report detects within 2 x of Method Blank level, if possible; otherwise, narrate issues	< QL
Internal Standard	Added to each sample extract prior to analysis and used for quantitation of targets and to ensure accuracy of analysis.	Per sample (including MS/MSD, LCS, and blanks)	Area 50-200% of CCV Area; retention time (RT) within ± 30 sec of CCV RT	If IS Area < 20% of CCV Area, re-extract sample, if possible; otherwise flag and narrate the issue	Area 50-200% of CCV Area; RT within ± 30 sec of CCV RT
LCS/LCSD	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Laboratory statistical limits	Re-extract and reanalyze entire prep batch if > 10% of compounds fail criteria. If not possible to re-extract, flag and narrate issue	Recovery within laboratory control limits

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 set per prep batch of up to 20 samples	Laboratory statistical limits	If LCS for prep batch and surrogates for un-spiked sample are acceptable, report the data flagging and narrating issues	Recovery within laboratory control limits RPD ≤ 20% for aqueous samples; RPD ≤ 30% for solid samples
Surrogates	Surrogates added to LCS samples and blanks are used to assess recovery in a matrix known to be free from interference. This information can be used to determine the magnitude of matrix interference effects on environmental sample results.	Per sample (including MS/MSD, LCS, and blanks)	Laboratory recovery limits	If ≥ 2 Base-Neutral or ≥ 2 Acid Surrogates fail criteria, reextract, if possible; otherwise, flag and narrate the issue	Recovery within laboratory control limits
ISM Replicates A,B,C	Replicate ISM samples will be collected with a total of approximately 1 kilogram soil each replicate to assess overall precision and representativeness of ISM sample results.	3 replicates to be collected / analyzed in each DU	%RSD ≤ 35%	No lab corrective action unless reanalysis requested by project manager. Potential data usability issue.	%RSD ≤ 35%
Matrix: All	**Analytical Group: VOCs**			**SOP: 767r13, 764r13**	

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 field duplicate per 10 discrete samples	Not applicable	Review sampling procedures	RPD ≤ 30% for aqueous samples RPD ≤ 50% for solid samples
Trip Blank	Analyte-free water or methanol (for solids) taken from the laboratory to the sampling site and returned to the laboratory unopened. Used to assess sample VOC contamination originating from the sample transport, shipping, or site	1 per cooler shipped with samples for VOC analysis	Not applicable	Review sampling/shipping/ handling procedures, flag data	< QL
Equipment Blank	Measure of potential field contamination	1 per 20 samples per sampling methodology	Not applicable	Review decontamination procedures	< QL
Method Blank (MB)	Analyte-free matrix that is carried through all preparation/extraction and analytical procedures.	1 per analytical batch of up to 20 samples	Result < QL or < 5% the amount measured in any sample or 10% the regulatory limit (SOP limits)	Reanalyze samples if samples have detections within 2 x Method Blank level, if possible. If not, flag and narrate issues	< QL
Internal standards	Added to each sample extract prior to analysis and used for quantitation of targets and to ensure accuracy of analysis.	Per sample (including MS/MSD, LCS, and blanks)	Area 50-200% of CCV Area RT within ± 30 sec of CCV RT	Inspect mass spectrometer and GC for malfunctions. Reanalyze samples while system was malfunctioning is mandatory.	Area 50-200% of CCV Area RT within ± 30 sec of CCV RT

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
LCS/LCSD	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per analytical batch of up to 20 samples	Laboratory statistical limits	Reanalyze entire Analytical Batch, if possible; otherwise, flag and narrate issue	Recovery within laboratory control limits
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 set per analytical batch of up to 20 samples	Laboratory statistical limits	If LCS/LCS duplicate for analytical batch and surrogates for un-spiked sample are acceptable, report the data flagging and narrating issues	Recovery within laboratory control limits RPD ≤ 20 % for aqueous samples RPD ≤ 30% for solid samples
Surrogates	Surrogates added to LCS samples and blanks are used to assess recovery in a matrix known to be free from interference. This information can be used to determine the magnitude of matrix interference effects on environmental sample results.	Per sample (including MS, LCS, and blanks)	Water and soil criteria dependent on in-house control limits.	Reanalyze sample, if possible, otherwise flag and narrate issues	Recovery within laboratory control limits
Matrix: All	**Analytical Group: TOC/DOC**			**SOP: 320r14, 321r17**	

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 per 10 samples	Not applicable	Review sampling procedures	RPD ≤ 30% for aqueous samples, RPD ≤ 50% for solid samples
Method Blank (MB)	Analyte-free matrix that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	< ½ QL (SOP limit)	All associated samples are reanalyzed unless sample concentrations are > 10x blank value	< QL
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures	1 per prep batch of up to 20 samples	Recovery limits: aqueous: 80-120%, soil/sediment: 75%-125% or vendor limits for SRM (SOP limits)	All associated samples are reprepared and reanalyzed	Recovery limits: aqueous 80-120%, soil/sediment: 75%-125% or vendor limits for SRM (SOP limits)
LCSD	Matrix-matched blank duplicate matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples (only required if no MSD or lab duplicate)	RPD ≤ 25% (SOP limit)	Flag data	RPD ≤ 25%
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 per prep batch of up to 20 samples (MSD not required if laboratory duplicate performed)	Recovery limits: aqueous: 75%-125% RPD ≤ 25%, soil/sediment: 75-125%, RPD ≤ 50% (SOP limits)	Flag data	Recovery limits: aqueous: 75%-125% RPD ≤ 25%, soil/sediment: 75-125%, RPD ≤ 50% (SOP limits)

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Matrix: All	**Analytical Group: Alkalinity**			**SOP: 394r010**	
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 per 10 samples	Not applicable	Review sampling procedures	RPD ≤ 30%
MB	Analyte-free water that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	< QL, evaluate at ½ QL (SOP limit)	1. Evaluate samples. Samples having a concentration of >10x blank value may be reported. Samples having a concentration <10x QL must be reanalyzed. 2. Evaluate the DI water. 3. Recalibrate and reanalyze all affected samples.	< QL
LCS/	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Recovery limits 85%-115% (SOP limits)	1. Check accuracy of titrant concentration. 2. Reanalyze.	Recovery limits 80%-120%
Laboratory Sample Duplicate	A sample that is split into subsamples at the lab. Each subsample is then analyzed and the results compared. Used to test the precision of the laboratory measurements.	1 per batch of 10 or fewer samples.	RPD < 25%	1. Ensure that sample is well mixed 2. Reanalyze.	RPD < 25% (use SOP limits)
Matrix: All	**Analytical Group: Sulfide**			**SOP: 368r14**	

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 per 10 samples	Not applicable	Review sampling procedures	RPD ≤ 30%
MB	Analyte-free water that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	< QL	Evaluate samples: concentration > 5x MB contamination or < QL are ok to report. Reanalyze all samples not fitting the criteria above.	< QL
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Recovery limits 75%-125%	All associated samples are reprepared and reanalyzed	Recovery within 75-125%
LCSD	Matrix-matched blank duplicate matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples (only required if no MSD or lab duplicate)	RPD ≤ 25% (SOP limits)	Flag data	RPD ≤ 25%

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
MS/MSD	MS is a field sample to which known concentrations of target analytes have been added. MSD is a second aliquot of the same sample prepared and analyzed along with the parent sample and MS. MSD prepared same as MS. The MS/MD or MS/MSD results are used to evaluate matrix effects on the precision and accuracy of results.	1 per prep batch of up to 20 samples (MSD not required if laboratory duplicate performed)	Recovery limits 75% 125% RPD ≤ 25%	Flag data	Recovery within 75-125% RPD ≤ 25%
Matrix: All	**Analytical Group: Sulfate & Chloride**			**SOP: 318r28**	
Field Duplicate	Used to assess improper homogenization of the samples in the field; reproducibility of sample preparation and analysis; and, heterogeneity of the matrix.	1 per 10 samples	Not applicable	Review sampling procedures	RPD ≤ 30%
MB	Analyte-free water that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	< QL	Evaluate samples before further analysis. If < QL – no reanalysis is required. If > 5x concentration in blank – no reanalysis is required. All other samples using the contaminated blank for QC will be reanalyzed.	< QL
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Recovery +/- 10% (SOP limits)	Rerun once, reanalyze samples	Aqueous – recovery within 80-120%

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
MS	MS is a field sample to which known concentrations of target analytes have been added. Results are used to evaluate matrix effects on the accuracy of results.	1 per prep batch of up to 10 samples	Recovery +/- 10%	Evaluate if LCS is in control, then flag as matrix interference	Recovery 75-125%
Matrix: All	**Analytical Group: IVBA**			**SOP: ENV-SOP-SHRT-0162v00**	
MB	Analyte-free reagent water that is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	< QL for lead	Re-analyze; make new extraction fluid and reanalyze effected samples	< QL for lead
LCS	Matrix-matched blank matrix spiked with a known quantity of the target analytes, which is carried through all preparation/extraction and analytical procedures.	1 per prep batch of up to 20 samples	Recovery limits 85%-115% (SOP limits)	Re-analyze or re-extract and reanalyze	Recovery limits 80%-120%
NIST SRM 2711a for lead	Standard Reference Material independently verified by NIST	1 per batch of 20 or fewer samples.	Recovery limits 75.2-96.2%	Re-analyze	Recovery limits 75.2-96.2%
MS	MS is a field sample to which known concentrations of target analytes have been added. Results are used to evaluate matrix effects on the accuracy of results.	1 per prep batch of up to 20 samples (MSD not required if laboratory duplicate performed)	Recovery limits 75% 125%	Re-analyze; make new extraction fluid and reanalyze effected samples	Recovery within 75-125%

QC CHECK	EXPLANATION	RUN FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	DATA VALIDATION CRITERIA[1]
Laboratory Sample Duplicate	A sample that is split into subsamples at the lab. Each subsample is then analyzed and the results compared. Used to test the precision of the laboratory measurements.	1 per batch of 20 or fewer samples.	RPD < 20%	Re-analyze; make new extraction fluid and reanalyze effected samples	RPD < 20%
Reagent Blank	Extraction fluid not run through the extraction procedure	1 per batch of samples	< QL for lead	Prepare new batch of extraction fluid and reprocess effected samples	< QL for lead

TABLE 8 LABORATORY DATA DELIVERABLES

RECORD	Metals	Hardness	Mercury	PAHs	SVOCs	VOCs	TOC/DOC	Alkalinity	Sulfate & Chloride	Sulfide	IVBA
Narrative	X	X	X	X	X	X	X	X	X	X	X
COC	X		X	X	X	X	X	X	X	X	X
Sample Receipt Report	X		X	X	X	X	X	X	X	X	X
Summary results	X	X	X	X	X	X	X	X	X	X	X
QC summary results	X		X	X	X	X	X	X	X	X	X
Chromatograms/Spectra				X	X	X					
Instrument raw data (all calibration reports, QC sample raw data, sample raw data for all analyses including dilutions and re-analyses, and preparation/extraction information)	X		X	X	X	X	X	X	X	X	X
Laboratory bench sheets/logs (all bench sheets must be included)	X		X	X	X	X	X	X	X	X	X
Electronic data deliverables	X	X	X	X	X	X	X	X	X	X	X

TABLE 9 DATA VERIFICATION PROCEDURES

ITEM/DESCRIPTION	VERIFICATION (COMPLETENESS)	VALIDATION (CONFORMANCE TO SPECIFICATIONS)
Planning Documents/Records		
1. Approved QAPP	X	
2. Contract	X	
3. Field SOPs	X	
4. Laboratory SOPs	X	
Field Records		
5. Field logbooks	X	X
6. Equipment calibration records	X	X
7. COC forms	X	X
8. Sampling diagrams/surveys	X	X
9. Sediment description logs	X	X
10. Relevant correspondence	X	X
11. Deviations	X	X
12. Field corrective action reports	X	X
Analytical Data Package		
13. Cover sheet (laboratory identifying information)	X	X
14. Case narrative	X	X
15. Inter-laboratory COC documents	X	X
16. Sample receipt records	X	X
17. Sample chronology (i.e., dates and times of receipt, prep, analysis)	X	X
18. Laboratory bench sheets and instrument logs	X	X
19. Limit of Detection/QL establishment and verification	X	X
20. Standards traceability	X	
21. Instrument calibration records	X	X
22. Definition of laboratory qualifiers	X	X
23. Results reporting forms	X	X

TABLE 10 DATA VALIDATION PROCEDURES

Data Validator: Environmental Data Quality, Inc.

Summary: All received data packages will be verified externally. Third-party validation will be performed by Environmental Data Quality, Inc. according to the data validation procedures specified in the USEPA *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*; USEPA's *National Functional Guidelines for Inorganic Superfund Methods Data Review*, Office of Superfund Remediation and Technology Innovation (OSRTI), USEPA, OLEM 8240.1-66, EPA-542-R-20-006, November 2020 (NFG); the analytical methods; and the DQOs defined in this SAP/QAPP. Professional judgment will be applied as necessary and appropriate. Stage 3 validation will be conducted for the first SDG for each medium for metals, SVOCs, PAHs, VOCs, and IVBA. For the remaining SDGs, a modified Stage 2B validation for metals, SVOCs, PAHs, VOCs, and IVBA will be conducted. Data for TOC, hardness, alkalinity, sulfate, chloride, sulfide, and DOC will undergo a Stage 1 validation.

Analytical group/method:	Metals, SVOCs, PAHs, VOCs, IVBA, TOC, hardness, alkalinity, sulfate & chloride, sulfide, DOC
Data deliverable requirements:	Table 8 (full data report as bookmarked pdf file and electronic data deliverable, EDD)
Analytical specifications:	Equipment calibration included in lab SOPs, see Table 4; Lab QA/QC & Measurement Performance criteria in Table 7; QLs in Tables 6A & 6B
Percent of data packages to be validated:	100%
Measurement performance criteria (MPC):	Lab SOPs and Tables 6A, 6B, & 7
Percent of raw data reviewed:	One sample result per parameter per matrix for first data package
Percent of results to be recalculated:	One sample result per parameter per matrix for first data package
Validation procedure:	MPC in Table 7, method requirements, Lab SOPs, & National Functional Guidelines
Validation code*:	S3VM for organics (SVOCs, PAHs, VOCs) S3VM for inorganics (metals & IVBA) S2bVM for the aqueous EBs and trip blanks for SVOCs, PAHs, VOCs, metals, and IVBA S1VM for TOC, hardness, alkalinity, sulfate, chloride, sulfide, and DOC.
Electronic validation program/version:	EQuIS compliant EDD

Validation Code*	Validation Label	Description/Reference
S1VE	Stage 1 Validation Electronic	EPA 2009 Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use
S1VM	Stage 1 Validation Manual	
S1VEM	Stage 1 Validation Electronic and Manual	
S2aVE	Stage 2A Validation Electronic	
S2aVM	Stage 2A Validation Manual	
S2aVEM	Stage 2A Validation Electronic and Manual	
S2bVE	Stage 2B Validation Electronic	
S2bVM	Stage 2B Validation Manual	
S2bVEM	Stage 2B Validation Electronic and Manual	
S3VE	Stage 3 Validation Electronic	
S3VM	Stage 3 Validation Manual	
S3VEM	Stage 3 Validation Electronic and Manual	
S4VE	Stage 4 Validation Electronic	
S4VM	Stage 4 Validation Manual	
S4VEM	Stage 4 Validation Electronic and Manual	
NV	Not Validated	

The following data qualifiers will be applied during data validation. Potential impacts on project-specific DQOs will be discussed in the data usability report.

Qualifier	Definition
U	The analyte was analyzed for but was not detected above the level of the reported sample-specific QL.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been “tentatively identified” or is “presumptively” present and the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was not detected above the reported concentration. The reported QL is approximate and may be inaccurate or imprecise.
R	The sample result is rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

APPENDIX A: STANDARD OPERATING PROCEDURES

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Multi-Incremental Soil Processing for Semi-volatile & Metal Analyses	**Revision Date:** September 2021
Method No: ITRC February 2012	**SOP No:** 149r08
Page: 1 of 8	**Supersedes:** 149r07

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/7/21	Tamara Rentz	9/7/21
Stephen C. Ede	11/14/22	Tamara Rentz	11/14/22
Stephen C. Ede	11/6/23	Tamara Rentz	11/6/23

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This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Tamara Rentz 2022.11.16 07:37:15 -09'00'

Tamara Rentz 2021.09.08 11:48:01 -08'00'

Summary of Changes from Previous Revision:

- Section 11.1.2. addendum incorporated and altered for clarification.
- Section 11.1.1. updated for clarification.
- Section 11.3.2 updated for clarification.
- Grammar corrected throughout.

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1.0. OBJECTIVE:

Multi Incremental (MI) soil processing, for semi-volatile and metal analyses, as part of remediation of a contaminated site is designed to provide an accurate average concentration representative of soil contained within a defined area.

2.0. SCOPE AND APPLICATION:

Samples collected in the field with MI protocol may be sent to the lab as a bulk sample from a "decision unit." This SOP details how to process that soil into discrete MI analytical increments for non-volatile analyses.

This SOP covers laboratory procedures for MI processing of soils for semi-volatile and metals methods, not field handling. The client is responsible for collecting 30-50 increments of 30-60 g each (i.e., >900 g) from the field.

Note: This procedure is not appropriate for volatile analyses. Refer to 2012 ITRC Incremental Sampling Methodology guidance.

3.0. DEVIATIONS FROM REFERENCE METHOD:

N/A

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP, and submit needed SOP revisions to the QA Office.

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4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

N/A

6.0. SAMPLE HANDLING:

6.1 Sample Matrix – Soil

6.2 Sample Collection – Samples are typically collected and relinquished in a bucket or plastic bags.

6.3 Sample Size – >900 grams

6.4 Sample Preservation – Note: Samples requiring refrigeration are maintained at 0-6°C.

6.5 Holding Times – Analysis should be performed at the same time as weigh out for other analytical procedures; preferably upon receipt and generally within 14 days of sample collection.

6.6 Criteria for Acceptance/Rejection of Samples – Alert Project Manager, QA, or Production Manager if there is not enough sample available for weighing out of all analyses; and/or if large pebbles/rocks comprise most of the sample volume.

7.0. APPARATUS:

7.1. #10 (2mm) sieve, Teflon or brass.

7.2. Large plastic tray

7.3. Large disposable aluminum pans

7.4. Micro Spatulas

7.5. Wooden tongue depressors

7.6. Large plastic or wooden spatulas

7.7. Weigh boats

7.8. Sample jars: 4-8oz amber, 16 oz. extraction jars, and Metals digestion vials

7.9. Analytical Balance (at least 3 decimal places)

8.0. REAGENTS:

N/A

9.0. EXTRACTION:

N/A

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

11.1. Sample Drying:

11.1.1. If the sample intended for metals and/or PCB analyses is too wet to sieve, it can be air dried at ambient temperature.

11.1.2. Soil for any analyses other than metals and/or PCB (PAH, 8270, Pesticide and/or DRO) should **not** be dried.

11.1.2.1. If the sample is too wet to divide into 30 sections or there is standing water, contact QA and the PM for instructions.

11.1.3. Dry soil only until the sample can be sieved, not to exceed 3 days.

11.1.3.1. Turning the soil daily is allowed.

11.2. Sieve the **entire** dry sample using a 2mm sieve. The sieved sample should equal approximately 500 to 1000 grams. Spread the sieved sample evenly, approximately1/2 inch in depth on the bottom of the tray.

11.2.1. Any material left on the sieve is returned to the original container for storage.

11.3. Divide the tray into 30 similar area sections.

11.3.1. **For Semi-Volatile Methods:** Use a micro spatula to collect approximately 1 gram from each section. Because fines tend to settle, scrape the spatula along the bottom of the tray to make sure that every particle size is equally represented in the sample. Place all scoops into a single extraction jar for each analysis (**minimum 30 grams**, but no more than 50 grams, regardless of method) and the entire jar must be extracted.

11.3.1.1. Repeat this process to collect a second sample into a separate container as backup (in case re-analysis is required).

11.3.2. **For Metals Analysis:** Use a micro spatula to collect approximately 0.33 grams from each section (total mass 10-11 grams), making sure to sample all the way to the bottom of the tray. Place all scoops into a weigh boat and homogenize. Record the total weight of the sample onto the spreadsheet. Split this sample into 10 digestion vials, each vial not to exceed 1.1gr. Print a label containing the total sample weight for all 10 digestion vials and label each vial.

11.3.2.1. **For BMS/BMSD/DUP Analysis:** Collect a total mass of 13-14gr. Follow the above procedure separating into 10 vials. Label each **"PS".** Label 3 additional vials: BMS, BMSD, & DUP. Place 1.0 – 1.1gr into each of these 3 vials & attach appropriate labels. Weigh an additional 13-14-gram sample and store in one vial. This can be separated for use if re-analysis is required.

11.3.3. **% solids**: Weigh an additional 30 gram sample for % solids analysis following SOP 143.

11.3.4. **For Reference Lab Analysis:** Follow the instructions in 11.3.1. for a sample and duplicate sample. Place samples into a 4 oz. amber jar. A 3rd jar is sent for % solids analysis.

11.3.4.1. **For Herbicides:** 2 jars, 30 – 50 grams.
11.3.4.2. **For EPH:** 2 jars, 50 – 60 grams.

11.3.4.3. **For Hexachrome:** 2 jars, 50 – 60 grams.
11.3.4.4. **For Percent solids:** 1 jar, 30 – 50 grams, for each analysis.
NOTE: *It is not necessary to record the weights for Ref. Lab samples.*

11.3.5. Save any leftover sieved sample in a separate container.

11.4. To clean the sieve, remove debris stuck in the mesh, rinse thoroughly with water and air or pat dry.

12.0. QUALITY CONTROL:

N/A

13.0. CALCULATIONS, REVIEW AND REPORTING:

N/A

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative, a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

N/A

20.0. REFERENCES:

The Interstate Technology & Regulatory Council Incremental Sampling Methodology, February 2012
Todd Blessing (ADEC), April 2015

21.0. ATTACHMENTS:

N/A

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Determination of Inorganic Anions by Ion Chromatography	**Revision Date**: September 2023
Method No: EPA 300.0; SW 9056A; SW 5050	**SOP No**: 318r28
Page: 1 of 27	**Supersedes:** 318r27

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/6/23	Jamaica Rentty	9/6/23

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Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
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Signature: _____ Printed Name: _____ Date: _____

Addendum #1 added 10/06/2023 TPR

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Determination of Inorganic Anions by Ion Chromatography	**Revision Date**: October 2023
Method No: EPA 300.0; SW 9056A; SW 5050	**SOP No**: 318r28 Addendum #1
Page: 1 of 1	**Supersedes**: 318r27

Signatures below reflect approval for the following changes to the current SOP. These changes will be incorporated into the SOP during the next review. This addendum will be incorporated into the electronic SOP (i.e., PDF file).

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Edl	10/6/23	Samara Beatty	10/6/23

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

The above referenced SOP should be modified as follows:

Insert after section 7.1.8:

7.1.9 For instrument maintenance and troubleshooting, refer to the instrument manual. For computer hardware and/or software, refer to IT. All routine and non-routine maintenance must be noted into the IC maintenance logbook.

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Summary of Changes from Previous Revision:

- Addendums 1 and 2 incorporated
- Section 7.6 – Vial caps are not used.
- Section 8.4 – Updated to current practices.
- Section 10.2.1 – Auto dilution functions are not employed for calibrations, 15 mg/L calibration point added.
- Figure 1 was updated
- Section 10.2.3 – included QC standard preparation reference.
- Section 11.13 – CCV volume updated
- Section 11.15.1 – Example updated to current practices.
- 11.15.8 – Auto-vial information added.
- 13.6.2 – Additional information added from the standard method.

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1.0. OBJECTIVE:

This document outlines a procedure for analyzing samples for anion concentration by ion chromatography.

2.0. SCOPE AND APPLICATION:

2.1. The matrices applicable to this method/SOP are drinking water, surface water, mixed domestic and industrial wastewaters, ground water, reagent waters, solids (after water extraction), and leachates (e.g., landfill). Note: TCLP leachates cannot be analyzed by this technique.

2.2. Total halogens as chloride can be analyzed by following SW846 Method 5050, and this SOP after the samples are combusted in a Parr bomb calorimeter. (See ASTM D 808-95 for combustion protocols.) Refer to SGS SOP352 for calorimeter procedure.

2.3. Total sulfur can be analyzed by following SW 846 Method 5050 and this SOP after the samples are combusted in a Parr bomb calorimeter. (See ASTM D 808-95 for combustion protocols.) Refer to SGS SOP352 for calorimeter procedure.

2.4. A small volume of sample, 20 µL, is introduced into an ion chromatography column. The anions of interest are separated and measured, using a system comprised of a guard disk, analytical column, suppressor device, and conductivity detector.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. Standard viability as referenced in Method 300.1 has been extended to this method/SOP; whereas 300.0 and 9056 indicate 1 month for standards prepared from neat stock. SGS uses multi-anion standard solutions of 1000 mg/L F, Cl, Br, NO3-N, PO4-P and SO4, and allows for a 6 month stock standard viability for them. Working solutions are prepared daily by addition of NO_2-N. The nitrite standards are replaced monthly.

3.2. Method 300.0 requires that duplicate LCS samples be run quarterly. Our normal sample load and batch size meet criteria. If circumstances develop where that is not true, duplicate LCS analysis will be documented for the three month period.

3.3. Method 300.0 requires that all samples analyzed for total nitrate/nitrite with concentration greater than 0.5 mg/L be re-sampled and reanalyzed for Nitrate and Nitrite separately. *Deviation: The laboratory does not require that samples be reanalyzed if the concentration for total nitrate/nitrite is greater than 0.5 mg/L.*

3.4. Method SW9056A states that an ICV/CCV solution should be made from a second source external standard. Deviation: The ICV/CCV solutions are made from the primary source standard. The QCS is made from a second source and is analyzed on a daily basis.

3.5. This method deviates from SW 5050 section 8.4. MS and LCS recoveries are ±30%.

3.6. This SOP deviates from method 300.0 defining residual error as "read back" (section 10). EPA Method Update Rule 2012 understands this method will not have a linear calibration. We adopt the practice from Standard Methods (22nd edition) of checking each calibration point and apply ±50% criteria for the low point and ±10% for all other calibration points.

4.0. RESPONSIBILITIES:

4.1. The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion(s) of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

5.2. The water dip or negative peak that elutes near the fluoride peak can usually be eliminated by the addition of the equivalent of 1mL of concentrated eluent (see section 8.2) to 100 mL of each standard and sample. If the water dip is interfering, replacement of the analytical column may be necessary.

5.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

5.4. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems. Currently, every injection into the system is filtered through a 0.2 µm membrane filter by the 858 Autosampler.

5.5. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Carbonate and other small organic anions cause known coelution. At concentrations of fluoride above l.5 mg/L, this interference may not be significant. It is the responsibility of the analyst to accurately generate and interpret information in each sample matrix.

5.6. The quantitation of unretained peaks should be avoided. Low molecular weight organic acids (formate, acetate, propionate, etc.) are conductive, coelute with or near fluoride, and will bias the fluoride result in some drinking and most waste waters. In addition, this method is not recommended for leachates of solid samples (i.e. TCLP extracts) where acetic acid has been used for pH adjustment.

5.7. In the case of an interrupted injection, analytes that have not yet eluted may interfere with an immediately following injection.

6.0. SAMPLE HANDLING:

6.1. Sample Matrix – The matrices applicable to this method/SOP are drinking water, surface water, mixed domestic and industrial wastewaters, ground water, reagent waters, oils, solids (after water extraction), and leachates.

6.2. Sample Collection and Size – Water samples are typically taken and stored in either 60-250 mL HDPE bottles. Documentation of the date and time of collection is important for those analytes that have short hold times.

6.3. Sample Preservation: Samples requiring analysis for Nitrite-N, Nitrate-N, o-Phosphate, and Sulfate need to be cooled to 0-6°C. Those to be analyzed for Bromide, Chloride, and Fluoride do not require refrigeration.

6.4. Holding Times:

6.4.1. For aqueous samples: -Bromide, Chloride, Fluoride, Sulfate 28 days
-Nitrite-N, Nitrate-N, o-Phosphate 48 hours

6.4.2. For soil samples: Soil samples should have the extraction and analysis completed within the 28 day hold time, starting from collection date and time. Following the extraction process described in Section 9, the extract must be analyzed with a 48 hour window for nitrate, nitrite, and o-phosphate; all other analytes are to be analyzed with the 28 day hold time.

6.5. Criterion for Acceptance/Rejection of Samples: No sample will be run on the IC that contains a preservative (e.g. H_2SO_4) or one that may severely damage the instrument and/or column. Samples preserved for Total Nitrate+Nitrite should be scheduled for analysis by Flow Injection.

7.0. APPARATUS:

7.1. Ion Chromatograph: Metrohm Modular Ion Chromatography system (or equivalent) comprised of the following:

7.1.1. IC Detector.

7.1.2. MSM 3 channel suppressor unit, supplied with regenerate by a 2mL Dosino unit and clean water post analysis from the detector

7.1.3. Sample processor capable of intelligent dilution with a 2mL sample Dosino and 10mL dilution Dosino. DI water is drawn from a water vessel.

7.1.4. IC Pump. This pump is able to deliver 0.7 mL of eluent per minute at a pressure between 5.0 and 15.0 MPa. The minimum pressure is set at 1.0 MPa and the maximum pressure is set at 12.0 MPa.

7.1.5. IC Separation Center. This includes an injection valve, a 20 µL sample loop, guard column, separator column (Metrosep A Supp 5), suppressor, temperature-controlled (or temperature compensated) small-volume conductivity cell.

7.1.6. Computer

7.1.7. Column: Metrosep A Supp 5 - 100 mm x 4.0 mm, or 150 mm x 4.0 mm, or equivalent

7.1.8. Metrohm MagIC Net Chromatography Software

7.2. Volumetric flasks of various volumes

7.3. Autopipettors and appropriate tips

7.4. Disposable 10 mL syringes

7.5. 0.45 µm syringe filters

7.6. Sample vials

7.7. Analytical Balance with ±0.1 mg sensitivity to weigh salts for stock standards.

7.8. Analytical Balance with ±10 mg sensitivity to weigh reagents to prepare eluent.

7.9. Conductivity meter.

7.10. Graduated Environmental Express Digestion Vessels with a certified tolerance of ± 0.2 mL.

8.0. REAGENTS:

8.1. Reagent water: Deionized (DI) water directly off the filter line, free of the anions of interest. The DI water should contain particles no larger than 0.20 microns.

8.2. Working Eluent: Using "snips" of concentrated eluent purchased from vendor, add 1 snip per 1L of eluent to be made to volumetric flask and dilute to volume with degassed DI water. Pour eluent slowly from volumetric flask into instrument eluent container to avoid dissolving gasses as this can cause instrumentation issues. Eluent should be made daily.

8.3. If no eluent snips are available, eluent may be prepared using the following steps:

8.3.1. Eluent Concentrate: 3.2mM sodium carbonate and 1.0mM sodium bicarbonate. Let DI water sit in DI container at least overnight to degas before making eluent. Dissolve 33.9163 g sodium carbonate and 8.4022 g sodium bicarbonate in water and bring to a volume of 1 L. Each new lot of concentrate should be checked with a CCV before using for analysis. Eluent concentrate expires 1 year from date made or at the expiration date of either stock standard, whichever is sooner.

8.3.2. Working eluent from concentrate: Add 10 mL of concentrate to 1 L of degassed DI water.

8.4. Regeneration Solution (suppressor): 0.2N sulfuric acid is purchased from vendor. If unavailable, dilute 5.56 mL of concentrated sulfuric acid to 1 liter with DI water.

8.5. Primary source standard: multi-anion solution of 1000 mg/L F, Cl, Br, NO_3-N, PO_4-P and SO_4 and solution of 1000 mg/L NO_2-N. Purchased commercially prepared from AccuStandard or equivalent.

8.6. Secondary source standard: multi-anion solution of 1000 mg/L F, Cl, Br, NO_3-N, PO_4-P and SO_4 and solution of 1000 mg/L NO_2-N. Purchased commercially prepared from SPEXCertiprep or equivalent.

8.7. Stability of standards: Multi-anion stock standards are stable for at least six months when stored at 0-6°C. The primary and secondary multi-anion stock standard will be replaced six months after open date or manufacturer's expiration date. As NO_2-N is less stable and will oxidize to NO_3 over time, the primary and secondary Nitrite-N stock source standards should be ordered separately and replaced on a monthly basis. The primary stock standards are evaluated daily against the second source to verify continued validity up to the manufacturer's expiration date. Working standards containing nitrite are prepared daily.

8.8. If analyte degradation or contamination is noticed in any standard prior to its assigned date of expiration, it must be replaced immediately.

**NOTE: Pay special attention, when ordering calibration and QC standards, to the units used by the manufacturer. Some standard suppliers express the concentration of nitrate as nitrate-nitrogen and others as simply nitrate. The same is true for nitrite/nitrite-nitrogen. See sections 8.5 and 8.6 for appropriate concentrations.*

8.9. Quality Control Samples: See section 13 for acceptance criteria.

8.9.1. Primary working standard 100mg/L: Dilute 1.0mL of each of the primary stock standards (8.5.) to 10.0 mL with DI water.

8.9.2. Initial and Continuing Calibration Verification (CCV): Dilute 2.0mL of each of the primary stock standards (8.5.) to 200.0mL with DI water in a volumetric flask. This will give a final concentration of 10 mg/L. Place the solution in the bottle in position 150 on the sampler rack.

8.9.3. 2nd source working standard 150mg/L: Dilute 1.5mL of each of the 2nd source stock standards (8.6.) to 10.0 mL with DI water.

8.9.4. The QCS is prepared by diluting 1.0mL of the working secondary source standard (8.9.3) to 10mL with DI water. This will give a final concentration of 15mg/L.

8.9.5. Method Blank (MB) Aqueous: This is an extraction blank that follows all of the steps used in the extraction batch preparation process including filtration through a 0.45 µm filter *if the samples require filtration.*

8.9.6. Laboratory Control Sample 5.0mg/L (LCS) Aqueous: Add 0.5 mL of primary working standards (8.9.1.) to 9.5 mL of DI Water.

8.9.7. Matrix Spike & Matrix Spike Duplicate (MS/MSD) Aqueous: This is prepared by diluting 0.5mL of primary working standard (8.9.1) into 9.5mL of sample.

8.9.8. Method Blank (MB) Solid: This is an extraction blank that follows all of the steps used in the extraction batch preparation process including filtration through a 0.45 µm filter. This is prepared by adding 4.0-4.2 g of pre-rinsed Teflon chips to a 50 mL digestion vessel (7.10.) and following the extraction procedure in section 9.4.

8.9.9. Laboratory Control Sample (LCS) Solid: 50mg/Kg: This is prepared by spiking 4.0–4.2 g of pre-rinsed Teflon chips for soils with 2.0mL of primary working standard (8.9.1) and following the extraction procedure in section 9.4.

8.9.10. Matrix Spike & Matrix Spike Duplicate (MS/MSD) Soil: This is prepared by spiking 4.0–4.2 g of sample with 2.0mL of primary working standard (8.9.1) and following the extraction procedure in section 9.4

8.9.11. MB, LCS, MS/MSD Oil – Total Halogen as Chloride: See SOP#352 for extraction procedure. All bomb batch QC and samples are run on the IC at a 2X dilution.

8.9.12. Calibration Blank (CB): DI water placed in the bottle in position 149 on sampler rack.

8.10. Working Standards and Reagents are made on a daily basis and do not need a logbook entry.

9.0. EXTRACTION:

Total Halogens

9.1. Extraction should be used for solid materials and for oil where the level of analyte resulting from leaching is desired. The extraction of solid waste, oil, and fuel for analysis of Total Halogens as chloride will be performed on the Parr Bomb Calorimeter, via methods D808/SW5050, and SW846/5050 (See ASTM D 808-95 for combustion protocols) per SGS SOP#352. Extracted samples and QC are analyzed at a 2X dilution.

Soil Extractions

9.2. Label plastic digestion vessels (section 7.10) with work order and sample number.

9.3. Weigh out 4.0-4.2 g of a representative portion of the homogenized sample directly into a tared digestion vessel. Record the actual weight in the extraction logbook. Refer to SOP#143 for correct weighing procedure.

9.4. Add 40 mL of DI water to each weighed sample and mix for ten minutes. Filter the resulting slurry with a 0.45 µm membrane filter before loading onto the autosampler. The slurry may be centrifuged at 2200 rpm for 12 minutes before filtration to assist in filtration. If different weights are used, maintain a water to sample ratio of 1 to 10 for solid materials.

9.5. Batch QC for the solid batch (MB, LCS, MS, MSD) must be included with each extraction batch. See section 8.9 for preparation instructions and section 13 for acceptance criteria.

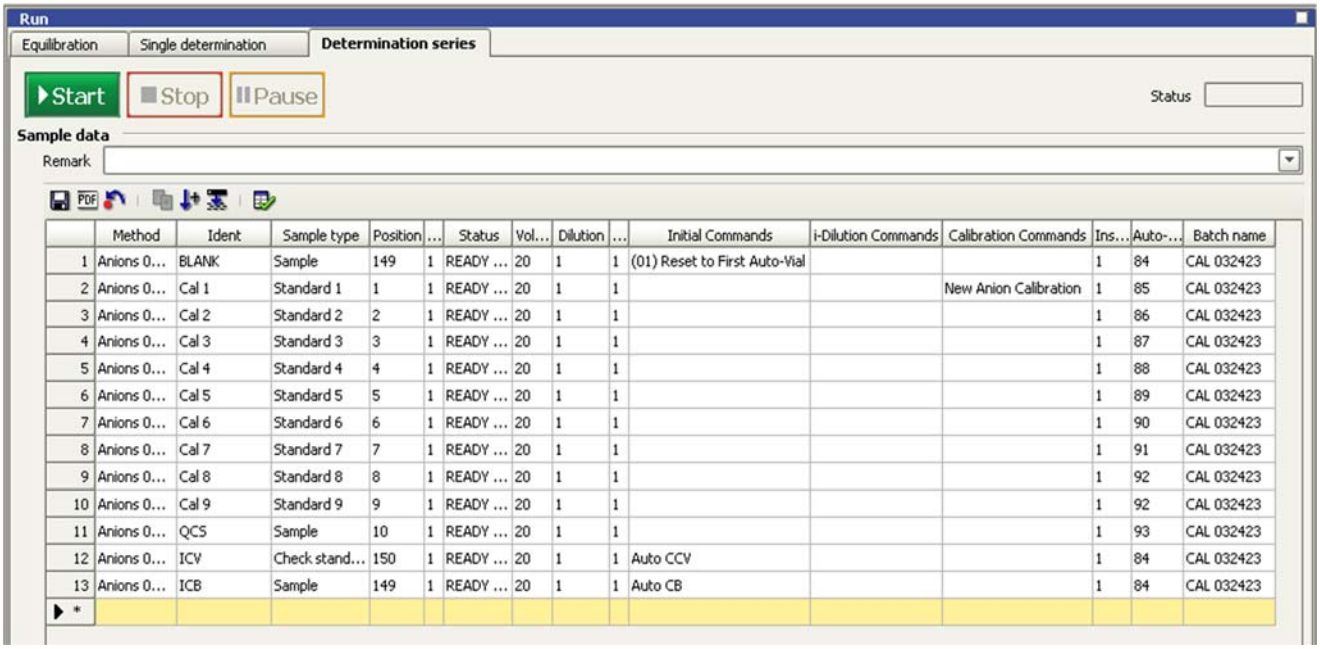
10.0. CALIBRATION:

10.1. Recalibration for the instrument does not need to be done daily. It should be done when the QC fails more than once for instrument related reasons. It should also be done following any major maintenance on the instrument, such as changing the column. **Full instructions can be found in the MagIC Net User Guide, which can be found on the IC computer or in the Instrument Manuals folder on the network: \\USFS700\ANK_Groupdata\Public\DOCUMENT\Instrument_Manuals\IC Metrohm**

10.2. To recalibrate the instrument, the following steps should be followed:

10.2.1. For each analyte of interest, prepare intermediate calibration standards of two vials of 10.0 mg/L (0.1 mL of primary standards (8.5) diluted to 10.0 mL) and one vial of 100.0 mg/L (1.0 mL of primary standards (8.5) diluted to 10.0 mL). Prepare the calibration standards at the concentrations of 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, 10, 15 and 20 mg/L. Set up determination series as shown below and place calibration standards into positions as shown in Figure 1. Be sure to select "New Anion Calibration" in the Calibration Commands field for the first sample.

10.2.2. At some specific instances (e.g. Linear Calibration Range failure), additional concentration between 0.1 to 20 mg/L can be included in the calibration.



	Method	Ident	Sample type	Position	...	Status	Vol...	Dilution	...	Initial Commands	i-Dilution Commands	Calibration Commands	Ins...	Auto-...	Batch name
1	Anions 0...	BLANK	Sample	149	1	READY ...	20	1	1	(01) Reset to First Auto-Vial			1	84	CAL 032423
2	Anions 0...	Cal 1	Standard 1	1	1	READY ...	20	1	1			New Anion Calibration	1	85	CAL 032423
3	Anions 0...	Cal 2	Standard 2	2	1	READY ...	20	1	1				1	86	CAL 032423
4	Anions 0...	Cal 3	Standard 3	3	1	READY ...	20	1	1				1	87	CAL 032423
5	Anions 0...	Cal 4	Standard 4	4	1	READY ...	20	1	1				1	88	CAL 032423
6	Anions 0...	Cal 5	Standard 5	5	1	READY ...	20	1	1				1	89	CAL 032423
7	Anions 0...	Cal 6	Standard 6	6	1	READY ...	20	1	1				1	90	CAL 032423
8	Anions 0...	Cal 7	Standard 7	7	1	READY ...	20	1	1				1	91	CAL 032423
9	Anions 0...	Cal 8	Standard 8	8	1	READY ...	20	1	1				1	92	CAL 032423
10	Anions 0...	Cal 9	Standard 9	9	1	READY ...	20	1	1				1	92	CAL 032423
11	Anions 0...	QCS	Sample	10	1	READY ...	20	1	1				1	93	CAL 032423
12	Anions 0...	ICV	Check stand...	150	1	READY ...	20	1	1	Auto CCV			1	84	CAL 032423
13	Anions 0...	ICB	Sample	149	1	READY ...	20	1	1	Auto CB			1	84	CAL 032423
*															

Figure 1 Standard calibration table

10.2.3. Prepare QCS and ICV/CCV samples as outlined in section 8.9.4 and 8.9.2.

10.2.4. Start up the instrument as outlined in section 11. After 30 minute equilibration period has passed, begin determination series.

10.2.5. After analysis has completed, highlight all of the standards and select reprocess.

10.2.6. In the reprocess window, select the standard with the lowest concentration. Zoom in on the calibration to verify the integration parameters, listed in the integration tab, are selecting the peaks appropriately. The basic settings are generally effective.

10.2.7. Select the components tab and update the component retention times to match peaks as needed. Press the "Update Retention Times" button, then the "Update" button to apply any changes and verify correct peak labeling.

10.2.8. Once all changes are made and applied to the lowest point, press the "Reprocessing" button and check the "From standards of reprocessing" option. This will build the curve point by point. For instance, the lowest point will have a calibration only containing itself, while point two will have itself and point one, etc.

10.2.9. Select the top point (the highest concentration). The calibration on this standard will contain all points. Press the "Reprocessing" button again, this time choosing the "From selected determination" option to apply the complete curve to all points. Once the system has completed this, select the "Method" button and choose "Save As" to save the method.

10.2.10. At this point, the calibration is complete. Exit the reprocessing window, then select the blank, QCS, CCV, and CB as well as any calibration point and press the reprocessing button again. With the calibration point selected, press "Reprocessing" and choose the "From selected determination" option to apply the completed curve to the Blank, QCS, CCV, and CB.

10.3. Calibration Verification:

10.3.1. Each calibration point must be evaluated with read back. Validation for points ≤ LOQ is ± 50% of the true value. The criterion for all other points is ± 10% of the true value.

10.3.2. The calibration curve will be verified by the analysis of QCS (second source). QCS data will be submitted (true value of 15mg/L ±10%) with the calibration packet for peer review.

10.3.3. See section 8.9.4 for preparation of the QCS (true value of 15mg/L ±10%).

10.4. Assemble the calibration packet with the following components: 1) IC Calibration Cover Sheet, 2) IC Calibration and Standards Spreadsheet, FW-0102, 3) Calibration Graphs, 4) Electronic Run Log, 5) Chromatograms (raw data), 6) QCS chromatograms for calibration verification. The calibration packet is to be submitted for review through the peer review process.

10.5. Linear Calibration Range (LCR): If a linear fit is chosen for the curve fit. Linearity is verified initially and verified every 6 months by the analyst. *Note: as per Method Update Rule 2012: "Linear calibration models do not adequately fit calibration data with one or two inflection points."*

10.5.1. Calibrate the instrument as described in Sections 10. Verify the linearity of the curve by filling out the Linear Regression Spreadsheet (CW-0013_300.0_9056_Full_LR_Template.xlsx). Input the Area and the expected concentration for each analyte. Once all needed points are entered, enter the slope and intercept value into the corresponding cells. Each calculated value should be within ± 10% of the true value and the regression line should have an $R^2 > 0.995$.

10.5.2. If the calibration fails for linearity using all points, linearity will need to be established for the lower and higher ranges of the calibration. Perform 10.6.1. using the blank and the lowest five calibration points initially and adjust the total number of points, by removing the highest points in the range, until the lower range passes for linearity. Repeat 10.6.1. using the remaining calibration points to determine linearity for the upper range. If the upper range fails for linearity or has fewer than three calibration points, recalibrate with an additional point.

11.0. ANALYSIS:

11.1. Prepare fresh eluent and ensure that the regenerant and water bottles are full. See section 8 for instructions on how to prepare these reagents.

11.2. Check the filter on the Sample Processor and replace if necessary.

11.3. Launch the MagIC Net software.

11.4. The instrument will go through self-checks. This is complete when the "Status" fields (in the Configuration tab) for both the instrument and sample processor say "ok". **Do not attempt to enter manual mode or start the hardware until this completes.**

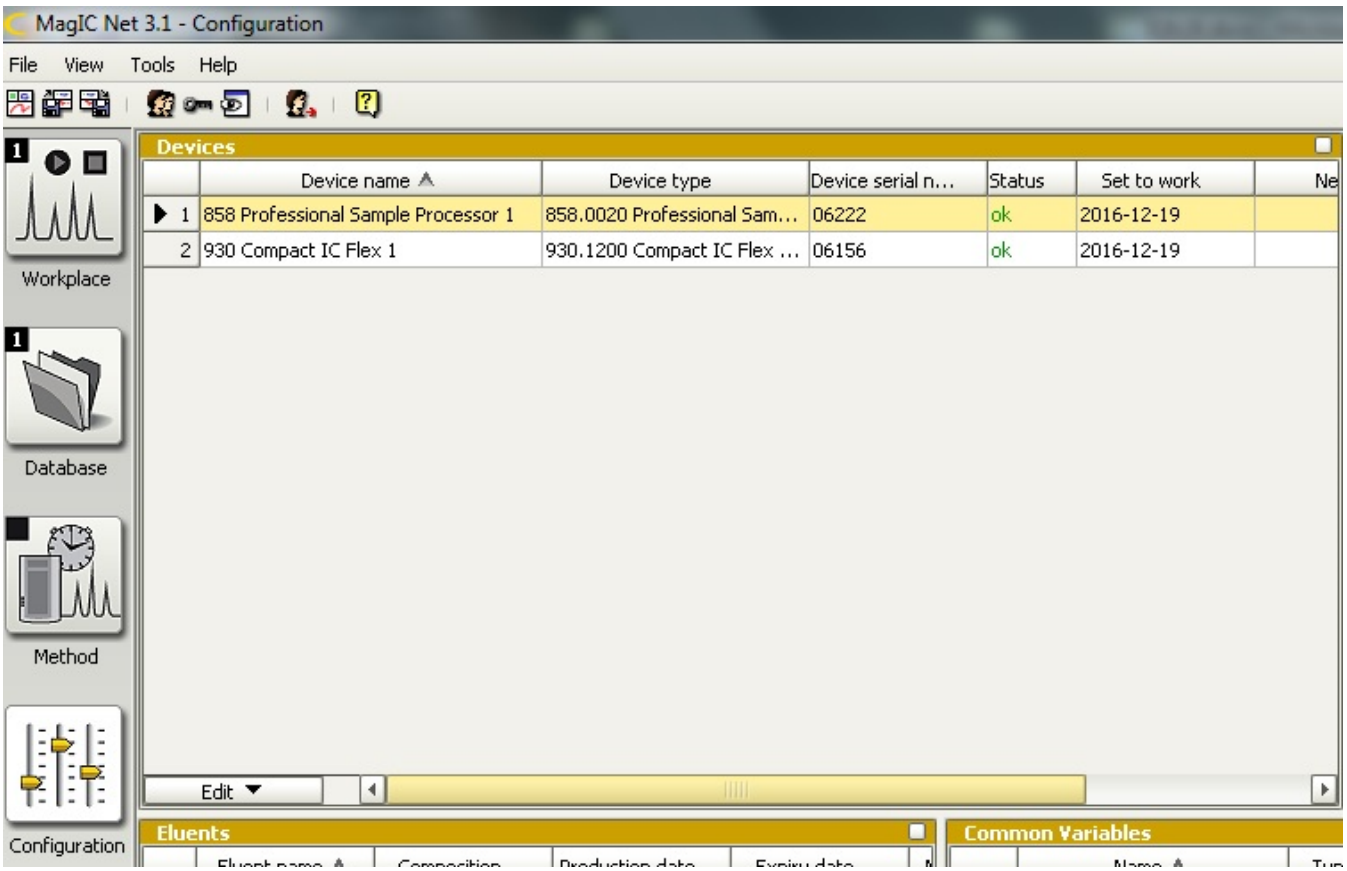


Figure 2 Configuration tab

11.5. Remove all old vials and replace all used vials in the Auto-Vial positions with new vials if needed. The current Auto-Vial is shown in the configuration tab; all used Auto-Vials are from that position back to position 84.

11.6. Once the self-checks are complete, purge the eluent line and pump of bubbles by going to the Manual tab on the bottom left of the screen, selecting "All devices" from the drop down menu, the clicking on the 930 Compact IC Flex. Attach a syringe to the outlet tube on the IC pump purge valve and twist the valve counter clockwise to open. Type "2.0" in the flow Input field and hit Start. This will pump eluent at a rate of 2.0 mL/min into the syringe. Continue until about 6 mL has been pumped through, then hit Stop and close the valve.

***NOTE:* DO NOT RUN THE PUMP AT THIS FLOW RATE WITHOUT ENSURING THE VALVE IS OPEN AS THIS CAN DAMAGE THE COLUMN.**

11.7. Next, go to the Workplace tab, then select the Equilibration tab and press "Start HW" to begin equilibration.

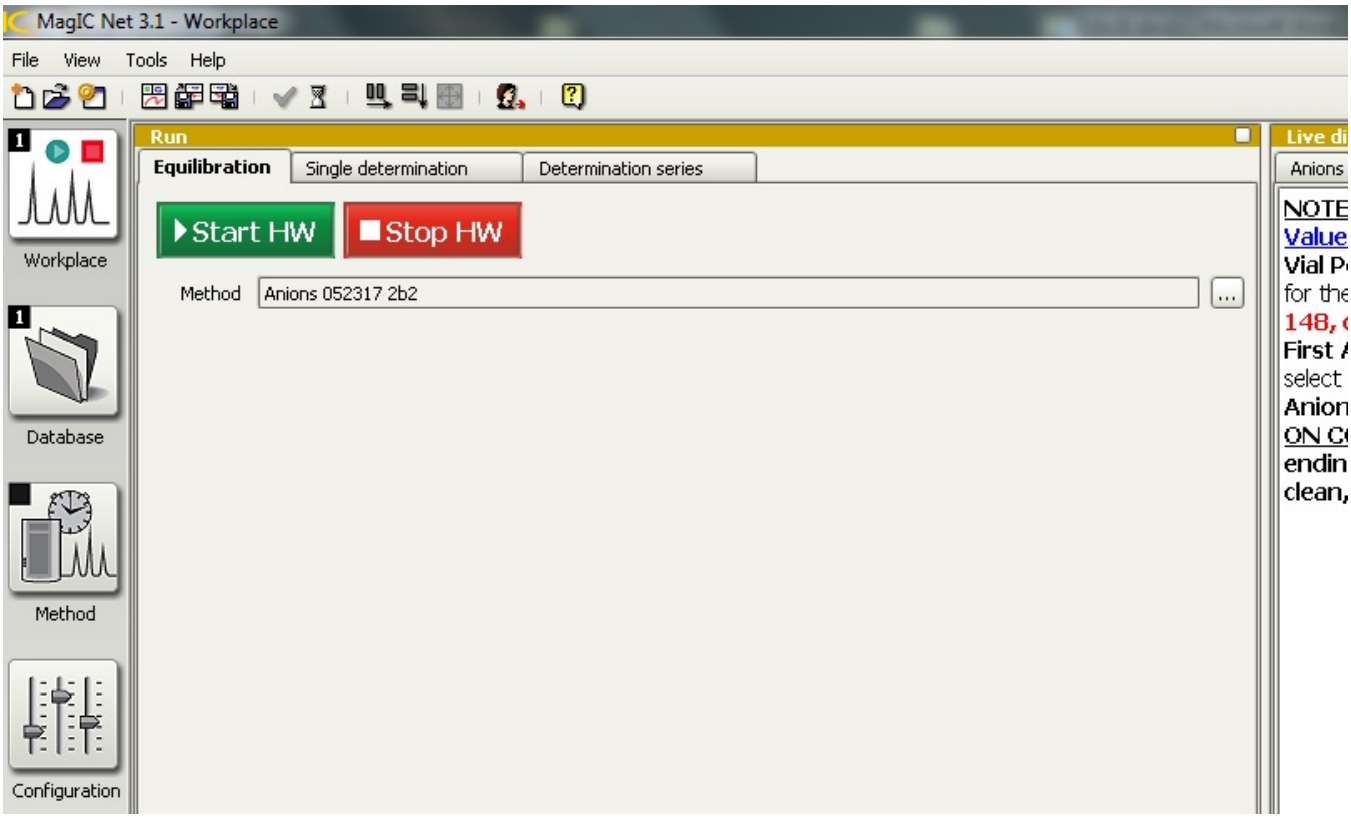


Figure 3 Equilibration tab

11.8. The instrument will begin to equilibrate and will show a graph of the conductivity in the Live display window. It is set up to automatically "step" the suppressor every 10 minutes when not analyzing a sample, therefore the instrument should be allowed to equilibrate for 30 minutes to allow each path to be flushed with regenerant solution and water.

11.9. To ensure the longevity of the instrument, a screening technique has been developed in order to avoid potential harm to the instrument. The conductivity can be taken of each sample, using a portable conductivity meter, prior to analysis in order to determine the most appropriate dilution factor for a sample. The following table can be used to determine the dilution from the conductivity:

Conductivity Reading, µmhos	**Recommended Dilution**
0 – 300	1X
300 – 800	1X (F, Br, NO_2, NO_3) 5X (Cl, S)
800 – 1000	1X (F, Br, NO_2, NO_3) 10X (Cl, S)
1000 – 1700	2X (F, Br, NO_2, NO_3) 20X (Cl, S)
1700-2000	2X (F, Br, NO_2, NO_3) 25X (Cl, S)

2000-3000	5X (F, Br, NO_2, NO_3) 25X (Cl, S)
3000-4000	10X (F, Br, NO_2, NO_3) 50X (Cl, S)
4000-5000	10X (F, Br, NO_2, NO_3) 100X (Cl, S)
Above 5000	500X to screen

Table 1. Conductivity Screening

**Note – Salinity samples should always be tested by the conductivity screening technique. High salinity samples analyzed at wrong dilution can jeopardize the instrument.*

11.10. Set up the samples to be run. The following standards are required:

11.10.1. A QCS will be run at the beginning of each analytical run preceding the analysis of an ICV and ICB. See section 8 for preparation instructions and section 13 for acceptance criteria.

11.10.2. An ICV and ICB will be run at the beginning of every analytical run. A CCV and CB will automatically be run at a frequency of once every ten injections within the batch. Instrument QC does not count as one of the ten samples in-between CCV/CB. All prep QC, paying samples, MS/MSD, and rinses count towards the number of injections run in-between CCV/CB. See section 8 for preparation instructions and section 13 for acceptance criteria.

11.10.3. A MB will be run at the beginning of each extraction batch at a frequency of one per twenty field samples and one per matrix (water, soil, oil and SW5050). See section 8 for preparation instructions and section 13 for acceptance criteria.

11.10.4. An LCS will be run at the beginning of each extraction batch at a frequency of one per twenty field samples and one per matrix (water, soil, oil and SW5050). See section 8 for preparation instructions and section 13 for acceptance criteria.

11.10.5. **For SW 9056A:** A MS/MSD must be run for 5% of field samples for each matrix (1 set in each batch of 20 samples of less). See section 8 for preparation instructions. See section 13 acceptance criteria. **For EPA 300.0:** A MS must be run for 10% of field samples (1 MS for every 10 samples or less).

11.10.6. For drinking water samples, a low level quantitation (LLQC) needs to be evaluated each analysis day. The calibration point at the LOQ will be used as the LLQC.

11.11. In the Database tab, select Determinations from the top of the screen, hover over Batch, then select New Batch and type in WIC YYYMMDD.

11.12. Go back to the Workplace tab and select Determination series. Load the Anions Auto Dilution Template and verify that the method listed in the QCS line is the most current. Double click on the QCS line to open it and select the batch for the new run from the drop down menu.

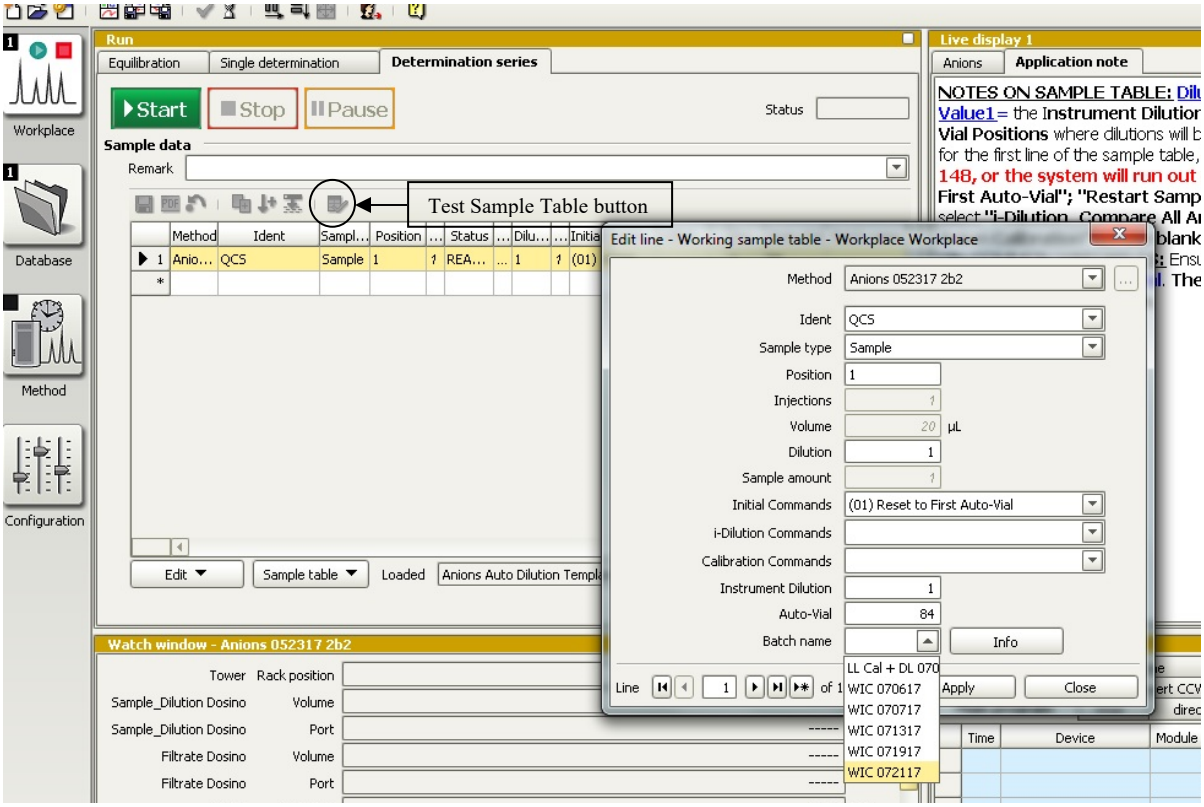


Figure 4 Sample scheduling window

11.13. After the 30 minutes equilibration period has elapsed and the baseline is stable, load the QCS into position 1, fill the bottle in position 149 with DI water, and place 200-250 mL of CCV solution in the bottle in position 150.

11.14. Press Start to begin the analysis. A CCV/CB pair will automatically be scheduled and correctly begin the counter after the CB so that a CCV/CB pair will be scheduled every 10 samples thereafter.

11.15. Double click on the line beneath the CB to schedule the MB, LCS, and all samples.

11.15.1. Fill out the Ident field with the sample ID (with container ID), ex. 1234-1A.

11.15.2. The Sample type field is Sample for all schedules except for CCV.

11.15.3. The Position field automatically moves to the next position when the next button is pressed, but care should always be taken to ensure the position matches the position on the rack that the sample is in.

11.15.4. While scheduling samples, to indicate that a manual dilution was done on the sample before placing in the rack, type X(dilution factor) after the sample name (see Table 1 for dilution guidelines). For example, if a ten-fold dilution was done on a sample, type “X10” after the sample name. If no dilution was done, type only the sample name. The dilution field should stay 1 for all samples.

11.15.5. The Initial Commands field should be left blank for all schedules except for the QCS, which should have "Reset to First Auto-Vial" selected.

11.15.6. The Calibration Commands field should be left blank unless a new calibration is being performed.

11.15.7. The Instrument Dilution field should be 1 unless the desired dilution is known. **If this is not 1, causing the instrument to perform an auto dilution for that schedule, and the sample is over range for any analyte, the instrument will not perform further dilutions.** As such, it is better to prepare any necessary dilutions as suggested by Table 1 by hand so the instrument can catch any unknown over range analytes.

11.15.8. The Auto-Vial field should always be 1 higher than the previous schedule. **Do not exceed auto-vial 148** as this is the last auto-vial. Scheduling beyond this number causes a system error and the instrument will not continue the run sequence.

11.15.9. The batch name should be the batch for that day.

11.16. The "Test Sample Table" button (see Figure 4) should be pressed after all entries are complete to ensure all entries are complete and the program will run through the table without any errors.

11.17. The instrument will run through the sample table, adding CCV/CB pairs every 10 samples and scheduling any auto dilutions at the end of the run, until it hits the end, where it will run a closing CCV/CB pair then shut off as long as the "Stop hardware when sample table is finished" box is checked.

11.18. To retrieve and post a batch, go to the Database tab and select the batch from the drop-down menu. This will allow the user to go through the chromatographs and see the concentrations.

**A note on the results window: the "Concentration_On-Column" field represents the actual result measured by the instrument and should be within the calibrated range or have triggered an intelligent dilution. The "Concentration" field is the Concentration_On-Column field multiplied by the manual dilution, while the "Final Concentration" field is the Concentration_On-Column field multiplied by both the manual and intelligent dilution factors*.

11.19. Reports for any samples requiring manual integration should be printed prior to reprocessing.

11.20. The batch is exported to a folder for posting via LimsBridge. The chromatographs are printed along with a Determination overview (run log).

12.0. LOW LEVEL ANALYSIS

12.1. Some clients may request a very low DL for specific analytes. These requests are handled in a case-by-case manner and are worked closely with the Technical Director and Project Manager. A working Low Level method is described below.

12.2. Working method for low level Nitrate and o-Phosphate

12.2.1. Calibration was built as described in section 10, replacing calibration standards with 10 point calibration: 0.005 mg/L, 0.010 mg/L, 0.020 mg/L 0.050 mg/L, 0.100 mg/L, 0.200 mg/L, 0.500 mg/L, 1.000 gm/L, 2.000 mg/L, 5.000 mg/L.

12.2.2. QC Requirements:

12.2.2.1. QCS is spiked at 4.0 mg/L

12.2.2.2. CCV is spiked at 2.5 mg/L **and is placed in the bottle in position 150.**

12.2.2.3. LCS is spiked at 1.0 mg/L

12.2.2.4. MS/MSD are spiked at 1.0 mg/L

12.2.3. Analysis is performed as described in section 11. It is good practice to replace the filtration membrane on the sample processor and ensure that all other filters are replaced within manufacturer recommended timelines to be sure there is no contamination before analyzing low level samples.

13.0. QUALITY CONTROL:

13.1. Accuracy and Precision Measurements – Aqueous Samples EPA 300:

13.1.1. Laboratory Control Sample (LCS): This must be run at the beginning of every batch of twenty or fewer field samples. The acceptance criteria are ±10% of the true value. Recoveries outside this range will require the identification and repair of the problem and the successful analysis of the LCS before analysis of samples can begin.

13.1.2. Sample Matrix Spike (MS): A MS is required every 10 samples or less. The acceptance criteria are ±10% for the spike recovery. If spike fails to meet control limits and the LCS is within control limits, sample will be flagged for suspected matrix interference.

13.2. Accuracy and Precision Measurements – 9056 water and soil samples

13.2.1. Laboratory Control Sample (LCS): This must be run with every extraction batch. Acceptance criteria is from laboratory based control limits that are generated for each analyte as described in SOP 145.

13.2.2. Sample Matrix Spike/Duplicate (MS/MSD): A MS/MSD is required for every batch of 20 samples or less. The acceptance criteria is from laboratory based control limits that are generated for each analyte as described in SOP 145.

13.2.3. The acceptable Relative Percent Difference (RPD) between the MS/MSD is ≤ ±20. If spike fails to meet control limits and the LCS is within control limits, sample will be flagged for suspected matrix interference.

13.3. Accuracy and Precision Measurements – Total Halogens, SW5050

13.3.1. Laboratory Control Sample (LCS): This must be run with every extraction batch. The acceptance criteria for the LCS is ± 30% of the true value.

13.3.2. Sample matrix spike/duplicate (MS/MSD): A MS/MSD is required every batch of 20 samples or less. The recovery criteria is ±30%. If spike fails to meet control limits and the LCS is within control limits, sample will be flagged for suspected matrix interference. The Relative Percent

Difference acceptance criteria is ≤ 15. If RPD fails to meet criteria, re-extract samples. If RPD still fails to meet criteria, flag samples as suspected matrix interference

13.3.3. The project manager **must** be notified when any total halogen sample extracted and analyzed by the 9056/5050 method reads between 1,000 and 4,000 ppm. This will allow project manager to contact the client and determine if the sample needs to be confirmed by gas chromatography methods.

13.4. Calibration Verification Criteria-

13.4.1. Quality Control Sample (QCS): The QCS is a second source standard run once at the beginning of each run. The acceptance criteria are ±10% of the true value. Recoveries outside this range will require the identification and repair of the problem and the successful analysis of the QCS sample before analysis of samples can begin. This standard must be from a source independent of the calibration standards.

13.4.2. Calibration Verification (ICV/CCV): The acceptance criteria is ±10% of the true value. Recovery outside this range will require the identification and repair of the problem and the successful analysis of a calibration verification sample before the analysis of paying samples can proceed.

13.4.2.1. **For non-DOD samples**: Samples analyzed since the last acceptable calibration verification within the same analysis sequence will need to be reanalyzed once the analysis of another CCV is within acceptable limits. The initial calibration verification (ICV) sample will be analyzed at the beginning of the run; the continuing calibration verification (CCV) after every ten samples and at the end of the run. Additionally, a CCV must be analyzed after the preparation of additional eluent solution. The target analytes' retention times should be within the established retention time windows; however, analyst experience and expertise are vital for proper analyte identification.

13.4.2.2. **For DOD samples:** The analyst may immediately (within 1 hr of the failing CCV and before any other samples have acquired) rerun 2 successive CCVs. If both meet QC criteria for all analytes of interest, then the samples already analyzed may be reported and the run sequence may be continued. Samples following the two passing CCVs are also valid. See SOP 500.

13.4.3. Calibration Blank (ICB/CB): The ICB is analyzed before any samples can be run and must read < LOQ. Thereafter, the calibration blank (CB) is analyzed after every ten samples and at the end of each run. The acceptance criteria for the calibration blank are <LOQ (see section 19 for LOQs). A measured value outside allowable limits may require the reanalysis of some samples. ICB values exceeding the LOQ may be an indication of laboratory reagent contamination. Samples that have an apparent concentration <LOQ will not require reanalysis. Samples having a concentration >10X the ICB contamination will not require reanalysis. All other samples analyzed since the last acceptable blank require reanalysis after appropriate corrective action is performed.

13.5. Blank Criteria-

13.5.1. **Aqueous Samples**: Method Blank (MB): This must be run at the beginning of every batch of twenty or fewer field samples. The MB is analyzed before any samples can be run and must read < LOQ. MB values exceeding the LOQ may be an indication of laboratory reagent contamination. Samples associated with a MB that exceeds this limit must be carefully evaluated; i.e., if the sample is < LOQ or is > 5X the level of contamination exhibited by the MB, it may be reported and flagged. All other samples must be re-extracted and reanalyzed.

13.5.1.1. For DOD clients only, MB recoveries ≥ ½ LOQ require the identification and repair of the problem and the successful analysis of the MB before analysis of samples can begin. Any samples with detectable results (unless they are >5x MB) associated with a MB that exceeds this limit must be reanalyzed.

13.5.1.2. The MB will be evaluated at the DL for drinking water samples.

13.5.2. **Soil Samples (9056):** A method blank (MB) must be prepared with every extraction and analyzed as part of the batch. For soil and oil, the allowable limit is < LOQ. Samples associated with a MB that exceeds this limit must be carefully evaluated; i.e., if the sample is < LOQ or is > 5X the level of contamination exhibited by the MB, it may be reported and flagged. All other samples must be re-extracted and reanalyzed.

**Note:* *For DOD clients only, MB recoveries ≥ ½ LOQ require the identification and repair of the problem and the successful analysis of the MB before analysis of samples can begin. Any samples with detectable results (unless they are >5x MB) associated with a MB that exceeds this limit must be re-extracted and reanalyzed.*

13.5.3. **Total Halogens, SW9056:** A method blank (MB) must be prepared with every extraction and analyzed as part of the batch. For total halogens, the allowable limit is < LOQ. Samples associated with a MB that exceeds limits must be carefully evaluated; i.e., if the sample is < LOQ or is > 5X the level of contamination exhibited by the MB, it may be reported and flagged. All other samples must be re-extracted and reanalyzed.

Note: DOD criteria are* ***not* *applicable total halogens as chloride due to oil matrix.*

13.6. Demonstration of Instrument Operating Specifications:

13.6.1. On a periodic basis it must be shown that the instrument is operating within specifications. The parameters and verification intervals are outlined below.

13.6.1.1. Initial Demonstration of Performance (IDC): Conducted before each new operator can independently operate the instrument, and then annually for each analyst thereafter. This includes the preparation and analysis of four successful laboratory control samples and successful analysis of a blind performance evaluation sample.

13.6.1.2. Detection Limits (DL): Performed annually or when major maintenance occurs (e.g. column change) to determine the acceptance window for identifying the analytes. The DL should also be re-established if there has been a significant change in instrument response.

13.6.1.3. Retention Time Width Study: Performed annually or when major maintenance occurs (e.g. column change) to determine the acceptance window for identifying the analytes. The window used for daily measurements will be three times the standard deviation of the retention times established using three standards measured on analyses performed over a 24 hour period. The minimum setting is 5%.

13.6.2. Analyte Retention Time: Observed times vary with conditions. If the retention time for any analyte in the daily initial CCV varies from the last set value for the initial CCV by more than +/- 5% then the RT will be reset using the RT from the current initial CCV. See Method 300.0, section 11.4 for retention time criteria.

**Note – The retention time window within MagIC Net will be set to 5% of the current retention times. When a peak is not named in the opening QC, this is an indication that the retention time window has shifted and appropriate action should be taken.*

13.6.3. If the response for any analyte in the CCV varies from the calibrated values by more than 10% the test must be repeated using a fresh calibration standard. If the results are still more than ±10%, a new calibration curve must be prepared.

13.6.4. When warranted, manual integration can be done on individual chromatograms. For guidance regarding manual integration, refer to SOP 144.

13.6.5. For all samples or QC requiring any changes to integration, the analyst must provide chromatograms of both before and after the manual integration and initial and date each with a brief comment justifying the integration changes. Each before and after must be included in the Peer Review Report for review.

13.7. Low Level Quantitation (LLQC) will be analyzed at the LOQ for drinking water samples. The LLQC requirement is daily; however, for practical purposes, include the LLQC in every batch.

13.8. Any corrective action needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

14.0. CALCULATIONS, REVIEW AND REPORTING:

14.1. Equations:

14.1.1. Sample concentration determination: prepare separate calibration curves for each anion of interest by plotting peak size in area of standards against concentration values. Sample concentration is computed by comparing sample peak response with the standard curve.

14.1.2. Calibration Graphs: Calculate the following parameters: slope (s), intercept (I), and correlation coefficient (r). The slope and intercept define a relationship between the concentration and instrument response:

$$Y = K_2 X_i^2 + K_1 X_i \text{ EQUATION 1}$$

Where: Y = predicted instrument response
K_2, K_1 = coefficients
X_i = concentration of standard i

MagIC Net software does these calculations automatically when a new calibration is run.

14.1.3. Relative Percent Difference:

Where: A = Observed sample concentration
B = Observed sample duplicate concentration

$$RPD = \frac{|A - B|}{\left(\frac{A + B}{2}\right)}$$

14.1.4. Spike Recovery:

$$\%\,\mathrm{Recovery} = \frac{(V_0 - S_0)}{V_E} \times 100$$

Where V_0 = observed value of the spike
S_0 = observed value of the sample
V_E = expected value of the spike

14.1.5. Total Halogens are Chloride:

$$H_T = Cl + (0.44369 * Br) + (1.8661 * F)$$

Where: H_T = total halogens as chloride
Cl = observed chloride concentration
Br = observed bromide concentration
F = observed fluoride concentration

14.1.6. Salinity:

$$S = [(Cl*1.65)/(1000)]$$

Where: S = Salinity
Cl = observed chloride concentration

14.2. Review/Peer Review – A peer reviewer must date and initial all chromatograms that have been manually integrated.

14.3. For any drinking water sample that report **Nitrate-N over 10 mg/L, or Nitrite-N over 1.0mg/L.**

14.3.1. **Notify the client immediately** (within 24 hours) of unsatisfactory results by emailing a completed *ALERT: Nitrate-N over 10 mg/L or Nitrite-N over 1.0mg/L report.* See attachment C.

14.3.1.1. Record the date, time, and name of the person contacted in the appropriate area of the form

14.3.2. If **a PWSID number is on the Chain of Custody, notify ADEC.**

14.3.2.1. Email the form, attachment C, to the *correct* ADEC office**. Refer to SOP 109** for details regarding the appropriate ADEC office for the PWSID number. A document of ADEC offices can be found at: \\USFS700\ANK_Groupdata\Public\DOCUMENT\FORMS\Approved\FW\FW-0098_Currentcontacts_ADEC_Divisions.docx

14.3.2.2. If the Public Water System box is checked, or if the client requests the report be emailed to ADEC, but does not supply a PWSID number, contact the Project Manager so they can request clarification.

14.3.3. **Notify the Project Manager** immediately so the results may be submitted to ADEC via CMDP.

15.0. HEALTH AND SAFETY:

15.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

15.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

15.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

15.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

15.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

16.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

17.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

18.0. DETECTION LIMIT (DL) STUDY:

Detection Limit (DL) studies are verified annually, and performed when a new operator is trained for drinking water analyses, when a significant change in instrument response is observed, or when a new instrument is purchased for analysis. The DL study is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document.

The DLs are subject to update for the factors given earlier. The current maximum (MAX) DLs are given below.

	Water	**Soil**	**SW5050**
Anion	**Max DL (mg/L)**	**Max DL (mg/Kg)**	**Max DL (mg/Kg)**
Fluoride	0.05	0.62	1.24
Chloride	0.05	0.62	1.24
Nitrite-N	0.05	0.62	
Bromide	0.05	0.62	1.24
Nitrate-N	0.05	0.62	
Sulfate	0.05	0.62	

19.0. LIMIT OF DETECTION (LOD):

The LOD is defined per SOP 116. LOD verification shall be performed quarterly according to the schedule set by the QA Office.

20.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP 116. LOQ verification shall be performed quarterly according to the schedule set by the QA Office. DLs/LOQs may change at the lab's discretion and need not require an update to this document. Current lab LOQs are listed below.

	Water	**Soil**	**Oil**
Anion	**LOQ (mg/L)**	**LOQ (mg/Kg)**	**LOQ (mg/Kg)**
Fluoride	0.20	2.0	4.0
Chloride	0.20	2.0	4.0
Nitrite-N	0.20	2.0	
Bromide	0.20	2.0	4.0
Nitrate-N	0.20	2.0	
Sulfate	0.20	2.0	

The LOQ for Total Halogens as Chloride is 400 mg/Kg.

21.0. REFERENCES:

21.1. EPA 300.0/4-79-020, Revision 2.1, August 1993
21.2. SW 846 Method SW9056A, Revision 1, February 2007
21.3. DOD QSM (most recent version)
21.4. EPA Method Update Rule 2012
21.5. Standard Methods 22nd Edition
21.6. MagIC Net User Guide v3.2, Metrohm USA Inc., 2014

22.0. ATTACHMENTS:

Attachment A: Quick Reference Guide
Attachment B: Corrective Action Table
Attachment C: Alert Form

Attachment A: QUICK REFERENCE GUIDE

1. Replace eluent, 1 snip per liter
2. Fill water tank and regenerant if needed
3. Remove old vials and replace used auto-vials
4. Purge eluent line and pump for 3 minutes
5. Start hardware and allow to equilibrate for 30 minutes
6. Place fresh DI in bottle in position 149
7. Prepare standards:
   a. Make working 1° solution: 1mL of 1° standards diluted to 10mL
   b. Make working 2° solution: 1.5mL of 2° standards diluted to 10mL
   c. Prepare CCV: 2mL of 1° standards diluted to 200mL, pour into bottle in position 150
   d. Prepare LCS: 0.5mL of working 1° standards diluted to 10mL, mix and pour into vial in position 3
   e. Prepare QCS: 1mL of working 2° solution diluted to 10mL and placed in position 1
8. Low Level standards:
   a. Make LL working 1° solution: 0.1mL of 1° standards diluted to 10mL
   b. Make LL working 2° solution: 0.1mL of 2° standards diluted to 10mL
   c. Prepare CCV: 0.5mL of 1° standards diluted to 200mL, pour into bottle in position 151
   d. Prepare LCS: 1.0mL of LL working 1° solution diluted to 10mL and placed in position 3
   e. Prepare QCS: 4.0mL of LL working 2° solution diluted to 10mL and placed in position 1
9. Filter 10mL of DI and place in position 2
10. Load Anions Auto Dilution template, change batch ID, start sample table
11. Manually measure conductivity of samples to be analyzed and write on lids along with dilution according to table 1
12. Select sample to be MS/MSD
    a. Spike 0.5mL of working 1° solution into 9.5mL of filtered sample for each
    b. Low Level: spike 1.0mL of LL working 1° solution into 9.0mL of sample for each
13. Filter all samples, diluting as needed, place in rack, and fill out sample table
14. Check “Stop hardware when sample table is finished” box
15. Export files to folder, print chromatographs, print Determination overview

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Attachment B: CORRECTIVE ACTION TABLE

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA300.0 And SW9056A	Initial Calibration Verification (ICV)	After calibration & at beginning of each analytical batch.	Recovery ±10%	1. Repeat analysis once. 2. Recalibrate
	Calibration Verification Read Back	With each calibration	Low point ± 50% All other calibration points ± 10%	Correct the problem Recalibrate
	Initial Calibration Blank (ICB) Calibration Blank (CB)	Immediately after ICV After each 10 analysis and at the end of the run	Concentration < LOQ	1. Repeat analysis once. 2. Evaluate samples before further analysis. If < LOQ – no reanalysis is required. If >5x concentration in blank – no reanalysis is required. All other samples using the contaminated blank for QC will be reanalyzed.
	QCS (second source check)	After calibration & 1 per analytical batch or daily.	Recovery ±10%	1. Repeat analysis once. 2. Recalibrate
	Continuing Calibration Verification (CCV)	1 per 10 samples and at end of the run.	Recovery ±10%	1. Repeat once and repeat analysis of all associated samples. Recalibrate if second analysis of CCV remains outside criteria. 2. DOD requires 2 successful CCV runs
	Method Blank (MB)	1 per batch of ≤ 20 samples	< LOQ Evaluate at DL for DW ***DOD clients < ½ LOQ (soils and waters)**	1. Evaluate samples before further analysis. If < LOQ – no reanalysis is required. If > 5x concentration in blank – no reanalysis is required. All other samples using the contaminated blank for QC will be reanalyzed. 2. DOD clients – reanalyze MB if > ½ LOQ, and any associated samples.
EPA 300	Laboratory Control Sample (LCS)	1 for each batch of ≤ 20 samples	Aqueous – Recovery ±10%	1. Rerun once. 2. Reanalyze samples.
	Matrix Spike (MS)	1 set per batch of 10 or less	Recovery **±10%**	1. Evaluate LCS if in control, then flag as matrix interference.

Attachment B: CORRECTIVE ACTION TABLE (continued)

SW9056A	Laboratory Control Sample (LCS)	1 for each batch of ≤ 20 samples	Acceptance criteria is from laboratory based control limits that are generated for each analyte as described in SOP 145.	1. Rerun once. 2. Reanalyze samples.
	Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 set per batch of 20 or less	Recovery Acceptance criteria is from laboratory based control limits that are generated for each analyte as described in SOP 145. Duplicate RPD ≤ 15	1. Evaluate LCS if in control, then flag as matrix interference. 1. If RPD is out of control, reanalyze parent & MS/MSD. 2. If RPD is still out of control, flag the parent sample & comment matrix interference.

SW5050/ 9056A (oils for Total Halogens)	Laboratory Control Sample (LCS)	1 for each batch of ≤ 20 samples	Recovery ± 30%	1. Rerun once. 2. Reanalyze samples.
	Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 set per batch of 20 or less	Recovery ± 30% Duplicate RPD ≤ 15	1. Evaluate LCS if in control, then flag as matrix interference. 1. If RPD is out of control, reanalyze parent & MS/MSD. 2. If RPD is still out of control, flag the parent sample & comment matrix interference

Attachment C: ALERT FORM

Use form below: usfs700\ANK-Groupdata\Public\Alert-Nitrate over 10mg/Lor Nitrite-N over 1.0 mg/L

ALERT: Nitrate-N over 10mg/L or Nitrite-N over 1.0mg/L

Sample # (s)	
PWSID (Yes or No)	
Client/Project	
NOTES	

If a PWSID is associated, the analyst must post the data and e-mail ADEC, and the data must be submitted via CMDP by Data Services

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Total Organic Carbon in Soil	**Revision Date**: December 2021
Method No: SW 9060A	**SOP No**: 320r14
Page: 1 of 12	**Supersedes**: 320r13

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephan C. Ede	12/20/21	Jamaica Rentty	12/20/21

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Summary of Changes from Previous Revision:

- Revised section 8.2
- Edited section 10.3
- Edited section 10.6
- Edited section 11.2.3.6
- Minor spelling and grammar changes were made throughout
- Section 10.7 was updated to current practices.

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1.0. OBJECTIVE:

This standard operating procedure (SOP) outlines a method for determining the total organic carbon (TOC) content in solid samples.

2.0. SCOPE AND APPLICATION:

2.1. This document applies to determining TOC concentrations in soil and sludge samples. Before analysis, the samples are acidified and warmed to remove the inorganic carbon (IC). This is not a validated method for IC removal in soils, but has been modified and tested according to *Application Note TOC-011*, Rosemount Analytical Inc.

2.2. Analysis is performed by high temperature catalytic combustion using a non-dispersive infrared CO_2 detector. The acidified sample is loaded on a ceramic receiving boat and pushed into the combustion chamber. The O_2 carrier gas reacts with carbon to form carbon monoxide and CO2 which then flows through cobalt oxide to complete oxidation to CO_2. The CO_2 is then directed to the infrared detector measured in mg/L carbon by the computer.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 Shimadzu SCA-130-513 and SSI-AP-TOC-104 state samples are acidified using phosphoric acid. Nitric acid is used instead.

3.2 Shimadzu SSI-AP-TOC-104 states samples are dried prior to TOC analysis. Wet weight is used for analysis, and results are adjusted using percent solids.

3.3 SW 9060A states that the method is applicable for ground and surface waters, and domestic and industrial wastes.

3.4 SW9060A states that samples are run in quadruplicate and the average is reported. SGS analyzes samples only once. For State of Alaska clients, samples are analyzed in triplicate and for DOD clients, samples are analyzed in quadruplicate. This is determined at the login process by scheduling multiple analyses which are reported separately.

3.5 SW9060A states that samples are homogenized using a blender. Samples are homogenized by mixing well in a sample weigh boat.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. When the samples are warmed to 105˚C, some volatile organics may be lost.

5.2. Samples need to be kept at 0-6°C to minimize the bacterial decomposition and volatilization of organic components.

5.3. Baseline fluctuation may cause the analysis to end early, resulting in a low bias. If this occurs the sample must be re-weighed and re-analyzed.

6.0. SAMPLE HANDLING:

A 28-day holding time is applied for soil and sludge samples being analyzed by this method. (The holding time may be extended by freezing. Contact the QA Manager and Project Manager for approval on a project-by-project basis.) The samples are collected in glass containers with Teflon-lined or aluminum foil lids. Samples are stored at 0-6°C and are disposed of in accordance with current SGS Alaska Division protocol.

7.0. APPARATUS:

7.1. Instrument: Shimadzu TOC-V (or equivalent) equipped with a solids sample module (SSM-5000A).

7.1.1. For instrument maintenance and troubleshooting, refer to the instrument manual. For computer hardware and/or software, refer to IT.

7.2. Eppendorf adjustable pipettes or equivalent.

7.3. Analytical balance.

7.4. Ceramic wool or equivalent.

7.5. Steel forceps.

7.6. Standard tongue depressors.

7.7. Ceramic sample boats.

7.8. Combustion Tube.

7.9. Platinum and cobalt catalyst.

7.10. Desiccator (houses sample boats after burn-out).

8.0. REAGENTS:

8.1 Reagent water: Deionized (DI) water.

8.2 Reagent acid, Nitric Acid (1:1): Slowly add one part of concentrated nitric acid to one part DI water.

8.3 Calibration Standard, 10,000 mg/L of organic Carbon: To make: dissolve 21.26g of Potassium Hydrogen Phthalate (KHP) in 1 liter of deionized water.

8.4 Continuing Calibration Verification (CCV), 10,000 mg/L: The same as solution 8.3.

8.5 Laboratory Control Sample (LCS), 33510 mg/Kg: The LCS sample is a commercially prepared soil Buffalo River Sediment from NIST that is ≈ 3% carbon from a source other than the calibration standard. Standard is stored in an area at room temperature free of dust accumulation. Expiration date for standard follows manufacturer's expiration date.

8.6 Disposal of Reagent: All standards must be disposed of as determined by laboratory protocol.

9.0. EXTRACTION:

9.1. Sample preparation:

9.1.1. Homogenize sample. Sample may need to be ground or pulverized to effectively homogenize. Use a 500-mg representative portion of the prepared sample for analysis. Less sample size may be used if TOC is above upper range of curve.

9.1.2. Tare analytical balance with sample boat and weigh ~ 500 mg of sample directly into boat.

9.1.3. Prepare a matrix spike by adding 100 uL of calibration standard (8.3.) to an appropriate pre-weighed sample size. A matrix spike is prepared for every 10 field samples.

9.1.4. Proceed to add 1:1 nitric acid drop-by-drop until effervescence stops. This acidifies the sample and removes inorganic carbon present. The analyst must make sure that the sample is thoroughly acidified.

9.1.5. Dry the sample in oven set at 105ºC for approximately 10 minutes, or until dry. (Note: When the samples are warmed to 105ºC, some volatile organics may be lost.)

9.1.6. At appropriate time remove sample from oven and place into the SSM 5000A. Enter sample weight when prompted by the software.

10.0. CALIBRATION:

10.1 A warm-up period of approximately 1 hour is required for the combustion chamber and the detector to reach temperature and to stabilize. The instrument monitors these parameters and will not allow you to operate until the system is stable. These parameters can be checked through the software by going to the tool bar at the top of the Sample Table window and selecting Instrument. Select background monitoring, which will allow you to check the temperature of the dehumidifier (1.0-1.1° C) and the stability of the

detector through the base line fluctuation indicator (green or red light). The combustion chamber temperature is displayed digitally on the SSM-5000A.

10.1.1 NOTE: Make sure that the UHP O_2 tank is turned on before the combustion chamber reaches optimal temperature. UHP O_2 pressure regulator must be set at 60psi.

10.1.2 Before beginning analysis, verify the water level of the drain vessel. Fill with DI water if necessary.

10.2 Calibrations are done as required. Calibrations may need to be done if there are new standards in use, or if QC standards do not meet QC criteria more than twice. Refer to corrective action table for when to recalibrate for QC failures.

10.3 Calibration Standard: The instrument is calibrated with a 7-point standardization using the calibration standard solution from section 8.3. The CCV solution (8.3), LCS standard (8.5), and calibration blank are all analyzed to verify the calibration before samples are analyzed.

10.4 Calibration curve: (Based off final mass of 500 mg)

10.4.1 0.00% standard: analyze a blank sample boat containing ceramic wool.

10.4.2 0.05% standard: Add 25 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.4.3 0.10% standard: Add 50 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.4.4 0.20% standard: Add 100 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.4.5 0.50% standard: Add 250 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.4.6 1.00% standard: Add 500 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.4.7 2.00% standard: Add 1000 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.5 Continuing Calibration Verification (CCV), 0.50%: Add 250 uL of calibration standard, 10,000 mg/L of Carbon (8.3.) to sample boat containing ceramic wool.

10.6 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS/LCSD), 33510 mg/Kg (3.35%): Weigh approximately 0.10-0.125 g of Buffalo River Sediment (8.5) directly into a sample boat.

10.7 **Getting into the TOC-Control V program:**

- Double click on TOC-Control V icon on desktop.
- Double click on Sample Table Editor. Leave the username and password field blank and hit “OK”.

10.8 **Creating a new calibration**

- Create a new file. The file must be saved as the day's date before continuing (curvemmddyysoil); soil is added to the end of the file name to identify it as a soils batch.

- Place cursor on the second row in the spread sheet and go to the *Insert* drop down menu at the top of the page and choose Calibration Curve. This will open the calibration folder. Select the most recent calibration curve, which will contain the necessary points and parameters.

- Press the "Go" button and enter and remove the samples when prompted. A new calibration file will be created when the analysis is finished.

11.0. ANALYSIS:

11.1. Start up the instrument and software as directed in 10.1 and 10.7.

11.2. **Set up for TOC soil run:**

11.2.1. Create a new file. The file must be saved as the day's date before continuing (mmddyysoil); soil is added to the end of the file name to identify it as a soils batch.

11.2.2. Go to the *Insert* drop down menu at top of page and choose Sample. This will cause the Sample Wizard to be activated. You have the option to add more than one sample at a time by using the Auto Generation option.

11.2.3. Sample Wizard:

11.2.3.1. At the end of the Calibration dialog box there is a button with three dots on it (…). Click on the button and choose the most recent calibration curve.

11.2.3.2. Leave the sample name as <Untitled>. The name will be entered on the spreadsheet later.

11.2.3.3. The next step tells the sample what units to use (%). Once this has been established in the method set up you will only have to verify the information.

11.2.3.4. The next steps of the sample wizard are not used. Just keep clicking next until it says finish. Once you hit finish it will put the sample into the spreadsheet.

11.2.3.5. Place the sample ID's into the spreadsheet.

11.2.3.6. Press the Start button to begin analysis. Insert and remove samples as prompted by the software.

11.3. Clean the sample boats by scraping soil residue out of them and placing them in a muffle furnace at 900^{o}C for approximately 1 hour.

12.0. QUALITY CONTROL:

12.1 Any corrective action(s) needed to address a QC outlier or other technical challenge that is not listed in the subject SOP requires the prior written approval of the QA Manager or Technical Director.

12.2 The calibration curve must have a correlation coefficient greater than or equal to 0.995. A standard may be reanalyzed during calibration if the peak area is outside the estimated value or the instrument fails to complete analysis. See the Inorganic Section Quality SOP (SOP 500) for further information.

12.3 A continuing calibration verification (CCV) is analyzed after the calibration. A CCV is analyzed at the start of every run, after every 10 samples and at the end of the analysis sequence. The CCV result must be within 25% of the true value.

12.3.1 All methods for DOD samples**:** In the event of a recovery failure for analytes of interest in continuing or closing CCV samples, the Analyst may immediately (within 1 hr of the failing CCV and before any other samples have acquired) rerun 2 successive CCVs. If both meet QC criteria for all analytes of interest, then the samples already analyzed may be reported and the run sequence may be continued. Samples following the two passing CCVs are also valid. **All CCVs must be reported in LIMS.**

12.3.2 All methods for non-DOD samples: In the event of a recovery failure for analytes of interest in CCV samples, the analyst may rerun the CCV once. If the rerun meets QC criteria, then samples already analyzed must be reanalyzed with successful bracketing CCVs; however, the run sequence may be continued. Samples following the passing CCV are valid. All CCVs must be reported in LIMS.

12.4 The method blank (MB) is analyzed once per 20 field samples before any samples can be run and must read < ½ the LOQ. Thereafter, the calibration blank (CB) is analyzed after every 10 samples and at the end of each run. The acceptance criteria for the CB is < the LOQ. A measured MB > ½ LOQ or CB > LOQ may require reanalysis of samples. Samples that have an apparent concentration < LOQ will not require reanalysis. Samples having a concentration > 10X the CB or MB contamination will not require reanalysis. All other samples analyzed since the last acceptable blank require reanalysis.

12.5 The initial CCV and CB analyses are immediately followed by a Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) once per 20 field samples. The LCS is a standard from a source other than the calibration standard. The LCS must be within 25% of the true value before sample analysis can begin. If the LCS recovers outside acceptance criteria, it may be reanalyzed once. Otherwise, recalibration is performed.

12.6 Refer to Attachment A for specific corrective actions. Any corrective action needed to address a QC outlier that is not listed in this SOP requires the approval of the QA Manager or Technical Director.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 The recoveries for standard solutions (both digested standards and the periodic quality control standards) are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{Vo}{Ve} \times 100 = \% \text{ recovery}$$

Vo = the observed value
Ve = the expected value

13.2 The calculation for spike recoveries requires the subtraction of the sample contribution from the response of the spiked sample, the division of this result by the expected value of the spike, and the multiplication by 100 yields percent recovery.

$$\frac{(Vo-Sc)}{(Ve)} \times 100 = \% \text{ recovery}$$

Vo = the observed value of the spiked sample in % (m/m).
Sc = the observed value of the sample in % (m/m).
Ve = the expected value of the spike in % (m/m).

13.3 The expected value of the spike requires the multiplication of the spike solution concentration and the volume used of spike solution, the division of this result by the mass of the spiked sample.

$$\frac{(Sc)(Vs)\ X100}{Sm} = \text{expected value of spike in \% (m/m)}$$

Sc = the concentration of the spike solution in g/L.
Vs = the volume of spike used in L.
Sm = the mass of the spiked sample in g.

13.4 The relative percent difference between duplicate samples is calculated as the absolute difference between the sample and the duplicate, divided by the average of the sample and the duplicate, all multiplied by 100.

$$\frac{|\ Sc - Dc\ |}{\{(Sc + Dc) / 2\}} \times 100 = \text{Duplicate Relative Percent Difference (DUP RPD)}$$

Sc = observed sample concentration
Dc = observed duplicate sample concentration

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.4. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples includes a lab coat, gloves and safety glasses. In addition, when handling concentrated acids or bases, a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

There are no method performance measures to report currently.

17.0. DETECTION LIMIT (DL) STUDY:

A detection limit (DL) study is performed when a significant change in instrument response is observed, or when a new instrument is purchased for analysis. The DL information given in this document is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. The suggested spiking amount is 0.025%.

18.0. LIMIT OF DETECTION (LOD):

The LOD for TOC is 0.025%.

19.0. LIMIT OF QUANTITATION (LOQ):

The lab LOQ for TOC is 0.05%.

20.0. REFERENCES:

SW 9060A

Shimadzu SSI-AP-TOC-104

Shimadzu SCA-130-513

21.0. ATTACHMENTS:

Attachment A: Corrective Action Table

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Attachment A: Corrective Action Table

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SGS SOP	Calibration Blank (CB)	1 per 10 sample readings, and at the end of the run.	Concentration < LOQ	1. Repeat analysis. 2. Evaluate samples before further analysis. If < LOQ, no reanalysis is required. If > 10X concentration in blank, no reanalysis is required. All other samples using the contaminated blank for QC will be reanalyzed.
	Calibration Verification Std. (CCV)	At the beginning of analysis, 1 per 10 field samples, and at end of the run.	Recovery ± 25%	**For non-DOD samples:** 1. Repeat analysis. 2. If repeat meets QC criteria, reanalyze affected samples. 3. If QC criteria not met, then recalibrate and reanalyze. **For DOD samples:** 1. Immediately (within 1 hr and before any other samples have acquired) run two successive CCVs. 2. If both meet QC criteria, then samples already analyzed may be reported and the run may continue.
	Method Blank (initial Calibration Blank)	1 per 20 field samples.	< ½ LOQ	1. Repeat analysis. 2. Evaluate samples before further analysis. If < LOQ, no reanalysis is required. If > 10X concentration in blank, no reanalysis is required. All other samples using the contaminated blank for QC will be reanalyzed.
	Laboratory Control sample (LCS)/ Laboratory Control sample duplicate (LCSD)	1 per 20 field samples.	Recovery ± 25% RPD 25%	1. Repeat analysis. 2. Recalibrate and repeat analysis of all associated samples.
	Matrix Spike (MS)/ Matrix Spike Duplicate (MSD)	1 per 10 field samples. As needed	Recovery ± 25% MS/MSD RPD 50%	1. Evaluate LCS; if in control, then flag as Non-homogenous. 2. Qualify with LCS/LCSD RPD.

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Total Organic Carbon and Dissolved Organic Carbon in Water	**Revision Date:** December 2020
Method No: SM5310B/SW9060A	**SOP No:** 321r17
Page: 1 of 13	**Supersedes:** 321r16

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C Ede	12/15/20	Tamara Rentz	12/15/2020
Stephen C. Ede	1/26/22	Tamara Rentz	1/26/22
Stephen C. Ede	4/6/23	Tamara Rentz	4/6/23

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Tamara Rentz 2022.01.26 14:41:05 -09'00'

Tamara Rentz 2020.12.15 13:29:22 -09'00'

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Summary of Changes from Previous Revision:

- Edited section 6.3.
- Edited section 7.6.
- Updated the referenced section in section 10.3.
- Edited section 10.5. and 10.5.1.
- Removed section 10.5.2. and incorporated drinking water MB requirement into 10.5.1.
- Updated the refenced section in section 11.3.
- Edited section 12.3 and 12.3.1.
- Edited section 12.6.
- Edited section 12.8.
- Updated edition of reference in section 20.0.
- Minor spelling and grammatical changes were made throughout this SOP.

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1.0. OBJECTIVE:

This standard operating procedure (SOP) outlines the method of determining the nonpurgeable total organic carbon (TOC) and dissolved organic carbon (DOC) content in water by high temperature combustion.

2.0. SCOPE AND APPLICATION:

This SOP is applicable to the determination of TOC in drinking, surface, ground waters, domestic and industrial wastewater.

Before analysis the sample is acidified to a pH of ≤ 2 with Hydrochloric Acid (HCL) to convert inorganic carbonates and bicarbonates to carbon dioxide. Samples for DOC are filtered prior to preservation and the pH is tested using a pH strip. A portion of the sample is measured out into a 40-ml non-preserved vial and loaded into the autosampler. The sample is then sparged with O_2 to remove carbon dioxide and some purgeable organic carbon compounds. Following purging, the sample is injected into the combustion tube where it is oxidized. The sample then passes through a dehumidifier where the remaining water is removed, and finally through a halogen scrubber to remove possible interference. The sample is delivered to the infrared CO_2 detector where the amount of CO_2 detected is directly proportional to the carbon content of the sample.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 SGS preserves samples with HCl. The instrument is equipped with a halogen scrubber to remove chloride.

3.2 SW9060A states that the samples should be homogenized with a blender. SGS homogenizes samples by thoroughly shaking them.

3.3 Samples are purged with Oxygen instead of Nitrogen for five minutes.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Removal of carbonate and bicarbonate by acidification and purging with purified O_2 gas may result in the loss of volatile organic substances. Volatiles may also be lost with increasing sample temperature.

5.2. Samples such as seawaters or high salinity liquids may have exceedingly high halogen content, which could inhibit the formation of CO_2 (approaching 0.1%). The TOC-V CSN analyzer comes equipped with a halogen scrubber that removes any interference due to the halogens present in the sample.

5.3. Samples preserved with H_2SO_4 must be diluted by a factor of no less than 10x due to possible damage to the detector by the sulfate (SO_4) content.

6.0. SAMPLE HANDLING:

6.1. Samples need to be kept at 0-6°C to minimize the bacterial decomposition and volatilization of organic compounds.

6.2. Samples must be collected in amber glass bottles and acidified to a pH of 2 with HCL upon collection. Samples should be stored at 0-6°C with minimum exposure to light and atmosphere. Analysis is to be completed within 28 days from sample collection.

6.3. Dissolved Organic Carbon (DOC): Samples that require dissolved organic carbon will be field filtered. If samples are not field filtered, they must be lab filtered when they are received. Unpreserved samples must be filtered within 48 hours of sampling prior to acid preservation. A 0.45-µm pore diameter syringe filter will be rinsed with 10-20mL of DI water. A filter blank is then collected with the same filter disc. Also, with the same filter disc, filter 30mL of the sample and preserve it with HCl. Test the pH of the samples to ensure that the pH is less than 2.

7.0. APPARATUS:

7.1. Instrument: Shimadzu TOC-V CSN with autosampler (ASI-V).

7.1.1. For instrument maintenance and troubleshooting, refer to the instrument manual. For computer hardware and/or software, refer to IT.

7.2. Glassware: 40mL amber vial, non-preserved, septa cover.

7.3. UHP Oxygen tank.

7.4. Combustion tube. (Purchased through Shimadzu).

7.5. Platinum Catalyst. (Purchased through Shimadzu).

7.6. ceramic wool or equivalent. (Purchased through Shimadzu).

7.7. Platinum Screen. (Purchased through Shimadzu).

7.8. Hydrochloric Acid.

7.9. 0.45-µm pore diameter filters.

8.0. REAGENTS:

8.1. External / Internal water source – Deionizer (DI) water directly off the filter line, daily.

8.2. Diluent: 0.2% Hydrochloric Acid (HCL). Add 2 mL of HCL to 1000 mL of DI water using a Class A volumetric flask. It is important that the pH of the diluent is ≤ 2. *Note: Changing the diluent may necessitate recalibration of the instrument.*

8.3. Standard Calibration Stock Solution, 1,000 mg/L: Purchased commercially

8.4. Inorganic Carbon (IC) Solution, 1,000 mg/L Carbon: Purchased commercially

8.4.1. Working Inorganic Carbon Check: 75mg/L: Dilute 1.125mL of stock QCS solution and 2.250mL of IC solution to 30mL with diluent.

8.5. Calibration Blank (CB)/Method Blank (MB): 30mL of TOC Diluent (section 8.2).

8.6. Quality Control Standard (QCS), 2000mg/L: Weigh out approximately 0.5 grams of KHP into a beaker and dry in a 105°C drying oven for half an hour. Using a volumetric flask, dissolve 0.425g of the dried KHP in 80mL of DI water and dilute to 100mL in a volumetric flask. Store in an amber jar at room temperature. *Note: refrigerating the QCS causes precipitation*. The shelf life is one year. This standard must be from a source other than the one used to calibrate the instrument.

8.6.1. Working CCV solution/LCS solution, 75mg/L: Dilute 1.125 mL of stock QCS solution to 30mL with diluent. The solution is prepared fresh, daily.

8.7. Instrument Cleanout (C/O), 0.000mg/L: 30mL of TOC Diluent (section 8.2).

8.7.1. Six injections per cleanout are needed.

8.7.2. A cleanout is the first injection before any calibration or analysis.

9.0. EXTRACTION:

N/A

10.0. CALIBRATION:

10.1. Calibrate the instrument periodically or as needed.

10.2. To start up the instrument for calibration refer to sections 11.4. through 11.6.

10.3. A standard curve of 8 standards is then prepared by serially diluting the working standard (section 8.3.). The standard concentrations are: 0.0 mg/L, 0.25mg/L, 0.5 mg/L, 1.0 mg/L, 2.0 mg/L, 10.0 mg/L, 50 mg/L, and 100 mg/L. The minimum acceptable r^2 value is greater than or equal to 0.995. Failure to meet this requirement necessitates recalibration.

10.3.1. 100 mg/L: dilute 3 mL of the calibration stock standard (section 8.3) to 30 mL with diluent (section 8.2.).

10.3.2. 50 mg/L: dilute 1.5 mL of calibration stock standard (section 8.3) to 30 mL with diluent (section 8.2.).

10.3.3. 10 mg/L: dilute 300 µL of calibration stock standard (section 8.3) to 30 mL with diluent (section 8.2.).

10.3.4. 2.0 mg/L: dilute 60 µL of calibration stock standard (section 8.3) to 30 mL with diluent (section 8.2.).

10.3.5. 1.0 mg/L: dilute 30 µL of calibration stock standard (section 8.3) to 30 mL with diluent (section 8.2.).

10.3.6. 0.5 mg/L: dilute 150 µL of calibration standard (section 10.3.1) to 30 mL with diluent (section 8.2.).

10.3.7. 0.25mg/L: dilute 75uL of calibration standard (section 10.3.1) to 30 mL with diluent (section 8.2.).

10.3.8. 0.0mg/L: 30mL diluents (section 8.2.)

10.4 Setting up instrument software for calibration.

10.4.1 After the program is opened, choose “Sample Table Editor” from window. Select new sample table and highlight instrument, ASI-V, from menu and connect (right-click and select ‘connect’).

10.4.2 Insert a clean out sample (section 8.7) prior to running the calibration.

10.4.2.1 Go to insert, then insert sample. The sample wizard will appear.

10.4.2.1.1 *Note: The following is for calibration only,* ***do not use*** *these steps to set up a C/O before an analytical run:* In the first page of the sample wizard select Method and then click the “…” button. Select “conditioningmethod.met”.

10.4.2.1.2 Enter the sample name as “C/O” on page two of the sample wizard.

10.4.2.1.3 On the Injection Parameters window confirm the following: the number of injections 6/6, the injection volume to 150 µL and the sparge time to 5:00 and Multiple Injections is selected. Then select finish on the sample wizard menu.

10.4.2.2 From the Insert file menu select ‘Calibration Curve’. Choose the latest valid calibration to copy the calibration parameters (i.e. curve points, amount per injection, number of injections, etc.). Number the location of the calibrants. To change any parameters of the curve, right click on “calibration” in the left most section of the main window. Select properties.

10.5 After calibration, a method blank (MB) (section 8.5.), a 75 mg/L CCV (section 8.6.1.) and IC check solution (section 8.4.1) are analyzed.

10.5.1 Acceptance criteria: MB less than the LOQ (less than ½ LOQ for DOD criteria, <DL for drinking water samples), CCV ± 10% and the TOC concentration between the CCV and IC should agree within 25 RPD. If all parameters above are met, calibration is complete, and samples are analyzed.

11.0 ANALYSIS:

11.1. Before analysis, the sample pH must be verified to be ≤ 2, which is done at sample receiving. Approximately 30mL of samples will be shaken and poured into a 40mL glass amber vial (non-preserved) for analyses. The samples are sparged with UHP O_2 for 5 minutes to remove inorganic and purgeable organic carbon which is done automatically by the instrument prior to analysis.

11.2 Samples are injected and analyzed with the CCV and CB at the beginning, after every 10 injections and at the end of the run.

11.3 Prepare a matrix spike and matrix spike duplicate (MS/MSD) at a frequency of one set per batch of no more then 20 paying samples. Prepare the MS/MSD samples by diluting 300µL of Calibration stock solution (section 8.3.) with 30 mL of sample. The spike level is 10 mg/L.

11.4 A warm-up period of 20 minutes is required for the combustion chamber and the detector to reach temperature and to stabilize. The instrument monitors these parameters and will not allow operation until the system is stable. These parameters can be checked through the software by going to the tool bar at the top of the Sample Table window and selecting “Instrument”. Select “background monitoring”, which will allow monitoring of the combustion chamber temperature (680°C, per instrument manufacturer instructions), the dehumidifier temperature (1.0-1.1°C), and the stability of the detector though the base line fluctuation indicator (green or red light).

11.4.1 NOTE: Make sure that the UHP O_2 tank is turned on before the combustion chamber reaches optimal temperature.

11.4.2 UHP O_2 pressure regulator must be set at 60psi. Instrument pressure gauge must read 210kPa.

11.5 Before beginning the analysis, check the contents of the waste container and empty as needed. Verify that the drain line is inserted securely into waste container.

11.6 Verification of the internal/ external container water levels.

11.6.1 The internal “Cooler Drain Container” and the “Humidifier container” of the TOC-V CSN instrument must be at the required water level before starting a run. Add DI water if water level is below marking.

11.6.2 The external “Rinse Water Container” of the Autosampler (ASI-V) must be at the required water levels before starting a run. Add DI water if the water level is below marking.

11.7 Sample results are derived from the average of four injections. If the SD of four injections is greater than 10% of the average, another injection is made and the closest four are reported. If the sample mean area is above the mean area of the high point on the calibration curve, sample must be analyzed again with a dilution factor.

11.8 Instrument Software Set-up.

11.8.1 After the program is opened, choose "Sample Table Editor" from window. Highlight instrument, ASI-V, from menu and connect (right-click and select 'connect').

11.8.2 Inputting sample ID into computer run-log.

11.8.2.1 From the Insert file menu select 'Sample'.

11.8.2.2 Parameter Source (page 1 of sample wizard): Choose the most current calibration curve that meets minimum requirements (section 10.3.).

11.8.2.3 Analysis Information (page 2 of sample wizard): The sample ID may be entered.

11.8.2.4 Calibration Curve (page 3 of sample wizard): Confirm the correct calibration is selected for calibration curve 1 and calibration curve 2 and 3 should be blank.

11.8.2.5 Injection Parameters (page 4 of sample wizard): Verify the following injection parameters:

Units	mg/L
Injection Volume	150 uL
Expected Conc. Range	100.0
No. of Injections	4-5
SD Max	0.1000
CV Max	2.00
No. of Washes	3
Auto Dilution	1
Sparge Time	5.00
Acid Addition	0.0
Multiple Injections	Checked
Auto Correction of inj. Vol and dilution	Not Checked

11.8.2.5.1 When setting up a C/O set the minimum injections to 6.

11.8.2.6 Peak Time Parameters (page 5 of sample wizard): Verify that the "Use default settings" checkbox is marked and select 'Finish'.

11.8.2.7 Enter the position number of the sampler tray where the sample will be placed.

11.8.2.7.1 NOTE: Vials need to be positioned properly into place-holders to prevent sample-needle from damage.

11.9 Starting an analysis:

11.9.1 Verify the O_2 gas (section 7.3.) valve on.

11.9.2 From the Instrument File Menu, choose 'connect'.

11.9.3 Verify instrument status by running a background monitor. When all the indicators have the green light on proceed to starting analysis.

11.9.4 From the Instrument File Menu, choose 'start (continue)'. Click on the Standby button.

11.9.5 Verify the vial position in the Sparging / Acid Addition window.

11.9.6 Start ASI Measurement window: Save as the sample table with the days date and click the "Start" button to begin run.

11.10 To shut down the instrument click the standby button. When the "Standby" window appears, select option 'shut down instrument.' The instrument will shut down after 30 minutes. Turn off the UHP oxygen.

12.0 QUALITY CONTROL:

12.1 Removal of inorganic carbon must be checked with each calibration. This is done by spiking a CCV with inorganic carbon (a commercially purchased solution) so that the inorganic carbon concentration is similar to that of the organic carbon in the sample. The TOC results from the unspiked (1st CCV) and spiked sample should agree within 25 RPD. The RPD needs to be documented in the logbook for every calibration (see calculation 13.3).

12.2 A Continuing Calibration Verification (CCV) solution and a calibration blank (CB) are analyzed at ten percent intervals. The CCV and CB are documented in the logbook and double posted as an LCS and MB with every twenty samples. Re-calibration is performed when the CCV or the CB does not meet quality control specifications.

12.3 Recoveries for the CCV and LCS samples must be within +/- 10% of the true value initially after calibration. Subsequent CCV and LCS recoveries must be within +/- 20% of the true value. If the CCV recovery is outside this range, two repeat analyses are permitted (for DOD samples). If the CCV is still outside limits, the analysis must be stopped, the problem corrected, and all samples analyzed since the last acceptable CCV are reanalyzed. Recalibration may also be necessary.

12.3.1 All methods for non-DOD samples: In the event of a recovery failure for analytes of interest in CCV samples, the analyst may rerun the CCV once. If the rerun meets QC criteria, then samples already analyzed must be reanalyzed with successful bracketing CCVs; however, the run sequence may be continued. Samples following the passing CCV are valid. All CCVs must be reported in LIMS.

12.4 The criteria for matrix spike recovery are +/- 25%. Recoveries outside this range will require the reanalysis of the matrix spike samples. Samples that are detectable may be diluted and respiked. Failure Action: Flag the original sample and the matrix spike sample with the appropriate comment indicating matrix interference and bias.

12.5 The acceptance criteria for the relative percent difference between the matrix spike and matrix spike duplicate is ≤ 25. If RPD is found to be > 25 the parent sample, MS and MSD will be reanalyzed; if RPD is still found to be > 25 the parent sample will be flagged as heterogeneous.

12.6 The Method blank (MB) acceptance criterion is less the LOQ (< ½ LOQ for DOD criteria, < DL for drinking water samples). Samples with a concentration > 10x the blank contamination will not require reanalysis. Samples that have an apparent concentration < LOQ will not require reanalysis. All other samples must be reanalyzed after the contamination problem has been fixed.

12.7 The calibration blank (CB) acceptance criterion is less than the LOQ. Measurements outside this range will stop the analytical process and may require sample qualifications. Samples with a concentration >10X the blank contamination will not require reanalysis. Samples that have apparent concentration < LOQ will not require reanalysis. All other samples must be reanalyzed after the problem has been fixed.

12.8 Low level Quantitation (LLQ): the LLQ is reagent water that has been spiked at the concentration of the LOQ. One LLQ will be prepared and analyzed each analytical day for drinking water samples.

13.0 CALCULATIONS, REVIEW AND REPORTING:

13.1 Percent recovery for CCV and LCS standard solutions are calculated by dividing the observed value (Vo) by the expected value (Ve). The result is multiplied by 100 to give a percent recovery.

$$\frac{Vo}{Ve} \times 100 = \% \text{ recovery}$$

Where Vo = the observed value of the sample
Ve = the expected value of the sample

13.2 Percent recovery calculation for spike samples requires the subtraction of the sample contribution from the response of the spiked sample, the division of this result by the expected value of the spike, and the multiplication by 100 yields percent recovery.

$$\frac{Vo-Sc}{Ve} \times 100 = \% \text{ recovery}$$

Where Vo = the observed value of the spiked sample
Sc = the observed value of the sample
Ve = the expected value of the spike

13.3 The relative percent difference between matrix spike and matrix spike duplicate samples is calculated as the absolute value of the difference between the MS and MSD, divided by the average of the MS and MSD, this value multiplied by 100.

$$\frac{Sc - Dc}{\{(Sc + Dc) / 2\}} \times 100 = \text{Duplicate Relative Percent Difference (Dup RPD)}$$

Where Sc = observed sample concentration
Dc = observed duplicate sample concentration

14.0 HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling any samples or solutions with any acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

17.1. A DL study is performed in accordance with SGS SOP 116 (i.e., a significant change to the instrumentation is implemented, when a new instrument is purchased for analysis). The DL is intended to demonstrate the capability of this method as it is implemented at SGS.

17.1.1. SW9060A: In order to meet the requirements of the DOD QSM, a DL study must be performed when a new instrument is put into use or as a result of a quarterly LOD verification failure. Please refer to SOP 116 for future guidance.

17.1.2. SM5310B: In order to meet the ADEC drinking water requirements, a DL study must be performed initially for each drinking water Analyst and then on an annual basis. Please refer to SOP 116 for further guidance.

18.0. LIMIT OF DETECTION (LOD):

The LOD is defined per SOP 116; verification shall be performed quarterly according to the schedule set by the QA Office.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP 116; verification shall be performed quarterly according to the schedule set by the QA Office.

20.0. REFERENCES:

SW 9060A
Standard Method 5310B, 23rd edition.

21.0. ATTACHMENTS:

ATTACHMENT A: Corrective Action Table

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Attachment A: Corrective Action Table

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM 5310B	Calibration Blank	1 per 10 sample readings, and at end of run.	Concentration less than LOQ.	1. Evaluate samples before further analysis If samples < LOQ or > 10x level of contamination, no reanalysis is required. All other samples must be reanalyzed with valid calibration blank. Re-calibration may be required.
	Filter Blank (if required)	1 per batch of filtered samples	Concentration less than LOQ.	1. Evaluate samples that correspond to the filter blank. If samples < LOQ or > 10x level of contamination, no reanalysis is required. All other samples must be reanalyzed with valid filter blank.
	Inorganic Carbon Check	After each calibration	RPD ≤ 25	1. Evaluate inorganic carbon scrubbing system and perform maintenance as needed prior to reanalysis
	CCV, 75mg/L 2nd source	After each calibration At the start of the run, after every 10 samples, and at end of run.	Recovery ± 10% Recovery ± 20%	1. Repeat analysis twice within 1 hr. of failure and before other samples (DOD). If both meet QC criteria, associated samples can be posted. 2. Repeat analysis once (non-DOD). If passing, reanalyze all associated samples. 3. Re-calibrate and repeat analysis of all associated samples.
	Method Blank (Initial Calibration Blank) MB for drinking water	After calibration; a MB is required for each 20 batch samples.	Concentration < LOQ (½ LOQ for DOD) Evaluate at the DL	1. Evaluate samples before further analysis. If samples < LOQ or > 10x level of contamination, no reanalysis is required. All other samples must be reanalyzed with valid calibration blank 2. Re-calibration may be required.
	LCS, 75mg/L (Initial CCV)	Once for every 20 samples	Recovery ± 20%	1. Repeat analysis twice within 1 hr. of failure and before other samples (DOD). If both meet QC criteria, associated samples can be posted. 2. Repeat analysis once (non-DOD). If passing, reanalyze all associated samples. 3. Re-calibrate and repeat analysis of all associated samples.
	MS/MSD	1 per 20 samples or 1 per batch	Recovery ± 25% MS/MSD RPD ≤ 25%	1. Evaluate LCS; if in control, then flag as possible matrix interference. 2. If RPD is out of control, reanalyze parent sample, MS and MSD. 3. If RPD is still out of control, flag as parent sample as being non-homogeneous.
	Multipoint Calibration	Periodic or as needed	$R^2 \geq 0.995$	1. Determine source of problem and correct. 2. Repeat calibration. 3. Calibrate pipettes before making curve points.

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Determination of Metals by ICP-MS	**Revision Date**: April 2023
Method No: SW6020B	**SOP No**: 342r28
Page: 1 of 29	**Supersedes:** 342r27

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	4/17/23	Samara Rentz	4/17/23

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Addendum #1 added 9/17/2023 TPR

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Determination of Metals by ICP-MS	**Revision Date**: September 2023
Method No: SW6020B	**SOP No**: 342r28 Addendum #1
Page: 1 of 1	**Supersedes**: 342r27

Signatures below reflect approval for the following changes to the current SOP. These changes will be incorporated into the SOP during the next review. This addendum will be incorporated into the electronic SOP (i.e., PDF file).

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/15/23	Samara Beatty	9/15/23

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

The above referenced SOP should be modified as follows:

Change Leachate Blank (LB) in Attachment E from:

SW6020B	Leachate Blank (LB)	1 per 3010 TCLP batch of 20 samples or less	Concentration ~~< LOQ~~ For DoD: ~~< LOD~~	1. Same as for the MB. See above.

To:

SW6020B	Leachate Blank (LB)	1 per 3010 TCLP batch of 20 samples or less	Concentration < ½ LOQ For DoD: < ½ LOQ	2. Same as for the MB. See above.

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Summary of Changes from Previous Revision:

- Incorporated Addendum#1 in section 8.0, 12.0 and Attachment E
- Updated 8.19 to reflect current calibration practices
- Updated 11.3.2.4 to reflect current Agilent practices
- Updated 12.3.7 and Attachment E to reflect current MB corrective action requirements
- Updated Attachment A to include the correct Kr isotope
- Updated Attachment letters and position order to reflect the order in which they are referenced within the SOP

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1.0. OBJECTIVE:

This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples according to SW846 Method 6020B for the elements listed as analytes in Attachment A using an Inductively Coupled Plasma-Mass Spectrometer or ICP-MS.

2.0. SCOPE AND APPLICATION:

2.1 This method is applicable to the following sample matrices: ground waters, surface waters, industrial wastes, sludge, oil, and soil samples.

2.2 NexIon 300D: Software Basics, Trouble Shooting Guide, and Hardware Basics may be found in the Perkin-Elmer Customer Training Course.

2.3 Agilent 7800 ICP-MS: Software Basics, Trouble Shooting Guide, and Hardware Basics may be found in Agilent 7800/7900 ICP-MS Hardware Maintenance Manual.

2.4 Detailed information regarding requirements by U.S. EPA SW-846 Method 6020B may be found in Revision 2 of Method 6020B.

2.5 Initial Performance Data: It is the responsibility of the user of this SOP to generate the method specific performance data on the user's specific instrument before the analysis of any samples.

2.6 Aqueous sample, digestates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into an R.F. plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 Section 11.1 of the reference method indicates that gold should be added to preserve the mercury and to prevent it from plating out in the sample introduction system. Section 11.4 of this SOP outlines the features of the current technology which address this concern.

3.2 EPA 6020B requires a digested LLQC at the LOQ level with a ± 30% recovery. SGS practice is to perform a quarterly LOQ verification at 1-2x LOQ with ± 50% recovery. As such, LOQ's are digested and analyzed every quarter and will fulfill this requirement.

3.3 EPA 6020B does not require an LLQC throughout the analytical run. LLIQCs shall be run at the beginning of the run with 20% recovery criteria for DOD and 30% recovery criteria for non-DOD.

4.0. RESPONSIBILITIES:

4.1. The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2. The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3. It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4. This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be returned to the QA Office. If there are no addenda to incorporate or updates to make, the SOP is reviewed and given a new revision number. The cover page is signed and dated by the Technical Director and QA then distributed.

4.5. A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff has "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0 INTERFERENCES:

5.1 Isobaric interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98). Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interferences are already present as default interference equations in the software.

Note: Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

5.2 Molecular interferences are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.) Predictions about the type of molecular interferences may be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $Ar^{40}Cl^{35}$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($Ar^{40}Cl^{37}$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl^{37} to Cl^{35}.

5.3 This instrument method contains correction equations to compensate for the above mentioned interferences. All samples analyzed by this method are subject to correction, the extent of which is based on sample matrix. It is the responsibility of the analyst to identify possible matrix problems and produce data of known quality. All data users should be aware that these corrections are possible without explicit notification.

5.4 Any sample with silver concentrations over calibration range (100 µg/L on actual concentration) at the standard dilution will be re-extracted at a lower sample mass/volume.

5.5 NexIon 300D handles interferences in three ways: Inter-element corrections, reaction cell gas, collision cell gas.

5.5.1 DRC (Dynamic Reaction Cell): Determines interfering molecular ion intensities at an alternative mass (polyatomic correction). A reactive gas (ammonia) selectively targets interferent or isotope of interest.

5.5.2 KED (Kinetic Energy Discrimination) Mode uses He gas and relies on interferent having a larger cross-sectional area. The gas collides more with the interferent, reducing its kinetic energy. The lower kinetic energy ions are then filtered out of the ion stream.

6.0 SAMPLE HANDLING:

6.1 Refer to SOPs 345 and 361 (Digestion of Aqueous Samples (including TCLP leachates), and Soil Digests respectively) for sample preservation. Digests are stored for one month prior to disposal in accordance with SOP#108.

6.2 Holding Times for all metals in all matrixes, EXCEPT MERCURY are six months. Mercury has a holding time of 28 days.

7.0 APPARATUS:

7.1. NexIon 300D:

7.1.1. Perkin-Elmer NexIon 300D includes the instrument, computer system, software, and autosampler. Instrument capable of scanning the mass range 5-250 amu with a minimum resolution of 1 amu peak width at 5% peak weight.

7.1.2. Recommended peristaltic pump tubing (other sizes may be used to enhance performance or reduce pulsations produced by the pump):

7.1.2.1. Black/Black - 0.76 mm i.d. (for carrier).

7.1.2.2. Orange/Green - 0.38 mm i.d. (for internal standard introduction).

7.1.2.3. Grey/Grey - 1.30 mm i.d. (for drain).

7.1.3. SC- 2DX FAST Autosampler or equivalent.

7.1.4. Argon gas: High purity grade (99.99%).

7.2. Agilent 7800 ICP-MS:

7.2.1. Agilent 7800 ICP-MS includes the instrument, computer system, software, and autosampler. Instrument capable of scanning the mass range 5-250 amu with a minimum resolution of 1 amu peak width at 5% peak weight.

7.2.2. Recommended peristaltic pump tubing (other sizes may be used to enhance performance or reduce pulsations produced by the pump):

7.2.2.1. White/White -1.02 mm i.d. (for carrier and tuning solution).

7.2.2.2. Yellow/Blue - 1.52 mm i.d. (for drain).

7.2.3. SC- 2DX prepFAST Autosampler or equivalent.

7.2.4. Argon gas: High purity grade (99.99%).

7.3. Calibrated mechanical pipettes: 20-200 µL, 200-1000 µL, 1000-5000 µL

7.4. Metal-free plastic pipette tips (for the pipettes specified in Section 7.2).

7.5. 14-mL disposable test tubes. (Fisher catalog No: 14-956-6C or equivalent).

7.6. 50-mL Class A vessels for standards (SCP #010-500-263 or equivalent).

7.7. Instrument maintenance logs must be maintained per SGS SOP 111 and 500.

8.0 REAGENTS:

8.1. All stock solutions/chemicals/reagents must be traceable back to documented records. Refer to SGS SOP 112 and 500 and PX-0001 for proper documentation procedures. The expiration dates of all reagents must be recorded and written on the container. Where applicable, this information must be entered into the LIMS.

Note: This SOP requires 50-mL digestion vessels, 100-mL digestion vessels or a class A Teflon volumetric flask to prepare stock standards and working level solutions. All solutions prepared in these vials are made up to volume using the graduations marked on the side of each vial. The accuracy of these markings has been found to be sufficient for the purpose of the analyses described by this SOP.

8.2. Nitric acid, concentrated. (JT Baker "Instra-Analyzed" # 9598-34) or equivalent.

8.3. Hydrochloric acid, (JT Baker "Instra-Analyzed" # 9530-33) or equivalent.

8.4. Reagent water equivalent to ASTM Type I water (ASTM D 1193).

8.5. Single element or multi-element stock solutions of the following elements as needed for Standards, Tuning Solutions, and Performance Solutions.

Li, Be, B, Al, Sc, V, Cr, Mn, Co, Ni, Cu, Zn, Ge, As, Se, Rh, Mo, Ag, Ir, Sr
Cd, In, Sn, Sb, Ba, Tl, Pb, Bi, U, Na, Mg, Si, P, K, Ca, Ti, Fe, Ce, Th, Hg, Au

8.6. Agilent Stock Tuning Solution (Agilent #5184-3566): 10ug/L Li, Y, Ce, Tl, and Co in 2% HNO or equivalent.

8.7. Agilent Daily Start-up Configuration Solution: Prepare by pipetting 5 mL of Agilent Stock Tuning Solution with approximately 20 mL of reagent water and 1.0 mL concentrated nitric acid. Dilute to 50 mL and mix well.

8.8. NexIon Stock Solutions:

8.8.1. Intermediate Fe Solution: For example, add 2.5 mL of concentrated nitric acid to approximately 20 mL of reagent water in a 50 mL digestion vessel. Pipette the appropriate amount of Fe stock solution. Dilute to 50 mL with reagent water. Refer to **CX-0019** for the current preparation.

8.8.2. NexIon Daily Tuning Stock Solution: Add 5 mL concentrated nitric acid to approximately 30 mL of reagent water in a 100 mL Teflon volumetric flask. Prepare as per **CX-0019**.

8.9. Daily Tune Solutions:

8.9.1. For NexIon: Prepare by pipetting 50 µL of NexIon Daily Tuning Stock Solution (Section 8.8.2.) into a 50-mL digestion vessel filled with approximately 20 mL of reagent water and 1.0 mL of concentrated nitric acid. Dilute to 50 mL with reagent water and mix well.

8.9.2. For Agilent: Prepare by pipetting 500 µL of NexIon Daily Tuning Stock Solution (Section 8.8.2.) into a 50-mL digestion vessel filled with approximately 20 mL of reagent water and 1.0 mL of concentrated nitric acid. Dilute to 50 mL with reagent water and mix well.

8.10. Dual Detector Solution is for normalization between the pulse and analog stages of the detector. Fill a 1-L nalgene bottle with approximately 400 mL of reagent water. Add 20 mL of concentrated HNO_3. Pipette the appropriate single element standards into the bottle and dilute to 1 L with reagent water. Refer to **CX-0009** for the current preparation.

8.11. Internal Standard Solution of Sc, In, Ge, and Ir. Prepare by pipetting the elements at listed volumes into a 4-liter container with approximately 1000 mL of reagent water and 80 mL of nitric acid. Dilute to 4 liters with reagent water. Refer to **CX-0017** for the current preparation.

Note: Internal Standard Solution is added inline through a mixing block, T fitting or equivalent.

8.12. Rinse: containing 4.0% (v/v) hydrochloric acid and 2.0% (v/v) nitric acid and 2 mg/L of Au. Volumes may be made as necessary at a ratio of 80.0 mL of nitric, 160.0 mL of HCL and 0.4 mL of 10,000 mg/L Au per 4000 mL of reagent water.

8.13. Carrier Solution:

8.13.1. For SW 3010/3050: Water containing 2.0% (v/v) nitric acid. Volumes may be made as necessary at a ratio of 20.0 mL nitric acid per 1000 mL of reagent water.

8.14. Stock Calibration Standard Solutions: While this SOP gives directions for making specific concentrations, the analyst may, at times, be directed to prepare alternate concentrations for "fit of purpose." Standards may be prepared from single element solutions or a custom blended solution. All solutions for each stock standard are pippetted into a 100 mL Class A Teflon volumetric flask with about 30 mL of reagent water and 5 mL nitric acid. After all elements for each solution have been added, fill the flask to the mark with reagent water. Refer to **CX-0008** for the current preparations for 6020 Cal 1-4. Refer to **CX-0022** for the current preparation of Hg Cal.

8.15. Stock QCS (Quality Control Sample) solution: The QCS stock must be from a source independent of that used for calibration. The concentrations of the analytes in the QCS are prepared at or near the mid-point of the calibration range and at a concentration not used for calibration. All solutions for each stock standard are pippetted into a 100 mL Class A Teflon volumetric flask with about 30 mL of reagent water and 5 mL nitric acid. After all elements for each solution have been added, fill the flask to the mark with reagent water. Refer to **CX-0008** for the current preparations for 6020 QCS 1-4. Refer to **CX-0022** for the current preparation of Hg QCS.

8.16. Post Digestion Spike (BND): Pipette 50 µL of QCS (1-4) + Hg QCS Standard (Section 8.15) into a 14 mL test tube, match the analytical dilution and pipette enough diluent for a final volume of 5 mL. Mix.

8.17. Interference Check Solution Stocks

8.17.1 Interference Check Solution A Stock (ICSA 1): 10,000 µg/mL of Cl^-; 2000 µg/mL of C; 1000 µg/mL each of Al, Ca, Fe, K, Mg, Na, P, and S; and 20 µg/mL each of Mo and Ti. **Note**: Inorganic Ventures Cat #6020ICS-0A (or equivalent).

8.18 ICSA (QC STD 7): Prepare by adding acids at volumes noted below to a 50-mL digestion vessel containing about 30 mL of reagent water. Add 1 mL of ICSA 1 Stock (Section 8.17.1) and dilute to 50 mL with reagent water. Prepare this solution as needed.

8.18.1 For soil/water matrices (SW 3050/3010): 1.0 mL HNO_3 and 80 µL of HCl.

8.19 Working calibration scheme: Prepare fresh calibration standards daily in 100 mL digestion vessels. Prepare the calibration standards according to the calibration setup table (Attachment B). Be sure to fill the vessel with about 20 mL of reagent water. Then add the acids followed by the appropriate spikes and/or dilutions found in Attachment B.

8.20 Diluent:

8.20.1 Standard Water and Soil Matrices (SW 3010 / 3050): 1.25% (v/v) nitric acid: Fill a 1-L container with approximately 500 mL of reagent water. Add 12.5 mL of concentrated nitric acid to the container and dilute to the 1-L mark with reagent water.

8.20.2 HCl Diluent: 2% nitric acid, 0.2% hydrochloric acid: This diluent should be used for dilutions equal to or greater than 25X, including the standard dilution for 3010 TCLP.

Fill a 1-L container with approximately 500 mL of reagent water. Add 20 mL of concentrated nitric acid and 2 mL of concentrated hydrochloric acid. Dilute to the 1-L mark with reagent water.

9.0 DIGESTION:

Refer to SOPs 345 and 361 (Digestion of Aqueous Samples (including TCLP leachates), and Soil Digests respectively) for sample preparation.

10.0 CALIBRATION:

10.1. The instrument must be calibrated before analysis of any samples with a blank and five calibration standards. If less than five calibration standards are used, refer to the SGS SOP 500 for the criteria used in dropping calibration points. **Note:** Perkin-Elmer's software documentation refers to "blank subtraction;" however, this is actually a zeroing of the instrument to the blank response before calibration is initiated. No blank subtraction is performed following calibration. The manufacturer describes the calibration technique as "force through zero." This technique has been shown to be linear at the blank response.

10.2. The concentrations of the standards used should be entered into the calibration page of the analytical method in the ICP/MS software according to the values of the standards prepared in Section 8.19.

10.3. The first standard run should be the lowest level standard containing analyte and be equal to or less than the LOQ, followed by standards of increasing concentration in order to minimize cross-contamination and carryover.

10.4. Software will label the calibration as follows: Blank, Standard 1, Standard 2, Standard 3, Standard 4, and Standard 5.

10.5. The R-value (linear regression correlation coefficient) for each element must be greater than or equal to 0.998.

10.6. The average of three replicates for all standards, QC, and samples will be used to determine concentrations.

11.0 ANALYSIS:

11.1. Initiate the plasma and allow a warm-up of at least 30-60 minutes. The tuning procedures may be carried out during warm-up. (When turning on Agilent 7800 ICP-MS, transfer the carrier probe to the Start-up configuration solution right away. The instrument will automatically run its start-up configuration process once the required plasma temperature is met.)

11.2. Before calibration and analysis of samples, the instrument must undergo a series of performance checks to ensure that the instrument is operating properly.

11.2.1. For the NexIon 300D:

11.2.1.1. While using the Dual Detector Solution (Section 8.10), select the "Dual Detector Calibration" tab, right click and select "Quick Optimize". When calibration is complete, open the interactive window, review the graph and print it to archive with the tune package. Run the dual detector calibration twice. It is a good practice to do the dual detector calibration every day, but it only needs to be done once a week or whenever detector voltages are changed.

11.2.1.2. Switch to the Daily Tune Solution (Section 8.9) after rinsing the lines.

11.2.1.3. Torch Alignment: Select "Torch Alignment," right click and select "Quick Optimize". There is no printout.

11.2.1.4. Mass Calibration and Resolution: Select "Mass Calibration and Resolution," right click and select "Quick Optimize". The software will automatically check the limits, make adjustments and rerun if there is a failure. After each run the results will print out.

11.2.1.4.1. The measured mass for each analyte must be +/- .05 AMU of the exact mass.

11.2.1.4.2. The measured peak width must be 0.7 amu, +/- 0.1 amu at 10% peak height.

11.2.1.5. Autolens STD/DRC: Select "AutoLens STD/DRC" right click and select "Quick Optimize". Print the graph to archive with the tune package and label it as "STD/DRC."

11.2.1.6. KED Mode Autolens: Select "KED Mode Autolens" right click and select "Quick Optimize". Print the graph to archive with the tune package and label it as "KED." Only needed if collision gasses are used.

11.2.1.7. Daily Performance: Select "Daily Performance Check," right click and select "Quick Optimize". The software will automatically check the limits except for the RSDs,

make adjustments and rerun if there is a failure. After each run the results will print out.

11.2.1.7.1. The RSDs for the five replicates of Be, Mg, In and U need to be less than or equal to 5%.

11.2.1.7.2. Monitor daily performance measures for Be sensitivity, Mg sensitivity, In sensitivity background, U sensitivity, % double charged and % oxide levels.

Background at mass 220	≤ 5 cps
Ce++	≤ 3%
CeO	≤ 2.5%
Be intensity	> 2,000 CPS
Mg intensity	> 15,000 CPS
In intensity	> 40,000 CPS
U intensity	> 30,000 CPS

11.2.1.8. After tuning, aspirate rinse for about 5 minutes before beginning the calibration to avoid carry-over contamination.

NOTE: These performance requirements must be achieved before any analysis is performed. Oxides and double charged levels can be adjusted by slightly varying the nebulizer flow rate.

**The Daily Performance Report must be printed and submitted with each analytical batch.

11.2.2. For Agilent 7800-ICP-MS:

11.2.2.1. Open ICP-MS Instrument Control (Online ICP-MS Mass Hunter) software. Click the "Batch" dropdown and select 6020 NO GAS.icpms.template. Rename the folder using the date (MM.DD.YYYY.P7). Click the "Batch" icon > Acq Method >Tune and select Start Auto Tune. Monitor the sensitivity and Resolution/Axis of Be, Mg, Co, In, and Pb.

Analyte and Mass	Sensitivity RSD%	Axis	W-5%
Be, 9	<5.000%	8.90-9.10	<0.9000%
Mg, 24	<5.000%	23.90-24.10	<0.9000%
Co, 59	<5.000%	58.90-59.10	<0.9000%
In, 115	<5.000%	114.90-115.10	<0.9000%
Pb, 208	<5.000%	207-90-208.10	<0.9000%

11.3. Set-up the analytical run.

11.3.1. For NexION: Open the "6020A" workspace

11.3.1.1. Calibration standards and QC solutions prepared in Section 8.19 are to be placed into their assigned autosampler (A/S) positions.

11.3.1.2. Edit the Sample window under the batch analysis to update the schedule with new sample information and autosampler locations. The default page will have rinse times

and pump speeds already entered. Ensure that the proper dilution factor is entered for each sample. Save the sample window with the date of analysis.

11.3.1.3. Typical Sample Dilutions: Dilute all samples and digested QC with the appropriate amount of diluent in the 14 mL disposable test tubes. Refer to Section 8.20 for the diluent preparations.

3010 (5X): 1.0 mL of sample into 4.0 mL of diluent for a final volume of 5 mL.

3050B (10X): 0.5 mL of sample into 4.5 mL of diluent for a final volume of 5 mL.

3010T (25X): 200 µL of sample into 4.8 mL of HCl Diluent for a final volume of 5 mL.

Note: The LB for TCLP extraction is diluted by prep. Check to ensure the final dilution of the LB matches that of the most concentrated sample in the batch.

11.3.1.4. The choice can be made to automate CCV/CB rates at every 10 samples analyzed or specify CCV/CB analysis at particular times during the run (not to exceed an interval of 10 samples) within the method under the QC tab / QC Frequency sub-tab. The default is automated at 10 samples.

11.3.1.5. Just prior to analysis, press the Restart QC button (this resets QC to run every ten samples) under the 'Analysis' drop-down menu. Then select the samples to be analyzed under the Batch tab in the sample window by highlighting the rows to be analyzed. Make sure that row 1 containing the calibration command is highlighted or that calibration is performed prior to the analysis of any samples.

11.3.1.6. Select "Analyze Batch".

11.3.1.7. All samples containing target analytes above linear range shall be diluted and re-analyzed.

11.3.2. For Agilent 7800: Click "Batch".

11.3.2.1. Calibration standards and QC solutions prepared in Section 8.19 are to be placed into their assigned autosampler (A/S) positions.

11.3.2.2. Check "Calibration Standards" and "Check Samples 1" to make sure there are no "Skip" boxes checked. Click "Add to Queue". (This step is meant to run the calibration and instrument QC first before entering sample information.) A new window, Online ICP-MS Data Analysis, will pop out once the calibration begins.

11.3.2.3. Typical Sample Dilutions: Dilute all samples and digested QC with the appropriate amount of diluent in the 14 mL disposable test tubes. Refer to Section 8.20 for the diluent preparations.

3010 (5X): 1.0 mL of sample into 4.0 mL of diluent for a final volume of 5 mL.

3050B (10X): 0.5 mL of sample into 4.5 mL of diluent for a final volume of 5 mL.

3010T (25X): 200 µL of sample into 4.8 mL of HCl Diluent for a final volume of 5 mL.

Note: The LB for TCLP extraction is diluted by prep. Check to ensure the final dilution of the LB matches that of the most concentrated sample in the batch.

11.3.2.4. Once the calibration is done and instrument QCs meet the criteria, go back to "Online ICP-MS MassHunter", click "Batch" icon and "Start Editing Mode". Click "Unknown Samples" and enter the necessary sample information. If prepFAST dilution is utilized, enter the appropriate dilution under "prepFAST Dilution". The "Total Dil." column will automatically be filled out. If bench dilution is done, the "prepFAST Dilution" and "Total Dil." columns will remain as 1.00. Add the bench dilution after the sample name (for example, "HSN X10"). Go to "Check Samples 2" and uncheck the "Skip" boxes for CCV and CB. Save the batch information and Click "Add to Queue" to start the analysis.

11.3.2.5. All samples containing target analytes above linear range shall be diluted and re-analyzed.

11.4. Each analysis consists of an injection followed by a rinse of the sample probe. Then, after acquisition of data, the sample introduction system is rinsed a second time before the next injection.

11.4.1. To ensure mercury is cleared from the system, the sample probe moves to the 1st rinse station where it is rinsed in a rinse solution containing 1 mg/L gold, then moves to rinse station 2 where it is rinsed a 2nd time with the same rinse solution. Following data acquisition, the valve switches back to the load position and the probe and sample loop are rinsed with the gold solution. After the probe and sample loop rinse, the valve switches to the inject position so that the nebulizer and spray chamber can be rinsed with the gold solution as well.

11.4.2. Analysis of mercury by method 6020B is accomplished by the addition of gold to both the rinse (Method 6020B 7.6.3), and the calibration standards. Gold acts as an oxidizing agent for mercury, preventing volatilization of elemental mercury, thus retaining it in solution and preventing plating out in the sample introduction system.

11.4.3. Analysis of mercury by ICP-MS is possible with the SC-FAST sample introduction system even without adding gold to the rinse. Addition of gold to the rinse improves on ESI's already superb mercury washout times. Testing done by ESI has shown complete washout times for 1 ppb of Hg was 23 seconds and 5 ppb of Hg was 30 seconds even without using gold in the rinse.

11.5. At the end of the analytical run, print out the calibration summary and instrument run log.

11.5.1. For NexIon: Open the "report option window."

11.5.1.1. Choose "CAL REPORT.rop" file. Print the report view.

11.5.1.2. Open "RUN-COVER.rop" file. Print the report view.

11.5.2. For Agilent 7800: Go back to "Online ICP-MS Data Analysis" window.

11.5.2.1. Click the "Report" dropdown and choose "Quick Batch Report". A new window, containing the instrument run log, will appear. Click "Print" to save a PDF copy.

11.5.2.2. Right Click any graph in the "Calibration Curve" section. Choose "Print Calibration" and "Simple". A new window, containing the calibration summary, will appear. Click "Print" to save a PDF copy.

12.0 QUALITY CONTROL:

12.1. Initial Demonstration of Laboratory Performance - The following items must be completed before the analysis of any samples is performed by each laboratory using this method.

12.1.1. Instrument Detection Limits (IDLs) - IDLs must be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and or/ at a frequency designated by the project. IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. This procedure states that the IDLs can be estimated by calculating the sum of the mean of the blank results and three times the standard deviations of 10 replicate analyses of the reagent blank solution (report mean as 0 if the result is negative). Each measurement must be performed as though it was a separate sample (i.e., with rinsing in between). The IDL must be less than or equal to the DL. Refer to SOP 500 for further guidance.

12.1.2. Detection Limit Study (DLs) - In order to determine the DL in a matrix, the analytes should be spiked into the matrix of interest (e.g., ground water matrix, or other sample preparation procedure matrix) at a level that is three to five times the estimated DL. The spiked matrix is then carried through the entire sample preparation procedure. Each replicate result must be calculated the same as if it were a final client sample result. The DL is calculated by multiplying the standard deviation obtained from a minimum of seven replicates by the one-sided 99% confidence level t-statistic. See Section 17 for specific conditions for performing DL studies. Refer to SOP 116 for further guidance.

12.2. Linear Dynamic Ranges

12.2.1. The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover with 10% of its true value, and if successful, establishes the linear range. The Linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.

12.2.2. Calibrate the instrument as described in Sections 10 and 11 and run a series of standards that increase in concentration until the measured value deviates 10% from the expected value. The upper limit is determined to be the value of the highest standard that is within 10% of the expected value.

Note: The Linear Dynamic Range should be re-evaluated on the following basis:

- Whenever new PA tube is installed in the RF generator
- Upon any significant change to the instrument (i.e. new detector or alternate sample introduction system)

Refer to SOP 500 for further guidance.

12.3. Analyses of Quality Control (QC) samples – QC samples are analyzed at periodic intervals to evaluate method and instrument performance. Each QC type is described below along with relevant evaluation criteria (see Attachment C for method nomenclature descriptions). All analyses for analytes of interest must be bracketed by acceptable QC to avoid performing corrective action

12.3.1. Quality Control Sample – *Note: SGS terms this the **QCS** while Method 6020B uses another identification for this second source standard.* Analysis of the QCS occurs immediately following the ICV to verify the accuracy of the calibration curve. The results of the target analytes must be within ±10% of the true value. If the 10% criteria is not met, the QCS may be analyzed a second time. If the QCS is still outside of acceptance criteria, the cause of the error must be determined, and a recalibration must be performed.

12.3.2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Analysis (standards are from the same source as the calibration curve):

12.3.2.1. The ICV is required at the beginning of the analytical sequence.

12.3.2.2. The CCV is analyzed after every 10 samples and at the end of the analytical sequence.

12.3.2.3. The limits are ±10% of the true value.

12.3.2.4. If the ICV criteria is not met, the ICV may be analyzed a second time. If the ICV is still outside of acceptance criteria, the cause of the error must be determined and a recalibration must be performed

12.3.2.5. If the CCV criteria is not met:

12.3.2.5.1. For non-DOD samples: the CCV may be analyzed a second time. If the CCV is still outside of acceptance criteria, the cause of the error must be determined and a recalibration must be performed. Samples run between CCV's not within limits must be reanalyzed. If the rerun meets QC criteria, then the run sequence may be continued. Samples following the passing CCV are valid.

12.3.2.5.2. For DOD samples: The analyst may immediately (within 1 hr of the failing CCV and before any other samples have acquired) rerun 2 successive CCVs. If both meet QC criteria for all analytes of interest, then the samples already analyzed may be reported and the run sequence may be continued. Samples following the two passing CCVs are also valid.

12.3.3. Continuing Calibration Blank (CB) analysis. The CB is required at the beginning of the analytical sequence, after every 10 samples and at the end of the analytical sequence.

12.3.3.1. The limits are < LOQ for each element.
Note: For DoD clients, the limits are < LOD (< ½ the LOQ).

12.3.3.2. If the CB criteria are not met, the CB may be analyzed a second time. If the CB is still outside acceptance criteria, the cause of the error must be determined and a recalibration must be performed. Samples run between CB's not within criteria must be evaluated and possibly reanalyzed. If the analyte concentration is less than the LOQ or 5X the level of that seen in the CB, the analyte data can be reported with an appropriate comment. All samples outside of these criteria must be reanalyzed. If failure occurs consistently, the IDL and DL must be re-evaluated.

12.3.4. Low Level Initial Quantitation Checks (LLIQC 1,2, and 3) are spiked at or below the LOQ. These are readbacks of calibration points 1,2, and 3 and analyzed at the beginning of the analytical

sequence. The LLIQC limits are ± 20% for DOD and ± 30% for non-DOD. If an analyte is greater than +20% for DOD or +30% for non-DOD but the analyte is non-detect (<LOQ) in the sample, it can be reported with a comment on the LLIQC.

12.3.5. ICSA (QC Std 6) solution - Required at the beginning of analytical run or every 12 hours, whichever is more frequent. Per client request, ICSA may be analyzed at the end of the run. The Internal Standard recoveries in the ICSA solution must be in the range of 70-130%.

12.3.5.1. ICSA criteria for DoD clients evaluates the absolute values of all non-spiked analytes and limits them to < LOD (unless they are a verified trace impurity from one of the spiked analytes). Corrective action: Flag any analytes that exceed limits. This criterion is in exceedance of the method.

12.3.6. Internal Standards: Intensities must be monitored in all solutions. The ICP/MS software will report the % recovery for each internal standard being used.

12.3.6.1. The RSD of the internal standards in the Calibration Standards must be within 10%. If the internal standards do not meet these criteria, re-run once. If they are still outside acceptance criteria, recalibrate.

12.3.6.2. Intensities of the internal standards in CCV and CB solutions must be within ± 30% of the levels in the original calibration blank. If the internal standards do not meet criteria, terminate the analysis, correct the problem, re-calibrate, and reanalyze all affected samples.

12.3.6.3. Intensities of internal standards in samples must be within 70-130% of that in the original calibration blank. If the internal standards do not meet criteria, dilute the sample five-fold and reanalyze. This procedure is followed until the internal standard intensities fall within the prescribed window.

12.3.7. Method Blank (MB): the MB is analyte-free reagent water that has been processed and analyzed identically to an unknown sample to assess systematic contamination. One MB will be prepared and analyzed for each sample batch of 20 samples or less. Results for analytes of interest in the MB must be less than half the LOQ OR are less than project specific requirement. Samples containing detectable amounts of contamination may require re-digestion. If the measured sample concentration is greater than 5 times the MB contamination or less than the LOQ, the samples will not require re-digestion, but **detect DoD data must be flagged with a 'B' in LIMS**. All other samples will require re-digestion and reanalysis.

12.3.8. Method Blank Filter (MBF): The MBF is analyte-free reagent water filtered the same way as regular TCLP glycol samples. It is evaluated in the same manner as an MB.

12.3.9. Leachate Blank (LB): The LB is the leachate used for TCLP samples. The LB is brought through the TCLP process as if it were a sample. It is evaluated in the same manner as an MB.

12.3.10. Laboratory Control Sample (LCS): the LCS is reagent water that has been spiked with known concentrations of analyte that is processed and analyzed identically to an unknown sample to assess system accuracy. One LCS will be prepared and analyzed for each sample batch of 20 samples or less. See Attachment D for the acceptance criteria for the LCS. If an analyte of interest has a %recovery that is greater than the acceptance criteria and the associated sample results for that analyte are below the LOQ, then those samples may be reported with appropriate

qualifiers. If an analyte of interest has a %recovery less than the acceptance criteria, then those samples affected will require re-digestion.

12.3.11. Matrix Spike (MS) and Matrix Spike Duplicate (MSD): the MS/MSD is prepared and analyzed to determine the accuracy and precision of the analytical system and are prepared for each set of 20 samples or less of a similar matrix. Samples are spiked at a level equal to that of the LCS. See Attachment D for recovery and RPD acceptance criteria. If the recovery for an analyte of interest does not meet criteria, perform a bench spike (BND). If the RPD is outside of criteria, and there is a sample DUP, evaluate the RPD for the DUP. If the DUP RPD passes, then post with comment. If the DUP RPD fails, then it is confirmed that the sample is non-homogenous. Post the data with a comment. If there is not a sample DUP and the analyte of interest is detected in the sample, the sample/MS/MSD must be re-extracted.

Note: If an LCSD was analyzed with the batch and is within RPD limits, then analytical precision is shown to be in control and all data can be posted with comment.

12.3.12. Post Digestion Spike (BND): the BND is performed only on spiked samples that do not meet MS recovery evaluation criteria. An aliquot of digested sample at standard or inflated dilution is spiked with a known concentration of analyte. Recoveries calculated on the BND for 3010/3050 should be between 75% and 125% of the expected values. The BND recoveries for 3050O (matrix 3) should be between 70-130% of expected values.

12.3.12.1. If the spike is not recovered within the acceptance limits and the LCS for that analyte is in control, then the recovery problem is judged matrix related and the sample will be diluted and re-spiked.

12.3.12.2. Analytes that do not meet acceptance limits will be commented on in the parent sample, or all associated samples in the prep batch should be run by method of standard additions (MSA).

12.3.13. Sample Duplicate (DUP): Analyze one DUP sample for every matrix in a batch of twenty samples or less. The RPD of the DUP should be ≤ 20. These limits should not be exceeded for analyte values greater than 100 times the IDL. Preferentially, the RPD requirement is fulfilled by the MS/MSD RPD. However, in some complex matrices, when the MS/MSD RPD is outside limits, the sample/sample DUP can be utilized as a separate evaluation for sample homogeneity.

12.3.14. Dilution Test (DT): A 5-fold further dilution on top of the standard dilution. If the analyte concentration in the selected sample is sufficiently high (50x above the LOQ), the DT must agree within ±20% of the original determination. If not, interference must be suspected, and the sample flagged. One dilution test must be performed for each digestion batch of twenty samples or less of each matrix.

12.4. See Attachment E for specific corrective actions. Any corrective action needed to address a QC outlier that is not listed in this SOP requires the approval of the QA Manager or Technical Director.

13.0 CALCULATIONS, REVIEW AND REPORTING:

13.1 The NexIon software performs all calculations necessary to convert raw data (ion counts/second) to concentration (µg/L). The calculated quantities are selected by choosing the desired options in the Report Options screen. The default report option for the NexIon 6020 Method is EPA6020 SGS1.rop. If the user desires, this format can be edited and saved under a new name.

13.2 All calculations performed in the software are based on the ratio of the analyte intensity (cps) to the internal standard intensity (cps). In all calculations where internal standards are used the ratio of the analyte intensity to internal standard intensity is taken before any other calculation is performed.

13.3 Quality Control Sample results may be checked using the QC Checking features in the ICP/MS software. All values entered in the default 6020 method should be checked and edited to match the true values used by the laboratory.

13.4 The recoveries for standard solutions (both digested standards and the periodic quality control standards) are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{Vo}{Ve} \times 100 = \% \text{ recovery}$$

Vo = observed value
Ve = expected value

13.5 The calculation for spike recoveries requires the subtraction of the sample contribution from the response of the spiked sample, the division of this result by the expected value of the spike, and the multiplication by 100 yields percent recovery.

$$\frac{Vo-Sc}{Ve} \times 100 = \% \text{ recovery}$$

Vo = observed value of the spiked sample
Sc = observed value of the sample
Ve = expected value of the spike

13.6 The RPD between duplicate samples is calculated as the absolute difference between the sample and the duplicate, divided by the average of the sample and the duplicate, all multiplied by 100.

$$\frac{|Sc - Dc|}{\{(Sc + Dc) / 2\}} \times 100 = \text{Duplicate Relative Percent Difference (Dup RPD)}$$

Sc = observed sample concentration
Dc = observed duplicate sample concentration

13.7 The post digestion spike (BND) recovery is calculated as the difference of the BND concentration minus the sample concentration all divided by the expected concentration of the BND. The resultant value is multiplied by 100 to give percent recovery.

$$\frac{BSc - Sc}{BSe} \times 100 = \% \text{ recovery}$$

BSc = observed value of post-digestion spike
Sc = observed value of sample
BSe = expected value of post-digestion spike

13.8 Samples that have been digested using concentration procedures must have the concentrations of the elements multiplied by the appropriate factor.

13.9 Data Archiving: The raw data files produced by the ICP/MS software are moved over to the network on a monthly basis. The archive location is: \\usfs700\ANK_Instrument_Data\METALS\ICPMS\DATA.

14.0 HEALTH AND SAFETY:

14.1 Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2 Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3 All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4 Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5 Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative, a face shield and apron must be worn.

14.6 Liquid argon represents a potential cryogenic hazard and safe handling procedures should be used when handling liquid argon tanks at all times.

14.7 Anhydrous Ammonia is an irritant and corrosive to the skin, eyes, respiratory tract and mucous membranes. Exposure to rapidly expanding gases may cause severe chemical burns and frostbite to the eyes, lungs, and skin.

14.8 The ICP/MS instruments are fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the instruments if any safety interlock is known to be disabled or malfunctioning.

14.9 Spilled samples, reagents, and water should be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills the acid should be neutralized with sodium bicarbonate solution before cleanup.

15.0 POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0 METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0 DETECTION LIMIT (DL) STUDY:

DL studies are performed when a significant change in instrument response is observed, and when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

18.0 LIMIT OF DETECTION (LOD):

The LOD is defined per SOP 116; LOD verification shall be performed quarterly according to the schedule set by the QA Office.

19.0 LIMIT OF QUANTITATION (LOQ):

LOQs may be adjusted for sample dilution or the presence of matrix interferences. LOQs are established based on experimental DL studies and comparison to program and project-specific reporting limit requirements**.** LOQ information is only provided to give the reader an idea of the reporting capabilities of the method as it is implemented at SGS. LOQs may change at the laboratory's discretion.

Element	LOQ - Oil (µg/Kg)	LOQ - Soil (µg/Kg)	LOQ - Water (µg/L)
Ag	500	500	2
Al	20000	20000	200
As	1000	1000	10
B	20000	20000	200
Ba	300	300	3
Be	100	100	1
Bi	200	200	2
Ca	50000	50000	500
Cd	200	200	2
Co	500	500	1
Cr	1000	1000	10
Cu	600	600	6
Fe	50000	50000	500
K	100000	100000	1000
Mg	50000	50000	500
Mn	200	200	2
Mo	1000	1000	5
Na	100000	100000	1000
Ni	200	200	2
P	20000	50000	500
Pb	200	200	1
Sb	1000	1000	3
Se	1000	2000	20
Si	NA	NA	NA
Sn	1000	1000	10
Sr	1000	20000	200
Ti	5000	5000	50
Tl	200	200	2
V	3000	5000	50
Zn	2500	2500	25
Hg	300	300	0.5

20.0 REFERENCES:

20.1 NexIon 300D ICP-MS Customer Training Course, 2000, Perkin-Elmer Corporation.

20.2 Agilent 7800/7900 ICP-MS Hardware Maintenance Manual, 2019, Agilent Technologies, Inc.

20.3 "Inductively Coupled Plasma-Mass Spectrometry", U.S. EPA SW-846 Method 6020B rev. 2.

21.0 ATTACHMENTS:

Attachment A: ICP-MS Isotopes
Attachment B: 6020 Calibration Setup
Attachment C: Method Nomenclature
Attachment D: Limits for LCS, MS, MSD and RPD
Attachment E: Corrective Action Table

Controlled copy distributed to Brie Hicknell with VHB on 2/5/2024.

Attachment A: ICP/MS Isotopes

The following isotopes should be monitored for ICP/MS analyses.
Determination of the primary isotope will be based on user knowledge and matrix. The tables below are to be used only as a general guideline. Interferences generally result in a biased high isotope. In most cases, an attempt should be made to report the lowest recovering isotope if there is a significant difference between isotopes.

Reportable Element Isotopes

Analyte		Preferred	Alternate	Notes
Lithium	**Li**	**7**		
Beryllium	**Be**	**9**		
Boron	**B**	**11**		
Aluminum	**Al**	**27**		
Vanadium	**V**	**51**		
Chromium	**Cr**	**52**		
Manganese	**Mn**	**55**		
Cobalt	**Co**	**59**		
Nickel	**Ni**	**60**		
Copper	**Cu**	**65**	63	Use 63 with high Ca
Zinc	**Zn**	**66**	68	
Arsenic	**As**	**75**		
Selenium	**Se**	**82**	78	
Strontium	**Sr**	**88**		
Molybdenum	**Mo**	**95**	98	Isotopes are essentially equal
Silver	**Ag**	**107**	109	Isotopes are essentially equal
Cadmium	**Cd**	**114**	111	Use 111 with high Mo
Tin	**Sn**	**118**		
Antimony	**Sb**	**121**	123	Isotopes are essentially equal
Barium	**Ba**	**135**	137	Isotopes are essentially equal
Mercury	**Hg**	**202**	201	If W is present; use lowest recovering isotope
Thallium	**Tl**	**203**	205	Isotopes are essentially equal
Lead	**Pb**	**206, 207, 208***		The reported value for lead is the sum of the three isotopes.
Bismuth	**Bi**	**209**		
Uranium	**U**	**238**		
Sodium	**Na**	**23**		
Magnesium	**Mg**	**24**	25	
Silicon	**Si**	**28**	29	
Phosphorus	**P**	**31**		
Potassium	**K**	**39**		
Calcium	**Ca**	**43**		
Titanium	**Ti**	**47**		
Iron	**Fe**	**54**	57	

*for Agilent 7800 ICP-MS (P7) data, the isotope sum is displayed as 208 Pb.

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Element Isotopes for Monitoring Only

Analyte		Isotope	Notes
Tungsten	**W**	**184, 186**	
Chlorine	**Cl**	**37**	
Carbon	**C**	**13**	
Scandium	**Sc**	**45**	Internal Standard
Chromium	**Cr**	**53**	
Nickel	**Ni**	**58, 62**	
Zinc	**Zn**	**67**	
Germanium	**Ge**	**74**	Internal Standard
Selenium	**Se**	**77**	
Krypton	**Kr**	**84**	
Molybdenum	**Mo**	**97**	
Cadmium	**Cd**	**106, 108**	
Gold	**Au**	**197**	
Indium	**In**	**115**	Internal Standard
Iridium	**Ir**	**193**	Internal Standard
Lead	**Pb**	**204**	
Titanium	**Ti**	**48**	
Iron	**Fe**	**56**	

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Attachment B: 6020 Calibration Setup

NexIon 3010/3050 - 6020 Calibration					
A/S		**6020 (SW 3010/3050)**			
		Standards	**Acids/Au**	**Final Vol**	
1	Blank/CB		1.000 mL HNO3 0.100 mL dHCl	50 mL	QC Std 8
2	Std 1	10 mL of Std 2	0.800 mL HNO3 0.080 mL dHCl	50 mL	
3	Std 2	5 mL of Std 3	0.900 mL HNO3 0.090 mL dHCl	50 mL	
4	Std 3	5 mL of Std 4	0.900 mL HNO3 0.090 mL dHCl	50 mL	
5	Std 4	10 mL of Std 5	0.800 mL HNO3 0.080 mL dHCl	50 mL	
6	Std 5	0.250 mL Cal (1 – 4) + Hg Cal	1.000 mL HNO3 0.100 mL dHCl	50 mL	
7	ICV/CCV	0.100 mL Cal (1 – 4) + Hg Cal	1.000 mL HNO3 0.100 mL dHCl	50 mL	QC Std 1/5
8	QCS	0.5 mL QCS (1 – 4) + Hg QCS	1.000 mL HNO3 0.100 mL dHCl	50 mL	QC Std 2

Agilent 7800 ICP-MS				
A/S		**6020 (SW 3010/3050)**		
		Standards	**Acids/Au**	**Final Vol**
1	Blank/CB		2.000 mL HNO3 0.200 mL dHCl	100 mL
2	Std 3	2 mL of Std 5	2.000 mL HNO3 0.200 mL dHCl	100 mL
3	Std 5	0.500 mL Cal (1 – 4) + Hg Cal	2.000 mL HNO3 0.200 mL dHCl	100 mL
7	ICV/CCV	0.200 mL Cal (1 – 4) + Hg Cal	2.000 mL HNO3 0.200 mL dHCl	100 mL
9	QCS	1.00 mL QCS (1 – 4) + Hg QCS	2.000 mL HNO3 0.200 mL dHCl	100 mL

*For Agilent 7800 ICP-MS, the prepFAST autosampler performs serial dilutions for calibration.

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Attachment C: METHOD NOMENCLATURE

METHOD SYMBOL	SGS SYMBOL	METHOD NAME	SGS NAME
CCV	ICV, CCV	Calibration Verification Solution	Continuing Calibration Verification
DUP	DUP	Duplicates	Duplicates
LCS	LCS	Laboratory Control Sample	Laboratory Control Sample
CCB	CB	Calibration Blank	Calibration Blank
MB	MB	Preparation Blank	Method Blank
DT	DT	Dilution Test	Dilution Test
LLQC	LLIQCS	Low Level Quantitation Check	Low Level Initial Quantitation Check
BND	BND	Bench Spike	Bench Spike
ICV	QCS	Initial Calibration Verification (second source)	Quality Control Sample (second source)

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Attachment D: ACCURACY LIMITS FOR LCS, MS, and MSD

	3010 Water/ 3010 TCLP		3050 Soils		3050O Oils
Analyte	Lower (%)	Upper (%)	Lower (%)	Upper (%)	Range (%)
Aluminum (Al)	84	117	78	124	
Antimony (Sb)	85	117	72	124	70-130
Arsenic (As)	84	116	82	118	70-130
Barium (Ba)	86	114	86	116	70-130
Beryllium (Be)	83	121	80	120	70-130
Bismuth (Bi)	-	-	80	120	
Boron (B)	73	130	74	128	
Cadmium (Cd)	87	115	84	116	70-130
Calcium (Ca)	87	118	86	118	
Chromium (Cr)	85	116	83	119	70-130
Cobalt (Co)	86	115	84	115	
Copper (Cu)	85	118	84	119	70-130
Iron (Fe)	87	118	81	124	
Lead (Pb)	88	115	84	118	70-130
Lithium (Li)	78	126	75	120	
Magnesium (Mg)	83	118	80	123	
Manganese (Mn)	87	115	85	116	
Mercury (Hg)	70	124	74	126	
Molybdenum (Mo)	83	115	83	114	70-130
Nickel (Ni)	85	117	84	119	70-130
Phosphorus (P)	80	120	80	120	
Potassium (K)	87	115	85	119	
Selenium (Se)	80	120	80	119	70-130
Silver (Ag)	85	116	83	118	70-130
Sodium (Na)	85	117	79	125	
Strontium (Sr)	82	118	75	129	
Thallium (Tl)	82	116	83	118	70-130
Thorium (Th)	87	121	81	116	
Tin (Sn)	86	115	82	121	
Titanium (Ti)	83	115	83	117	
Uranium (U)	87	120	83	120	
Vanadium (V)	86	115	82	116	70-130
Zinc (Zn)	83	119	82	119	70-130

Attachment E: CORRECTIVE ACTION TABLE

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020B	Tune	Daily.	See Section 11.2.1. and 11.2.2.	1. Find source of problem and correct. 2. Repeat Tune.
SW6020B	Daily Instrument Performance	Daily.	See Sections 11.2.1.7.1 and 11.2.1.7.2	1. Find source of problem and correct. 2. Repeat Daily Instrument Performance Check.
SW6020B	Calibration	Daily before each method.	Correlation coefficient ≥ 0.998.	1. Find source of problem and correct. 2. Repeat calibration
SW6020B	Calibration Blank (CB)	After calibration & 1 per 10 sample readings & at the end.	Concentration < LOQ **For DoD:** < ½ LOQ or <1/10th the amount measured in any sample	1. Initial CB: Correct problem and repeat once. If that fails, recalibrate. 2. CB: For DOD: All samples following the last acceptable calibration blank must be reanalyzed. For non-DOD. Repeat analysis once. 3. Fix problem 4. Recalibrate. CB failures due to carryover may not require a new calibration. 5. Samples that are non-detect may be reported. Comment on the CB. For level 2 DOD clients, report on the sample. 6. Samples that are >5x the CB may be reported. Comment on the CB. For level 2 DOD clients, report on the sample.
SW6020B	Initial Calibration Verification (ICV)	After Calibration	±10%	1. Repeat analysis once. 2. Fix problem. 3. Recalibrate.
SW6020B	Continuous Calibration Verification (CCV)	1 per 10 sample readings & at end.	±10%	1. Refer to SOP 500 Attachment D
SW6020B	Quality Control Std. (QCS)	After Calibration	±10%	1. Repeat analysis once. 2. Fix problem. 3. Recalibrate.
SW6020B	Lower Limit Initial Quantitation Check (LLIQCS)	After Calibration	±20% for DOD ±30% for non-DOD	1. Repeat analysis once. 2. Fix problem. Recalibrate 3. If LLIQCS>120% (DOD) or 130% (non DOD), samples that are less than the LOQ may be reported with a comment on the LLIQCS.

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Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020B	Method Blank (MB)/ Method Blank Filter (MBF)	1 per batch of 20 samples or less	Concentration < ½ LOQ OR project specific. **For DoD:** < ½ LOQ	1. Repeat analysis once. 2. Evaluate samples; if N.D. or >5X MB, OK to report. Apply B flag for DoD if detect results reported without compliant blank. 3. Re-digest and reanalyze samples with concentration of contaminant > LOQ & <5X MB value.
SW6020B	Leachate Blank (LB)	1 per 3010 TCLP batch of 20 samples or less	Concentration < LOQ For DoD: < LOD	1. Same as for the MB. See above.
SW6020B	Laboratory Control Sample (LCS)	1 per batch of 20 or less	See Attachment C	1. Repeat analysis once. 2. Evaluate samples; if LCS is high but samples are ND for that analyte, ok to report, but comment on sample. Re-digest and reanalyze samples with concentration of contaminant > LOQ. 3. If LCS is low, re-digest and reanalyze samples.
SW6020B	Matrix Spike (MS) /Matrix Spike Duplicate (MSD)	1 per batch of 20 or less.	See Attachment C for recovery criteria. RPD criteria for: 3010/3050 is 20 3050O is 30	1. If recovery is outside of control, perform a bench spike (BND) 2. If RPD is outside of control, and there is a sample DUP: 2.1. If DUP RPD is outside of control, comment on sample homogeneity. 2.2. If DUP RPD is in control, post with comment. 3. If RPD is outside of control, and there is not a sample DUP: 3.1. Evaluate parent sample. If the target analyte is ND, report with comment. 3.2. If target analyte is detected, re-digest and reanalyze. 3.3. If still outside of limits, flag sample as non-homogeneous.
SW6020B	Post Digest Spike (BND)	When required.	For 3010/3050: 75-125% For 3050O: 70-130%	1. Dilute and re-spike. 2. Run all associated samples by MSA. 3. Flag data as suspected matrix interference. Or, J-flag affected analytes in parent sample

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Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020B	Duplicate (DUP)	Once for every sample matrix in a batch of ≤ 20 samples. Can be derived from MS/MSD.	≤ 20 RPD for analytes greater than 100 times the IDL.	1. If RPD outside of control, evaluate the MS/MSD RPD. 2. If it is out as well, flag sample as non-homogenous.
SW6020B	Dilution Test (DT)	1 per batch of 20 or less.	1. Evaluate analytes greater than 50X LOQ in sample. 2. The 5X dilution must agree within ±20% of original.	Flag data as suspected matrix interference.
SW6020B	ICSA	After calibration, or every 12 hours, whichever is more frequent.	Internal Standards: 70 – 130% (For DoD: Absolute values of non-spiked analytes < LOD)	1. If internal standards fail to meet criteria, recalibrate. 2. Flag any analytes that fail DoD criteria
SW6020B	Internal Standards	In Calibration Standards	RSD is established at 10%.	1. Rerun once 2. Recalibrate
		In all solutions.	In CCV/CB: ±30% original cal blank intensity.	1. Terminate analysis. 2. Correct problem. 3. Re-calibrate.
		In all solutions.	In Samples: 70-130% original calibration blank intensity.	1. Dilute the sample fivefold and reanalyze. 2. Continue until criteria is met.

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S.O.P. Title: Digestion of Aqueous Matrices for ICP-MS Metals Analyses	**Revision Date:** April 2022
Method No: SW846 3010A	**SOP No:** 345r12
Page: 1 of 10	**Supersedes:** 345r11

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	4/12/22	Tamara Rentz	4/12/22
Stephen C. Ede	4/17/23	Tamara Rentz	4/17/23

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I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Tamara Rentz
Tamara Rentz 2022.04.12 13:45:55 -08'00'

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Summary of Changes from Previous Revision:

- Incorporated Addendum 1 into section 6.2.2.
- Edited section 9.4 to mention electronic laboratory notebook instead of prep logbook.
- Basic formatting, spelling and grammar corrections made throughout.

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1.0. OBJECTIVE:

To provide a procedure for processing raw aqueous samples and solid samples that have been extracted by TCLP (Toxicity Characteristic Leaching Procedure) extraction into a form amenable to analysis for total recoverable metals and/or dissolved metals.

2.0. SCOPE AND APPLICATION:

The 3010A method is applicable to the preparation of aqueous samples using an acid digestion procedure for analysis by Inductively Coupled Plasma - Mass Spectroscopy (6020B).

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. Sample volume has been reduced to one-quarter of that specified in the method. Acids have been reduced proportionally as has the volume the sample is reduced to during digestion.

3.2. The 3010 method specifies the addition of 10 mL 1:1 HCl (or 5 mL of concentrated acid) per 100 mL final volume just prior to the last reflux step. To allow digestion for analysis by ICP-MS (and to accommodate a reduced final volume), 0.3 mL of concentrated HCl is used instead.

3.3. The 3010 method has a second refluxing step at the end after adding HCl. A procedural change was made to add the HCl at the beginning of the digestion process and therefore, the second refluxing step is removed.

4.0. RESPONSIBILITIES:

4.1. The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2. The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3. It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4. This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5. A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF is a controlled copy of the SOP and is stored on the network.

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All staff has “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. The acid digestion of samples should be performed under a fume hood to prevent contamination. These fume hoods are in the Metals Preparation Clean Room, which is segregated from the rest of the laboratory. Fume hoods will be cleaned daily.

5.2. Samples requiring the analysis of the analytes Boron and Silicon should be contained in a plastic, Teflon, or quartz apparatus prior to analysis.

5.3. Samples containing large amounts of suspended or dissolved solids may have the tendency to splatter, causing cross contamination between samples. These samples should be monitored during the digestion.

6.0. SAMPLE HANDLING:

6.1. Samples scheduled for analysis of total recoverable metals must be preserved with nitric acid to a pH of less than 2.

6.1.1. Samples should be field preserved, using nitric acid, to a pH < 2.

6.1.2. If samples are not field preserved, they must be preserved upon receipt at the laboratory. Following acidification, the sample should be mixed, tagged with a "pH adjusted: wait 24 hours" sticker and held for 24 hours prior to digestion. The pH of samples preserved in the lab should be tested by the analyst immediately prior to taking an aliquot. The time, date, and initials from both the initial preservation and the 24 hour pH check will be recorded in the Metals pH Verification logbook. If the sample has not equilibrated to pH < 2, then this step must be repeated.

6.2. Samples scheduled for analysis of dissolved metals:

6.2.1. Samples must be filtered through a 0.45 µm membrane filter (purity tested) and then preserved with nitric acid (pH < 2). If the filtering and preserving are not done in the field, both must be completed immediately upon receipt in the lab. Preparation should be done immediately. If this is not feasible, the lab filtered samples will be preserved with nitric acid (pH < 2) with no minimum waiting time.

6.2.2. Do not shake samples prior to filtering. The syringe should NOT be immersed into the sample in order to reduce contamination. Submerge the tip of the syringe only and waste the first aliquot (10 mL) to remove particulates from the filter and syringe. For the method blank (MB) and laboratory control spike (LCS), filter 25 mL of Milli-Q DI water into the digestion vessel. Then filter 25 mL of the sample chosen for QC into the vessels for the parent sample (PS) as well as the matrix spike (MS) and the matrix spike duplicate (MSD). Add 0.500 mL of the ICP-MS Multielement Spike Solution (Section 8.4) and 0.250 mL of Silicon Spike (Section 8.5) to the LCS, MS, and MSD digestion vessels.

6.3. Solid matrix TCLP samples should be extracted by EPA method 1311 (TCLP) prior to digestion by 3010A. The extracts from EPA Method 1312 can also be used. Please see SGS SOP#366 for the TCLP extraction procedure.

6.4. A minimum of 75 mL of sample must be collected to allow for preparation of QC samples, i.e. 25 mL each of PS, MS and MSD.

6.5. Samples are stored at room temperature. The holding time for preserved samples requiring mercury analysis is 28 days. The holding time for preserved samples not requiring mercury analysis is six months. The digestates may be discarded one month after digestion.

7.0. APPARATUS:

7.1. Environmental Express Digestion Block with temperature control (or equivalent system that can be set to 95ºC).

7.2. SCP Science digestion vessels (or equivalent) with graduations at 10 mL, 25 mL and 50 mL (volumes of interest). Vessel graduations have a certified tolerance of ± 0.2 mL.

7.3. Eppendorf Variable Volume Pipettes (or equivalent): 0.020 mL - 0.200 mL, 0.100 mL - 1.0 mL, and 1.0 mL - 5.0 mL range pipettes are required.

7.4. Environmental Express disposable ribbed watch glasses (or equivalent).

8.0. REAGENTS:

8.1. Milli-Q filtered ultra-pure DI water.

8.2. Concentrated nitric acid (HNO_3): high purity.

8.3. Concentrated hydrochloric acid (HCl): reagent grade

8.4. ICP-MS Multielement Spike Solution: In a 100 mL class A volumetric flask, add 20 to 30 mL DI water then add 15 mL of HCl and 7 mL of concentrated HNO_3. Pipette the appropriate amount of each element stock solution into the flask. Dilute to the 100 mL mark with reagent water and mix well. Refer to **CX-0011** for the current preparation. Transfer to a 100 mL plastic black-taped Nalgene bottle. Cap bottle tightly, invert, and give to analyst to verify the concentrations.

8.5. Silicon Spike: 1,000 µg/mL single element standard. Solution is used without being mixed with any other solutions.

8.6. All reagents must be labeled using the criteria in SOP#112. Expiration dates for stock HCl and HNO_3 are 5 years from the date opened. The expiration dates for spike solutions are one year from the date made or the earliest expiration date from the stock standards used to make the spike solution, whichever is earlier.

9.0. EXTRACTION:

9.1. Label digestion vessels with sample ID. Label QC vessels (i.e., MB, LCS, MS, MSD).

9.2. Pour 25 mL of Milli-Q DI water into the digestion vessel (using the graduations) for the method blank (MB) and laboratory control sample (LCS). Shake samples prior to pouring 25 mL aliquots into correspondingly labeled digestion vessels. In three separate vessels, pour 25 mL of the parent sample chosen for quality control; one will serve as the matrix spike (MS) and another as the matrix spike duplicate (MSD). Measure out 25 mL of the remaining samples in the correspondingly labeled vessels.

For 3010/6020 TCLP digestion pour 25 mL of Milli-Q DI water for the MB and LCS. Pipette 2.5 mL (or 6.25 mL for some clients who require a lower LOQ) of QC (PS, MS, MSD) and all other samples into 50 mL digestion vessels. A leachate blank (LB) is required for 3010/6020 TCLP batches. The LB should be

prepped at 2.5 mL, unless the LB is associated with a client who requires a lower LOQ. In this case, prep the LB with 6.25 mL. Bring up to 25 mL with Milli-Q DI water.

9.3. Add 0.5 mL of the ICP-MS Multielement Spike Solution (Section 8.4) and 0.25 mL of Silicon Spike (1,000 µg/mL) to the LCS, MS, and MSD. For TCLP digestions, only add 0.5 mL of ICP-MS Multielement Spike Solution, the Silicon spike is not required.

9.4. To all vessels, add 0.8 mL of HNO_3 and 0.3 mL of HCl. Place the vessels uncapped in a preheated (95 ± 5ºC) hot block.

The temperature of the hot block is monitored using a thermometer. The thermometer is placed in a digestion vessel with a hole in the cap. The thermometer must be suspended above the bottom of the vessel and cannot touch the sides. Any space in the hole of the cap should be sealed to ensure that the water does not evaporate. The position of the thermometer must be placed in a different cell of the hot block each day of use in order to track the temperature of the entire hot block. A grid is used to identify the location of each cell in the hot block, where letters (i.e. A-I) read from left to right and numbers (i.e. 1-6) read from top to bottom (Figure 1. Hot Block Grid). The temperature and position will be monitored and recorded in the electronic laboratory notebook for each day of use. The thermometer will move to a new location each day of use using all locations in no more than a 60 day of use period.

	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
1	1A	1B	1C	1D	1E	1F	1G	1H	1I
2	2A	2B	2C	2D	2E	2F	2G	2H	2I
3	3A	3B	3C	3D	3E	3F	3G	3H	3I
4	4A	4B	4C	4D	4E	4F	4G	4H	4I
5	5A	5B	5C	5D	5E	5F	5G	5H	5I
6	6A	6B	6C	6D	6E	6F	6G	6H	6I

Figure 1. Hot Block Grid

9.5. Allow the 25 mL volume of the samples to reduce to approximately 5-10 mL (**Note**: Do not boil the sample or allow it to go dry). This will take 3 to 3.5 hours depending on the sample matrix.

9.6. Once the sample volumes are reduced to 5-10 mL, add another 0.8 mL of concentrated HNO_3 into all vessels. Cover over all vessels with disposable ribbed watch glass and reflux for 30 minutes. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

9.7. Remove the samples from the hot block, allow to cool.

9.8. Reconstitute the volume to 25 mL using Milli-Q DI water.

9.9. Cap the vessels tightly, invert, and deliver to analyst.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1. Method Blank: An MB is prepared for each digestion batch of ≤ 20 samples as a check for contamination.

12.2. Laboratory Control Sample: An LCS is prepared for digestion batch of ≤ 20 samples.

12.3. Matrix Spike and Matrix Spike Duplicate: An MS and MSD are prepared for each digestion batch of ≤ 20 samples.

12.4. TCLP extraction blanks (LB), are prepared as part of the batch in addition to the Method Blank. Label each TCLP extraction blank with the HSN and TCLP batch number.

12.5. The digestion batch will not exceed a total of 20 paying samples.

13.0. CALCULATIONS, REVIEW AND REPORTING:

N/A

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until it is determined that no further analysis will be required.

14.2. Once it has been established that no further analysis of a sample is required, acid preserved samples must be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP#108 for further instruction.

14.3. All surplus reagent acids shall be neutralized daily using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) is always required. Proper PPE when handling samples include lab coat, gloves and safety glasses. Additionally, when handling high volumes of concentrated acid, a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab regarding current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

DL Studies are completed in 3 sets with 3 spiked vessels, one MB and one LCS. The process for DL studies follow the same procedure listed in Section 9.0. For the DL spike solution refer to **CX-0015**. Spike levels may be adjusted based off the first DL Study trial. Refer to SOP#116 for further guidance.

18.0. LIMIT OF DETECTION (LOD):

Refer to SOP#116 for further guidance.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOD/LOQ spike is prepared using **CX-0015.** Prepare the digests following the same procedure listed in Section 9.0, using spike volumes specific to the LOD/LOQ sample preparation. Refer to SOP#116 for further guidance.

20.0. REFERENCES:

SW846 3010A
EPA Method 1311 and 1312 (TCLP & SPLP)

21.0. ATTACHMENTS:

Attachment A: Flow Chart

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Attachment A: FLOW CHART

Pour 25 mL of Milli-Q DI water for MB and LCS.
Shake and pour 25 mL of QC sample into three separate vessels (parent sample, MS, MSD).
Shake and pour 25 mL of the remaining samples into labeled vessel. (See Section 6.2 if lab filtering is required).
Spike LCS, MS, and MSD with 0.5 mL ICP-MS Multielement Spike Solution and 0.25 mL Silicon Spike (Section 9.3).

Add 0.8 mL of HNO_3 and 0.3 mL of HCl into all vessels.
Digest uncapped on 95 ± 5ºC hot block until volume is reduced to approximately 5-10 mL.

Add 0.8 mL of HNO_3.
Cap and place on hot block for 30 minutes reflux.
Repeat this step until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

Remove from hot block and cool.
Reconstitute volume to 25 mL with Milli-Q DI water.
Cap tightly, invert, and deliver to analyst.

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
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S.O.P. Title: Mercury in Water and Soil by Oxidation, Purge & Trap, and CVAFS	**Revision Date**: August 2023
Method No: EPA 1631E	**SOP No**: 354r15
Page: 1 of 18	**Supersedes**: 354r14

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/1/23	Samara Rentty	9/1/23

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- Update 3.5 to reflect current practice – SnCl2 in 2.0L reagent water.
- Update to 6.2 to reflect current practices.
- Update to 6.5 to include section reference for unpreserved bottles.
- Update to 6.6 to reflect current practices.
- Added sections 7.10, and 7.11 to refer to correct flask type used for analysis and updated 11.14.
- Added section 8.20 – preparing 1:1 HCl.
- Update 9.1.4 to include section reference for MB preparation.
- Labels the tables in section 10.2 and 10.3
- Updated final concentration for CCV soil in Table 1a.
- Updated section 11.7, 11.8, and 11.9 to reflect current practices for file naming and instrument method names.
- Minor grammatical changes made throughout.

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1.0. OBJECTIVE:

This Standard Operating Procedure outlines the procedure for determination of trace level mercury (<100 ng/L) in aqueous and soil samples using EPA Method 1631E.

2.0. SCOPE AND APPLICATION:

2.1 EPA method 1631: Mercury in Water and Soils by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (CVAFS), is for determination of Hg within the range of 0.5 – 100 ng/L. Prior to analysis, all Hg is oxidized to Hg (II), reduced to destroy the free halogens, then reduced again to convert HG (II) to volatile Hg (0). The Hg (0) is separated from solution and collected onto a gold trap by purging with argon and is evolved off to a CVAFS cell where the fluorescence emission of Hg (253.7 nm) is determined. The resulting fluorescence (peak height) is directly proportional to the mercury concentration.

2.2 The ease of contaminating samples with mercury and interfering substances cannot be overemphasized. Extreme care must be taken when handling samples, reagent water, and reagents. This method should be performed by analysts experienced in CVAFS techniques and who are trained thoroughly in the sample handling and instrument techniques described in method 1631E.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 The EPA method for 1631E sets the LOQ at 0.5ng/L; however, SGS operates with a standard LOQ of 1.0 ng/L with a DL of 0.31ng/L for waters and a LOQ of 0.25µg/Kg with a DL of 0.08µg/Kg for soils. For client specific cases the LOQ is set at 5.0 ng/L for waters.

3.2 The EPA method requires a reagent blank to be prepared and analyzed whenever new reagents are made. However, a method blank containing all reagents is run with every prep batch, making a separate reagent blank unnecessary.

3.3 The EPA method requires a system blank to be <0.5 ng/L. However, since this method's LOQ was doubled, the system blank criteria is <1.0 ng/L (which is to say, less than the LOQ).

3.4 The EPA method requires a system blank mean result to be <0.50 ng/L with a standard deviation (n-1) of <0.1 ng/l. However, the software SGS uses does not show a concentration value for the system blank until the system is calibrated. Instead, the software uses blank subtraction of the peak heights to give a percent recovery of the lowest standard. The percent recovery range must be between 75-125%.

3.5 The EPA method states that stannous chloride used in this method should be 200 g of $SnCl_2 \cdot 2H_2O$ into 100mL of distilled HCl diluted to 1.0L with reagent water. Due to instrumentation used and its pump settings, the stannous chloride can be reduced to 40 g of $SnCl_2 \cdot 2H_2O$ into 100mL of non-distilled HCl diluted to 2.0 L with reagent water.

3.6 The EPA method states that the stannous chloride should be purged overnight with nitrogen. SGS purges the stannous chloride for an hour with argon. System blanks, method blanks, and DL studies run with stannous purged for an hour meet all QC standards.

3.7 The EPA method requires three method blanks. SGS is inserting an additional method blank after the first MS/MSD pair in order to meet the requirements of three method blanks per batch and adding a fourth method blank for batches of more than ten samples to provide bracketing QC for the first ten.

3.8 The EPA method only requires a CCV at the end of the batch. In order to provide bracketing QC for the first half of the batch, SGS has inserted a CCV after the first MS/MSD pair.

4.0. RESPONSIBILITIES:

4.1. The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2. The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3. It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4. This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5. A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Contamination Control – Sample contamination during sample preparation and analysis constitutes one of the greatest difficulties encountered in trace level mercury determination. It is critical that extreme care be taken to avoid contamination. There are two key factors in avoiding/ reducing sample contamination: first, be aware of potential sources of contamination and second, pay strict attention to the work being done.

5.1.1 Ensure that any object or substance that contacts the sample is metal and mercury free. Also, try to minimize the amount of metals you carry into the HgLL clean room (i.e. Keys), times you enter/leave the HgLL clean room, and how often you open and close the storage cabinet in the HgLL clean room. These situations should be especially avoided when you are actively running samples, because of the possibility of dust contamination.

5.1.2 Sample containers – Only fluoropolymer or glass containers with vapor tight lids must be used for sample collection because mercury vapors can diffuse in or out of other materials. Polyethylene and/or polypropylene lab ware may be used for digestion because the sample exposure time to these materials is relatively short.

5.1.3 Sample preparation:

5.1.3.1 Digestions should be performed in a non-metal fume hood equipped with HEPA filtration. The preparation area should be cleaned with a lint-free cloth or wipe soaked in reagent water prior to working in the area.

5.1.3.2 Samples and reagents should only be handled within the digestion area to prevent samples from becoming contaminated. While not being used, the samples and reagents should be capped tightly and stored in a fume hood or in a clean zip-type bag.

5.1.3.3 Care must be taken to prevent substances in samples from contaminating the workstation and the instrumentation. Spill prevention should be the first priority, but if a spill does occur the workstation should be cleaned before continuing to process additional samples in the area.

5.1.3.4 While processing samples, clean non-talc gloves should be worn during all operations involving handling of apparatus, samples, and blanks. If another substance is touched the gloves must be replaced before continuing.

5.1.4 Sample analysis:

5.1.4.1 Contamination from sample carryover can occur if a sample is processed directly after a sample that contains a relatively high concentration of Hg. If a sample is known or suspected to have a high concentration, a rinse should be put in the position following the high concentration sample on the auto sampler (this process should be repeated until the detection is below LOQ).

5.1.4.2 Significant laboratory or instrument contamination may result when an untreated effluent, in-process waters, landfill leachates, and other undiluted samples containing concentrations above 100 ng/L are processed and analyzed. Samples known or suspected to contain Hg above concentration should be diluted before bringing them into the HgLL clean room.

5.1.4.3 Contamination by indirect contact must be considered at all times. It is imperative that every piece of the apparatus that is directly or indirectly used in collection, processing, and analysis of water samples be thoroughly cleaned.

5.2 Interferences:

5.2.1 Gold and iodide are known interferences.

5.2.2 Fluorescent intensity is strongly dependent upon the presence of molecular species in the carrier gas that can cause “quenching” of the excited atoms. Only pure argon or nitrogen should be used to limit possible interferences.

5.2.3 Water vapor may collect in the gold traps and subsequently condense in the fluorescence cell upon desorption, giving a false peak due to the scattering of the excitation radiation. This condensation is avoided by the Perma Pure Dryer system in the instrument which removes moisture from the sample before it enters the detector.

5.2.4 The use of hydroxylamine hydrochloride to remove free halogens is not needed for solid sample digestates; there is a sufficient amount of $SnCl_2$ in the bubbler to reduce both Hg (II) and free halogens in digestate aliquots smaller than 5 mL.

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6.0. SAMPLE HANDLING:

6.1. Aqueous samples must be collected in pre-cleaned fluoropolymer bottles with either fluoropolymer or fluoropolymer –lined caps. The EPA method allows the use of glass bottles if Hg is the only target analyte. Sample bottles must be tested by analyzing at a 5% frequency for each new box received to ensure that they are contaminate-free. **(Note: Bottles for DoD must be contaminate free <1/2 LOQ.)**

6.1.1. To prep sample bottles for contaminate testing, add 2.5 mL of pre-tested 1:1 12 N HCl to a bottle and fill it with reagent water (section 8.2). Add 2.5 mL of BrCl solution to the sample bottle and allow at least 24 hours at room temperature before analyzing. Sample bottles must be less than the LOQ to be considered contaminate free. If the bottles are above LOQ, do not use them and alert your supervisor.

6.1.2. Record the lot number for the sample bottles tested (the ESS lot number on the cleaning certificate, not the Nalgene© lot number), the HCl lot number used, and the date and analyst initials in the instrument Runlog.

6.2. To prepare sample bottles for field collection, add 2.5 mL of pre-tested 1:1 12 N HCl (Sec. 8.20) to a pre-cleaned 500 mL fluoropolymer bottle or add 1.25 ml of pre tested 1:1 12 N HCl to a pre-cleaned 250ml fluoropolymer bottle, tighten the lid, then secure it in two clean one-gallon polyethylene bags (zip close style Ziploc© bags or equivalent) and place a blue HCl sticker on the fluoropolymer bottle. **(Note: Nothing is placed inside the polyethylene bags except the unlabeled, preserved sample bottles.)**

6.3. If a trip bank bottle is requested for a kit, prepare the bottle according to section 6.2, but fill the bottle with reagent water. The number of trip blanks requested is per client request but should be 1 per 10 samples.

6.4. For dissolved Hg, a sample is filtered through a 0.45 µm capsule filter (not 0.45 µm hand filters) in a mercury-free clean area prior to preservation. If the sample is filtered, it must be accompanied by a reagent water blank that has been filtered under the same conditions.

6.5. Samples may be shipped to the laboratory unpreserved and un-refrigerated if they are collected in the above containers (6.1). If a sample is not collected in the above container, the sample must be preserved within 48 hours of collection. Holding time for preserved samples is 90 days. Samples must be sealed in the bags they are received in for all steps of processing. **Note: exceptionally high samples should be removed from the room as soon as they are identified to avoid contaminating other samples.**

6.6. After posting, samples should be sealed in the bags received in and packaged into cardboard boxes. Label outside of box with the work order number. Hold samples on designated Low Level shelves in sample control until disposal by current SGS protocol.

6.7. For soil, samples are collected into acid-cleaned glass, polyethylene, or fluoropolymer jars. For all except very low level and high water content samples, polyethylene bags are also acceptable. Dry solids such as coal and ores may be collected and stored in heavy gauge paper pouches commonly used by geologists.

6.8 Sample shipment, storage, preservation, and holding times

6.8.1 Dry samples - Samples such as ores, coal, paper, and wood may be shipped unrefrigerated and stored indefinitely in a cool, dry location known to have an atmosphere that is low in mercury.

6.8.2 Wet sediment samples are chilled and shipped to the laboratory at 0-6°C. Freezing and thawing may adversely affect homogeneity caused by clumping and separation of the solids from the

liquid. Therefore, wet sediment samples must be aliquoted and weighed at the laboratory and prior to freezing unless analyzed upon receipt. Wet sediment samples may be held for 1 year if aliquoted, weighed, and frozen at < -15°C. Sediment samples may be lyophilized and stored unrefrigerated for 1 year in a low-mercury atmosphere if only total Hg will be determined and no free elemental mercury (Hg0) is expected in the samples.

7.0. APPARATUS:

7.1. Instrument: PS Analytical Merlin System (or equivalent) mercury analyzer. This instrument is equipped with a high-intensity mercury lamp, a reference detector, a quartz cell, a 253.7 nm filter, and a transmission detector.

7.2. Pump: Two peristaltic pumps are incorporated in the PS Analytical Merlin Millennium System. It is capable of flow rate settings from 2-15 mL/min.

7.3. Tubing: Viton and Tygon tubing are used for sample injection, sample draining, and the reducing agent.

7.4. Drying system: Perma Pure Dryer System.

7.5. Auto sampler: An auto sampler is also incorporated in the PS Analytical Merlin Millennium System. It uses 50mL polyethylene digestion vessels and plastic sample trays.

7.6. Quality control vessels: 125mL graduated plastic digestion vessels purchased from Environmental Express (or equivalent). Results of MB's must be less than LOQ to be considered contaminant free. These vessels have an error of less than 2%.

7.7. Pipettes: Eppendorf 10-100µL, 100-1000µL, 500-5000µL (or equivalent).

7.8. Laminar flow fume hood with Class 100 clean workstation and HEPA filtered air supply.

7.9. Digi Tube 50ml digestion tubes. A C# should be assigned when received and the certificate needs to be attached.

7.10. Class A 50 mL Teflon volumetric flask for making dilutions.

7.11. Class A 100 mL Teflon volumetric flask for making intermediate and working standards.

8.0. REAGENTS:

8.1 All chemicals, reagents, and standards must be traceable back to documented written records. The expiration dates of all reagents must be recorded and written on the container. Where applicable, this information must be entered into the LIMS system. Refer to SGS SOP #500 for proper documentation procedures.

8.2 Reagent water – 18 MΩ minimum, ultra pure deionized water starting from a pre-purified (distilled, reverse osmosis, etc.) source. Water should be monitored for Hg, especially after ion exchange beds are changed.

8.3 All glassware must be thoroughly rinsed with reagent water prior to use.

8.4 The Teflon 100 mL volumetric flask must be first rinsed with 1:1 nitric acid and then three more times with reagent water

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8.5 Air – HEPA filtered source.

8.6 Reagent grade hydrochloric acid, concentrated

8.7 Reagent grade nitric acid, concentrated

8.8 Auto sampler wash solution –10% HCl solution.

8.9 Blank Solution – 2% v/v HNO_3, dilute 40 mL HNO_3 up to 2000 mL with reagent water.

8.10 Hydroxylamine Hydrochloride certified at 99.9% for mercury analysis– Dissolve 300 g of $NH_2OH \cdot HCl$ in reagent water and bring to 1.0 L. This solution may be purified by the addition of 1.0 mL of $SnCl_2$ solution and purging overnight at 500 mL/min with Hg free Argon (expires 12 months after preparation).

8.11 Stannous chloride ($SnCl_2$) – Dissolve 40 g of $SnCl_2$ into 100 mL of HCl. Bring solution up to 2.0 L with reagent water. Purge for one hour with Hg-free Argon at 500 mL/min to remove all traces of Hg. Make daily.

8.12 Bromine monochloride (BrCl) – In a fume hood, dissolve 27 g of reagent grade KBr in 2.5 L of HCl. Place a clean magnetic stir bar in the bottle and stir for approximately 1 hour in the fume hood. Slowly add 38 g of reagent grade $KBrO_3$ to the acid while stirring. When all of the $KBrO_3$ has been added, the solution color should change from yellow to red to orange. Loosely cap the bottle and allow it to stir another hour before tightening the lid (expires 12 months after preparation).

8.13 Stock Hg standard - 1000 mg/L Mercury commercially purchased. Stock Hg standard is stored outside the HgLL room in the mercury fume hood**.**

8.14 Calibration standard:

8.14.1 **Intermediate Hg standard 1 mg/L**: In a partially filled Teflon 100 mL volumetric flask, add 500 µL BrCl solution (8.12) and 100 µL of 1000 mg/L stock mercury standard (8.13) to a final volume of 100mL with reagent water. Store in a glass amber bottle for one year or until the NIST expiration date of the original stock, whichever is soonest.

8.14.2 **Working Hg standard 1.0 µg/L**: Working Hg standard is made new everyday. Using a partially filled Class A Teflon 100 mL volumetric flask, add 500 µL BrCl solution and 100 µL of 1 mg/L Intermediate Hg standard (8.14.1) to a final volume of 100 mL with reagent water. Pour into 125 mL digestion container.

8.15 Argon: 99.99% pure is controlled with an internal regulator that can be set between .13 and .50 LPM.

8.16 For each calibration, 3 System Blanks (SB) must be processed. For the SB sample pour 100 mL reagent water (section 8.2) into a 100 mL digestion vessel. Preserve with 0.5 mL of BrCl solution, cap the vessel and homogenize.

8.17 For each batch 4 Method Blanks (MB) must be processed. For the MB sample pour 100 mL reagent water (section 8.2) into a digestion vessel. Add 500 µL of BrCl solution, cap the vessel and agitate. Allow at least 24 hours before analyzing.

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8.18 An Initial Calibration Verification (ICV) and Continuing Calibration verification (CCV) must be processed. For the ICV/CCV pour 100 mL of reagent water (section 8.2) into a 100 mL digestion vessel. Add 1 mL of the working standard (section 8.14.2) and 0.5 mL BrCl solution, cap the vessel and homogenize.

8.19 A second source Quality Control Sample (QCS), (LCS in LIMS) must be processed. The QCS Spike is made the same as step (8.14) with a second source of 1000 mg/L Mercury standard. For the QCS, add 0.5 mL BrCl and 2.5 mL of the secondary working standard. Bring up to the final volume of 100 mL with reagent water (section 8.2) into a 100 mL digestion vessel. Cap the vessel and vortex.

8.20 1:1 HCl – add 100 mL concentrated HCl to 100mL reagent water for a final volume of 200 mL 1:1 HCl.

9.0. EXTRACTION:

9.1. New sample preservation for water samples.

9.1.1. Before adding BrCl, sample pH must be verified to be pH <2 by the analyst. This is accomplished by using plastic capillary tubes and narrow range pH paper. For samples with pH >2, the PM will be notified, and a sample comment will be made in the HgLL prep book as well as in LIMS.

9.1.2. Samples are preserved by analyst in the Low Level Mercury Clean room by adding 2.5 mL of BrCl solution to each 500mL sample bottle or 1.25ml of BrCl solution to each 250ml sample bottle. **If the sample contains a high amount of organic matter, add additional aliquots of BrCl until a yellow color remains.** Note in logbook amount of BrCl used to preserve each sample. Allow 24 hours at room temperature before analysis. Alternatively, pour 100 mL aliquot from a thoroughly shaken, acidified sample, into a 100 mL digestion vessel. Add 0.500 mL of BrCl solution, cap the digestion vessel, and allow a 12-hour minimum at room temperature before analysis.

9.1.2.1. Some highly organic matrices, such a sewage effluent, will require higher levels of BrCl. Add more BrCl to the sample, and allow longer oxidation times, and/or elevated temperatures (placing samples at 50°C for 6 hours). Complete oxidation can be determined by the next step 9.1.3. The sample may be diluted to reduce the amount of BrCl required, provided that the resulting level of mercury is sufficient for reliable determination.

9.1.3. An excess of BrCl should be confirmed visually for presence of a yellow color prior to sample processing or direct analysis to ensure the sample has been properly preserved.

9.1.4. Field blanks and Trip blanks are preserved along with the other samples. Method blanks should be prepared (Section 8.17) in the cleaned hood (Section 7.8) before samples are preserved.

9.2. Digestion of coal, ores, sediments, soils and other geological media.

9.2.1. Accurately weigh (to the nearest mg) an aliquot of the sample directly into a tared digestion vessel. For wet sediments and soils, weigh 0.5-1.5 grams; for dried materials such as coal, ores, and CRMs (Certified Reference Materials), weigh 0.5-1.0 gram. To better assure homogeneity, sediments and soils should be screened through a 2-mm plastic sieve to remove large rocks and sticks before digestion.

9.2.2. In a fume hood, add 8.0 mL of concentrated HCl, swirl, and add 2.0 mL of concentrated HNO_3 to the sample in the digestion vessel. Cap the vessel with a clean glass marble or inverted

fluoropolymer cone. Allow to digest at room temperature for at least 4 hours but preferably overnight.

9.2.3. For coal or other elemental carbon-containing sample, dilute the digestate to the calibration mark (40 ± 0.5 mL) with the 0.07 N BrCl solution and shake the flask to mix thoroughly. The addition of BrCl ensures that Hg will not re-adsorb to the carbon particles, producing low recoveries. After dilution and shaking, allow the sample to settle overnight, or centrifuge prior to analysis. Be sure that all fine-grained particles are completely settled prior to analysis. This settling can be hastened by centrifuging for 20 minutes at 3000 RPM or by filtering the sample through a 0.45µm filter.

9.2.4. For samples other than those containing coal or elemental carbon, dilute the digestate to volume (40 ± 0.5 mL) with reagent water so that the meniscus is at the calibration line in the neck of the digestion vessel. Shake vigorously and allow settling until the supernatant is clear prior to analysis.

9.2.5. The diluted digestates may be stored up to one year in glass or fluoropolymer containers prior to analysis, or for future re-analysis, if needed.

10.0. CALIBRATION:

10.1 Instrument operating conditions can be found in the low-level mercury maintenance log.

10.2 For soils, calibration points are made by placing 5 mL of HCl into a 50mL digestion vessel, then adding 1.25mL of HNO_3. The vessel is then filled to approximately the 30 mL mark with reagent water (Sec 8.2). The vessels are then spiked according to the following table (Table 1a.). The samples are brought up to 50 mL with reagent water and analyzed.

Table 1a. Concentration of Calibration Standards and Batch QC for Soils

Std ID	Final conc (ng/L)	Source	Initial vol (mL)	Final Vol (ml)
CB/MB	<LOQ	Sec 8.2	0	50
Std 1	5	Sec 8.14.2	.25	50
Std2	10	Sec 8.14.2	.5	50
Std3	25	Sec 8.14.2	1.25	50
Std4	50	Sec 8.14.2	2.5	50
Std5	100	Sec 8.14.2	5	50
QCS (LCS)	25	Sec 8.19	1.25	50
ICV/CCV	20	Sec 8.14.2	1	50
MS/MSD	25	Sec 8.14.2	1.25	50

10.3 The calibration must contain a minimum of 5 non-zero points and three system blanks, which are used for blank subtraction described in section 10.4 and 10.5. The amount of blank subtraction is determined by the instrument and is located in the calibration tab. The lowest calibration standard is at the minimum level (ML). For waters, calibration points are made by placing 70-80 mL of reagent water (8.2) into a 100 mL digestion vessel. The standards are then preserved with 0.5 mL of BrCl. Concentrations of the calibration standards are prepared by successive dilutions of a 1.0 µg/L Hg working standard (section 8.14.2). The solution in the vessel is then brought up to the 100 ml mark. A summary of the water standard preparation volumes is given in the table below (Table 2a.).

Table 2a. Concentration of Calibration Standards and Batch QC for Waters

Std ID	Final Conc(ng/L)	Source	Initial vol (mL)	Final Vol (mL)
CB/MB	<LOQ	Sec 8.2	0	100
Std 1	1	Sec 8.14.2	0.1	100
Std 2	5	Sec 8.14.2	0.5	100
Std 3	25	Sec 8.14.2	2.5	100
Std 4	50	Sec 8.14.2	5	100
Std 5	100	Sec 8.14.2	10	100
QCS	25	Sec 8.19	2.5	100
ICV/CCV	10	Sec 8.14.2	1	100
MS/MSD	25	Sec 8.14.2	2.5	100

10.4 Immediately prior to analysis of the standards, the excess BrCl is reduced with 0.250mL of NH_2OH solution (section 8.10). Prepare and analyze a minimum of 3 system blanks prior to analyzing the standards; begin with the lowest concentration and proceed to the highest.

10.5 Tabulate the peak heights. Calculate the mean peak height for the system blanks.

10.6 For each calibration point, subtract the mean peak height of the system blanks from the peak height for each standard. Calculate the calibration factor (CF_x) for Hg in each of the five standards using the mean reagent-blank-subtracted peak height and the following equation:

$$CF_X = ((A_X) - (A_{SB}))/ (C_X) \qquad \text{EQUATION 1}$$

Where:

A_X = Peak height for Hg in standard
A_{SB} = Mean peak height for Hg in calibration blanks
C_X = Concentration of standard analyzed (ng/L)

10.6 Calculate the mean calibration factor (CF_M), the standard deviation of the calibration factor (SD; n-1), and the relative standard deviation (RSD) of the calibration factor, where RSD = 100 x SD/CF_M

10.7 If the RSD $\leq$ 15%, calculate the recovery for the lowest standard (1.0 ng/L) using CF_M. If the RSD $\leq$ 15% and the recovery of the lowest standard is in the range of 75-125%, the calibration is acceptable, and CF_M may be used to calculate the concentration of Hg in samples, blanks, and standards. If RSD > 15% or if the recovery of the lowest standard is not in the range of 75-125%, recalibrate the analytical system and repeat the test.

10.8 Calculate the concentration of Hg in the system blanks using CF_M. The system blanks must meet the criteria in section; otherwise, mercury in the system must be reduced and the calibration repeated until the system blanks meet the criteria.

10.9 Calculation of solid phase concentrations.

10.9.1 The analytical system in Method 1631E will give analytical results in units of area (or height) for the volume of diluted digestate analyzed. To calculate the solid phase concentration, use the following equation:

$$C_{Hg} = (A_s - A_{BB}) \times V \times d \times 0.1 / (CF_m \times v \times w) \qquad \text{EQUATION 2}$$

where:

C_{Hg} = concentration of mercury in the sample (ng/g wet weight)
A_s = peak area (or height) for mercury in the sample
A_{BB} = peak area (or height) for the average of the bubbler blanks
V = volume of diluted digestate (mL)
d = dilution factor(s); e.g., a factor of 100 in
0.1 = volume in bubbler (L)
CF_m = mean CF from calibration (area (or height))/(ng/L)
v = digestate volume analyzed (mL)
w = sample weight (g)

11.0. ANALYSIS:

11.1 Power up the instrument, the auto sampler, and the computer and allow warming for at least half an hour to obtain stability.

11.2 Place the $SnCl_2$ line into the $SnCl_2$ solution (section 8.11).

11.3 Place the Blank solution line into the blank solution (2% HNO_3) (section 8.9). Check level before beginning and refill if needed.

11.4 Check the level of the auto sampler wash solution (10% HCl) (section 8.8) before beginning and refill if needed.

11.5 Connect the windings and tighten down the cassettes onto the peristaltic pumps.

11.6 Turn on the pumps by going to the analysis tab in the software and clicking the "On" toggle. Check the flow of the sample, $SnCl_2$, and blank lines to ensure proper flow to the instrument. If the flow is not appropriate, adjust tightness on cassettes or replace the winding.

11.7 Place three 50 mL digestion vessels filled with system blanks in the first three positions on the auto sampler (11-13). Load the default calibration. Initiate the rinse sequence by selecting Type: "Sample" and Name: "Rinse." Click the green arrow symbol in the software and save the file as 'LLHG yyyymmdd **R**.' Be sure to pay close attention to the location samples in auto sampler. Save the sequence, close Millennium, re-open Millennium.

11.8 Create a sequence table by clicking on the calibration icon. Reselect 'Method 1631 (EWSA)' and enter the number of blanks being used into the pop-up (3). The software should populate the page with the correct sequence and auto sampler positions (11+). If they are incorrect, correct them before pressing 'OK.'

11.9 Place the reduced system blanks and the calibration into the auto sampler. Initiate the analysis sequence by hitting the green arrow symbol in the software. Name your results file 'LLHG yyyymmdd.'

11.10 Under the column "Name," type the sample ID. After the calibration has run and passed, you may analyze the entry QC samples followed by paying samples. Samples must be inverted several times to ensure homogeneity.

11.11 For water samples, the initial volume is 50mL which is poured into a 50mL digestion vessel. Then add 0.125ml of NH_2OH into each vessel to neutralize the BrCl (the amount of NH_2OH solution required will be approximately 30 % of the BrCl volume). If extra BrCl has been added to a sample, be sure to add

additional aliquots of NH_2OH. Cap and homogenize the sample. The yellow color will disappear, indicating the destruction of the BrCl. Allow the sample to react for 5 minutes with periodic swirling to be sure that no traces of halogens remain. The final volume of the sample is 50mL.

11.12 For every 10 samples a Matrix Spike/ Matrix Spike Duplicate (MS/MSD) must be processed. Pour 100mL from a selected sample. Spike the sample by adding 2.5 mL of working standard (section 8.13.2) into the acidified sample. Process the MS/MSD in the same manner as the original sample. There must be a minimum of 2 MS/MSD pairs for each analytical batch of 20 samples.

11.13 Carryover may occur after analysis of a sample containing a high level of mercury. A rinse of reagent water should be run after samples containing high levels of mercury until the blank result is less than the LOQ.

11.14 If dilution is required, Class A 50ml teflon volumetric flask should be used.

12.0. QUALITY CONTROL:

12.1 If this method is to be used, a formal quality assurance program is required. The minimum requirements of this program consist of an initial demonstration of capability, ongoing analysis of standards and blanks as a test of continued performance, and the analysis of two Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) to assess precision and recovery.

12.2 Instrument calibration validation and calibration verification.

12.2.1 Quality Control Sample (QCS) will be analyzed after the calibration. The QCS serves as a Laboratory Control Sample (LCS) also. This standard must be from a source different than the calibration standards. The acceptance range is 77%-123%. For recoveries outside this range, repeat the analysis once. If recovery is still out, recalibration and possibly re-digestion is required.

12.2.2 An Initial Calibration Verification (ICV) standard will be analyzed after the QCS. The ICV standard is also subsequently analyzed after every 10 samples (See section 12.4 for set-up). Recovery must be 77%-123% of the true value

12.3 Preparation and recovery validation.

12.3.1 A Method Blank (MB) will be prepared with each set of samples. Number of MBs is determined by the number of samples in the batch. However, no less than four method blanks are to be run with each batch. The acceptance criterion is less than the LOQ. If the measured sample concentration is greater than 10 times the method blank contamination or less than the LOQ, the sample will not require redigestion, but the data must be flagged with a 'B' in LIMS.

12.3.2 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) will be prepared with each digest batch of 10 samples. The acceptance range is 71%-125%, RPD must be less than or equal to 24. If outside of acceptance criteria reanalyze once. If QCS is within control limits, mark as possible matrix interference. If RPD is outside acceptance criteria, recalibrate and redigest sample.

12.4 Quality of the analyses is controlled by an analytical batch. An analytical batch is a set of samples oxidized with the same batch of reagents and analyzed during the same 12-hour shift. A batch may be from 1 to 20 paying samples. Each batch must be accompanied by 3 system blanks, 1 CCV sample at the beginning and end of the batch, a QCS sample, at least 4 method blanks, and 1 set of MS/MSD at a frequency of 10%. A typical analytical sequence might appear as follows:

A) Three system blanks
B) A minimum of five, non-zero calibration standards
C) Quality control sample (LCS in LIMS)
D) On-going precision and recovery standards (ICV)
E) Method blank
F) Seven samples
G) Method blank
H) Three samples
I) Matrix spike
J) Matrix spike duplicate
K) Method Blank
L) CCV
M) Four Samples
N) Method blank
O) Six samples
P) Matrix spike
Q) Matrix spike duplicate
R) Ongoing precision and recovery standards (CCV)

The above sequence includes calibration and is an example of a typical sequence. Specifics of sample order will vary depending on analyst's judgement, number of samples and sample needs. If system performance is verified at the end of the sequence using the CCV, analysis of samples and blanks may proceed starting at line D, unless more than 12 hours has elapsed since verification of system performance. If more than 12 hours has elapsed, the sequence would be initiated at step C above.

12.5 Trip blanks are used to demonstrate that samples have not been contaminated by the sample collection or transport activities. These should be analyzed immediately before analyzing the samples in the batch (after step E above) to confirm that they are below LOQ. If the Hg concentration is above LOQ, the results for associated samples may not be reported or otherwise used for regulatory compliance purposes or DoD. However, it is up to each individual client as to whether they accept the results or not.

12.6 Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the Technical Director or QA Manager.

12.6.1 Reference Attachment A: Corrective Action Table

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 ICV/CCV/QCS Recovery: The recoveries for standard solution (both digested standards and the periodic quality control standards) are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{Vo}{Ve} \times 100 = \% \text{ recovery} \qquad \text{EQUATION 3}$$

Vo = observed value
Ve = expected value

13.2 MS/MSD Spike Recovery: The calculation for spike recoveries requires the subtraction of the sample contribution from the response of the spiked sample, and then the division of this result by the expected value of the spike. The result is multiplied by 100 to yield a percent recovery.

$$\frac{Vo - Sc}{Ve} \times 100 = \% \text{ recovery} \qquad \text{EQUATION 4}$$

Vo = observed value of the spiked sample
Ve = expected value for the spike
Sc = observed value of the sample

13.3 MS/MSD Relative Percent Difference (RPD): The relative percent difference between duplicate samples is calculated as the absolute difference between the sample and the duplicate and then divided by the average of the sample and the duplicate. The result is multiplied by 100.

$$\frac{|Sc - Dc|}{\{(Sc + Dc) / 2\}} \times 100 = \text{Duplicate Relative Percent Difference} \qquad \text{EQUATION 5}$$

Sc = observed sample spike concentration
Dc = observed sample spike duplicate concentration

13.4 When posting the prep batch data in LIMS, the posted sequence must exactly match the actual analytical run sequence. This is imperative for establishing the correct QC dependencies needed for accurate reporting.

13.5 Data from the instrument is stored on the computer hard drive and needs to be archived each month. The data is moved from C:\Program Files\P S Analytical to \\Usfs700\ank_instrument_data\MERCURY\DATA in the folder corresponding to the current year. Rename the file AA followed by the date (AALLMMDDYY) update the archive logbook.

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further

discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

17.1 Detection Limit (DL) studies are performed annually at a minimum, when a significant change in instrument response is observed or when a new instrument is purchased for an analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS.

17.2 The statistical DL must be less than or equal to the maximum DL of 0.31 ng/L for waters and 0.08 µg/Kg for soils. An update to the DL does not necessitate an update to this document.

17.3 The suggested DL spiking level is 0.5 ng/L for waters and 0.125 µg/Kg for soil. The maximum DL is 0.078 ug/Kg.

18.0. LIMIT OF DETECTION (LOD):

The LOD is set as 2x the established maximum DL.

19.0. LIMIT OF QUANTITATION (LOQ):

The Limit of Quantitation (LOQ) for mercury in water is currently 1.0 ng/L for waters and 0.25 µg/Kg for soils. This value may change under certain sample conditions (e.g., presence of matrix interferences, sample dilution).

20.0. REFERENCES:

EPA method 1631 (Revision E) Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (August 2002).

Appendix to EPA method 1631 (Revision B) Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (January 2001). Total Mercury in Tissue, Sludge, Sediment, and Soil by Acid Digestion and BrCl Oxidation.

21.0. ATTACHMENTS:

ATTACHMENT A: Corrective Action Table

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ATTACHMENT A: Corrective Action Table

System Blank (SB)	3 at beginning of run.	< 1.0 ng/L	1. Reanalyze once 2. Recalibrate 3. Reanalyze associated samples that are > LOQ and < 10x the CB contamination
Second Source Check (QCS) (LCS in LIMS)	1 per 20 samples or less (25 ng/L).	Recovery 77-123%	1. Reanalyze once 2. Recalibrate, and reanalyze 3. If still outside limits, reprep entire batch
Initial Calibration Verification (ICV)	At beginning of analysis (10 ng/L).	Recovery 77-123%	1. Reanalyze once 2. Recalibrate
Method Blank (MB)	4 method blanks per batch.	< 1.0 ng/L For DoD <0.5 ng/L	1. Reanalyze once 2. Recalibrate 3. Reanalyze associated samples that are > LOQ and < 10x the CB contamination
Matrix Spike /Matrix Spike Duplicate (MS/MSD)	1 per 10 samples (25 ng/L).	Recovery 71-125% RPD ≤ 24%	1. Reanalyze once 2. Check QCS recovery 3. If QCS is in control, note in QC summary as possible matrix interference 4. If RPD out, re-digest and reanalyze 5. If still outside control limits, flag sample as non-homogenous
Continuing Calibration Verification (CCV)	After the first MS/MSD pair and at end of analysis (10 ng/L).	Recovery 77-123 %	Non-DOD 1. Reanalyze once, if passing rerun the above samples so all have bracketing passing CCV'S. The run can be continued. 2. Failure, Recalibrate DOD 1. Rerun CCV twice if both are passing continue. Samples above CCV's may still be posted. 2. Failure, Recalibrate

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure	
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.		
S.O.P. Title: Acid Digestion of Soils, Sludges and Sediments for ICP-MS		**Revision Date**: May 2021
Method No: SW846 3050B		**SOP No**: 361r18
Page: 1 of 11		**Supersedes:** 361r17

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	5/24/21	Jamaica Rowtry	5/24/21

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Summary of Changes from Previous Revision:

- Edited section 9.5 to mention electronic laboratory notebook instead of prep logbook.
- Updated section 9.8.1 to digest oils until clear and no longer effervescing.
- Various grammatical/formatting corrections.

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1.0. OBJECTIVE:

This document outlines a procedure for acid digestion of sediments, sludges, soils, and oil samples for metals.

2.0. SCOPE AND APPLICATION:

This SOP outlines the preparation of sediments, sludges, soil, and oil samples for analysis by inductively coupled plasma mass spectrometry (ICP-MS).

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 The reference method (SW846 3050B) is designed to accommodate large reagent volumes, glassware, and hardware. The digestion times and reagent amounts have been reduced for this method because the hotblocks and digestion vessels used are smaller than those of the reference method.

3.2 Chapter Three of SW 846 discourages the use of HCl for the digestion procedure for ICP-MS analysis unless needed to enhance performance. SGS experience has shown that no significant interference occurs from the addition of HCl. The use of HCl has shown marked improvement in the recovery of Silver (Ag) and Barium (Ba). HCl is used in all digestions for ICP-MS digestions as documented in this procedure.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit necessary revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QA/QC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

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5.0. INTERFERENCES:

Sediment, sludges, and soil samples can contain diverse matrix types, each of which can pose its own analytical challenges. Laboratory Control Sample (LCS) and Matrix Spike/ Matrix Spike Duplicate (MS/MSD) samples are prepared with each digest batch to determine the validity of the method, 3050B, for a particular sample.

6.0. SAMPLE HANDLING:

Soils, sediments, and sludges are refrigerated upon receipt at 0-6°C and analyzed as soon as possible. The holding time is six months from collection to analysis. Digests are discarded one month after the analysis date. The holding time for samples is 180 days. If mercury analysis is required, the holding time is 28 days.

7.0. APPARATUS:

7.1. Environmental Express 36 or 54 well digestion block with temperature control (or equivalent).

7.2. Environmental Express HotBlock digestion vessels (or equivalent). Vessels are graduated every 5 mL from 5 mL to 50 mL with a maximum capacity of 62 mL. The graduations have a certified tolerance of $\pm$0.2 mL.

7.3. Eppendorf Reference Pipettes (or equivalent): 0.02 – 0.2 mL, 0.2 – 1.0 mL, and 1.0 – 5.0 mL with an accuracy of $\pm$1% (*note*, micro-pipettes accuracy tolerance may be extended to 2% per SGS SOP #104).

7.4. Analytical balance capable of measuring to 0.1 mg.

8.0. REAGENTS:

8.1. Milli-Q filtered ultra-pure water.

8.2. Concentrated nitric acid (HNO_3), reagent grade.

8.3. Concentrated hydrochloric acid (HCl), reagent grade.

8.4. Hydrogen peroxide, 30% (H_2O_2).

8.5. ICP-MS Matrix Spike: following table lists the concentration of each analyte in the spike solution.

Aluminum	50 µg/mL	Cobalt	25 µg/mL	Selenium	50 µg/mL
Antimony	50 µg/mL	Copper	50 µg/mL	Silver	5 µg/mL
Arsenic	50 µg/mL	Iron	250 µg/mL	Sodium	500 µg/mL
Barium	50 µg/mL	Lead	50 µg/mL	Strontium	50 µg/mL
Beryllium	5 µg/mL	Magnesium	500 µg/mL	Thallium	0.5 µg/mL
Bismuth	5 µg/mL	Manganese	25 µg/mL	Tin	5 µg/mL
Boron	50 µg/mL	Molybdenum	20 µg/mL	Titanium	5 µg/mL
Cadmium	5 µg/mL	Nickel	50 µg/mL	Vanadium	10 µg/mL
Calcium	500 µg/mL	Phosphorus	25 µg/mL	Zinc	50 µg/mL
Chromium	20 µg/mL	Potassium	500 µg/mL	Gold	5 ug/mL
Lithium	10 µg/mL	Uranium	5 µg/mL	Mercury	0.5 ug/mL

8.6. Preparation of the ICP-MS Matrix Spike.

8.6.1. In a 100 mL class A volumetric Teflon flask, add 20 to 30 mL DI water then add 15 mL of concentrated HCl and 7 mL of concentrated HNO_3.

8.6.2. Refer to CX-0011 for the list of element stock solutions and correlating spike volumes. Note that "ICAL" refers to a mixture of Ca, Mg, K, and Na.

8.6.3. Bring up to volume (100 mL) using deionized water and transfer to a 100-mL plastic Nalgene bottle wrapped in electrical tape. Cap bottle tightly, invert, and give to analyst to verify the concentrations.

8.6.4. Silicon Standard: 1,000 µg/mL single element standard of which 0.5 mL is used in digestion. Solution is used as is without being mixed into any other solutions.

8.7. All reagents must be labeled following the criteria in SOP #112. Expiration dates for HCL, HNO3, and H_2O_2 are one year from the date opened. The expiration dates for spike solutions are one year from the date made or the earliest expiration date from the stock standards used to make the spike solution, whichever is earlier.

9.0. EXTRACTION:

9.1 Label digestion vessels (section 7.1) with work order and sample number. Label QC vessels (MB, LCS, MS, MSD, DUP), see 9.3 below.

9.2 See SOP#143 for the proper procedure for weighing 1.0-1.1 grams of sample. For glycol batches, measure 1.0 mL of sample into digestion vessels. Record the actual weight in the logbook (except glycols, record 1 mL). See SOP#149 for Multi-Incremental Soil sample weighing procedure. Record total weight of MI sample in logbook (will be between 10-11 grams). Digest each MI sample in 10 separate vessels following the procedure outlined in section 9.3. Please note one MI sample (10 vessels) equates to one paying sample.

9.3 Preparation of batch QC is as follows:

9.3.1. Method Blank (MB): A MB is prepared for each digestion batch of $\leq$ 20 paying samples. Teflon stones are used for the MB to represent a solid matrix. For oils batches the base oil standard is used in place of Teflon. Weigh out 1.0-1.1 g of Teflon chips into a tared digestion vessel, then proceed to step 9.3.5. For oils batches, weigh out 1.0-1.1 g of the base oil standard. For glycol batches, measure 1.0 mL DI water.

9.3.1.1. For glycols batches, a Method Blank Filter (MBF) is prepared at the same time as any glycol samples requiring lab filtering. The MBF is batched in LIMS and given a sample ID number. The blank is carried through the digestion process along with the associated samples.

9.3.2. Laboratory Control Sample (LCS): An LCS is prepared for each digestion batch of $\leq$ 20 paying samples. Weigh out 1.0-1.1 grams of Teflon stones into a tared digestion vessel. For oils batches, weigh out 1.0-1.1 g of the base oil standard. For glycol batches, measure 1.0 mL DI water. The LCS is prepared by adding 1.0 mL of ICP spike solution (section 8.5) and 0.5 mL of Silicon Standard solution (section 8.6.4).

9.3.3. Matrix Spike (MS) and Matrix Spike Duplicate (MSD): Choose a random sample within the batch to spike (MS/MSD) unless client specifies BMS/BMSD on a sample. Weigh out 1.0-1.1 g (1.0 mL for glycols) of sample into the tared and labeled digestion vessels. The MS\MSD is prepared by

adding 1.0 mL of ICP spike solution (section 8.5) and 0.5 mL of Silicon spike solution (section 8.6.4).

9.3.4. Duplicate (DUP): The sample chosen for the MS/MSD needs to be prepared in duplicate. Weigh out 1.0-1.1 g (1.0 mL for glycols) of sample into the tared and labeled digestion vessel.

9.3.5 **Note:** This digestion procedure is applicable to the digestion of air sampling filters and sample wipes. The entire filter or wipe is used for the digestion – do not cut or otherwise subdivide a filter or wipe. Filters and wipes do not require weighing before digestion, record the initial weight as 1.0 g. Digest according to section 9.0, and fill to the final volume of 50 mL, as usual. Wipes are typically digested on batches containing other solid/soil samples. The parent sample of the MS/MSD/DUP is selected from the solid/soil samples. If there are only wipes making up the digestion batch, an LCS and LCSD set must be prepped (in place of MS/MSD/DUP).

9.4. Add 2.5 mL of Milli-Q water (section 8.1) and 2.5 mL of concentrated HNO_3 (section 8.2) to each digestion vessel.

9.5. Cover digestion vessels loosely with the lids (DO NOT TIGHTEN) and heat in the hot block for 15 minutes at 95 ± 5ºC. This step is to ensure that the samples are up to temperature. Monitor samples closely for reactivity as they heat.

The temperature of the hot block is monitored using a thermometer. The thermometer is placed in a digestion vessel with a hole in the cap. The thermometer must be suspended above the bottom of the vessel and cannot touch the side of the walls. Any space in the hole of the cap should be sealed to ensure that the water does not evaporate. The position of the thermometer must be placed in a different cell of the hot block each day of use to track the temperature of the entire hot block. A grid is used to identify the location of each cell in the hot block, where letters (i.e. A-I) read from left to right and numbers (i.e. 1-6) read from top to bottom, this is illustrated in Figure 1 below. The temperature and position will be monitored and recorded in the electronic laboratory notebook for a different cell during each day of use. The thermometer will move to a new location each day of use, using all locations in no more than a 60 day of use period.

	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
1	1A	1B	1C	1D	1E	1F	1G	1H	1I
2	2A	2B	2C	2D	2E	2F	2G	2H	2I
3	3A	3B	3C	3D	3E	3F	3G	3H	3I
4	4A	4B	4C	4D	4E	4F	4G	4H	4I
5	5A	5B	5C	5D	5E	5F	5G	5H	5I
6	6A	6B	6C	6D	6E	6F	6G	6H	6I

Figure 1. HotBlock Grid

9.6. Remove samples and allow to cool for 5 minutes; then add 2.5 mL of concentrated HNO_3.

9.7. Place samples into hot block and heat for 45 minutes at 95 ± 5ºC. If sample is emitting reddish-brown fumes after cooling (approximately 5 minutes after removing from hot block), repeat steps 9.5 and 9.6 until sample no longer emits fumes. Monitor samples closely for reactivity.

9.8. Allow samples to cool for approximately 5 minutes. For soils/wipes add 1.5 mL Milli-Q water and 1.5 mL 30% H_2O_2 (**for oil and glycol batches add 1.5-2.5 mL 30% H_2O_2**) to each vessel. Allow initial effervescence to subside. Return samples to the hot block to continue the peroxide reaction. Heat samples for 30 minutes (90 min-150 min for oils and glycols). If no effervescence is observed proceed to step 9.9. For oil and glycol batches, follow procedure below for additional required 30% H_2O_2 steps. If effervescence

is still observed then cool, add 0.5 mL of 30% H_2O_2. Heat for an additional 30 minutes. Repeat until effervescence mostly subsides. Do not exceed 5 mL of 30% H_2O_2 for soils/wipes.

9.8.1. For **oil** batches continue to add 0.5 mL-1.0 mL of 30% H_2O_2 every 30-60 minutes until a final volume of 8-10 mL 30% H_2O_2 has been added (or when digestate is clear and no longer effervesces). Heat for 30 minutes then proceed to step 9.9.

9.8.2. For **glycol** batches continue to add 0.5-1.0 mL of 30% H_2O_2 every 30-60 minutes until a final volume of 5.0-6.0 mL 30% H_2O_2 has been added (or when digestate is clear and no longer effervesces). Heat for 30 minutes then proceed to 9.9.

9.9. Remove samples and allow them to cool for approximately 5 minutes. Add 1.0 mL of concentrated HCl. Place samples on the hot block for an additional 15 minutes.

9.10. Remove from hot block and allow to cool.

9.11. Dilute to 50 mL with Milli-Q water, cap digestion vessels, invert and shake well. Prior to analysis, particulates in the digestate must settle.

9.11.1. For **MI** samples, dilute each vessel (10 per paying sample), to 50 mL with Milli-Q water. Shake, and combine all 10 subaliquots into a 1 L HDPE bottle. Record the final volume of 500 mL in the logbook.

9.12. Post the samples in LIMS, label them, and deliver the batch to the analyst.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1. Method blank: A MB is prepared for each digestion batch of $\leq$ 20 samples as a check for contamination.

12.2. Laboratory Control Sample: An LCS is prepared for each digestion batch of $\leq$ 20 samples.

12.3. Matrix Spike / Matrix Spike Duplicate: An MS/MSD set is prepared for each digestion batch of $\leq$ 20 samples. In the case of a "wipe-only" batch, this requirement will be met by preparing an LCS/LCSD set.

12.4. Duplicate: A DUP is prepared for each digestion batch of $\leq$ 20. The sample chosen for the MS/MSD will also be prepared as the DUP.

12.5. The digestion batch cannot exceed a total of 20 paying samples.

13.0. CALCULATIONS, REVIEW AND REPORTING:

N/A

14.0. HEALTH AND SAFETY:

14.1. Appropriate eye protection, goggles, and gloves will always be worn.

14.2. All digestion steps will be performed inside the designated hood.

14.3. A face shield, apron and sleeves will be worn when handling concentrated acid solutions.

14.4. Samples will be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.5. Once it has been established that no further analysis of a sample will be required, acid and alkaline preserved samples will be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP#108 for further instruction.

14.6. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.7. Proper Personal Protective Equipment (PPE) must always be worn. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield, apron and sleeves must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab regarding current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

DL Studies are completed in 3 sets with 3 spiked vessels, one MB and one LCS and digested on 3 separate days. The process for DL studies follow the same procedure listed in Section 9.0. For the DL spike solution refer to **CX-0015**. Spike levels may be adjusted based off the first DL Study trial. Refer to SOP#116 for further guidance.

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

The LOD/LOQ spike is prepared using **CX-0015.** Prepare the digests following the same procedure listed in Section 9.0, using appropriate spike volumes for the LOD/LOQ sample preparation. Refer to SOP#116 for further guidance.

20.0. REFERENCES:

SW846 3050B

21.0. ATTACHMENTS:

Attachment A: Flow Chart

Attachment A: FLOW CHART

Label digestion vessels and weigh 1 gram of homogenized sample into appropriately labeled vessel. Weigh 1.0-1.1 g Teflon stones for MB, LCS (and LCSD if applicable). OIL: 1.1-1.0 g oil base standard. GLYCOL: 1.0 mL DI water. Also requires filter blank. Ensure sample chosen for QC has MS, MSD, DUP (1.0-1.1 g each or 1.0 mL for glycols).

↓

Spike the LCS (and LCSD if applicable), MS, and MSD with 1.0 mL ICP/ICP-MS Matrix Spike Solution and 0.5 mL Silicon Spike.

↓

Add 2.5 mL of Milli-Q water, then 2.5 mL of concentrated HNO_3. Cover loosely with lids. Place into preheated 95±5ºC hot block and heat for 15 minutes.

↓

Remove from hot block. Cool 5 min. Add 2.5 mL of concentrated HNO_3.
Place samples back onto 95±5ºC Hot Block. Heat for 45 minutes.

- Red fumes observed after 5 minutes (see step 9.6) → return to "Remove from hot block. Cool 5 min. Add 2.5 mL of concentrated HNO_3."
- Red fumes not observed after 5 minutes (see step 9.6) ↓

Remove from hot block and cool to room temperature. Add 1.5 mL of Milli-Q water and 1.5 mL of 30 % H_2O_2 (or 0.5 mL 30% H_2O_2 if this step is being repeated). Place samples onto 95±5ºC Hot Block for 30 minutes. Do not exceed 5 mL H_2O_2 if this step must be repeated several times. See section 9.8 for oils and glycols.

- Effervescence observed after 30 minutes → return to "Remove from hot block and cool to room temperature. Add 1.5 mL of Milli-Q water and 1.5 mL of 30 % H_2O_2 (or 0.5 mL 30% H_2O_2 if this step is being repeated). Place samples onto 95±5ºC Hot Block for 30 minutes. Do not exceed 5 mL H_2O_2 if this step must be repeated several times. See section 9.8 for oils and glycols."
- Effervescence not observed after 30 minutes ↓

Add 1.0 mL of concentrated HCl
and reflux on hot block for 15 minutes

↓

Cool, dilute to 50 mL, cap tightly, invert and shake samples.
Particulates must be allowed to settle prior to analysis.

↓

Post, label and give to analyst.

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NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Sulfide in Water - Colorimetric	**Revision Date**: November 2022
Method No: EPA 376.2; SM4500S^{2}; Hach Method 8131	**SOP No**: 368r14
Page: 1 of 11	**Supersedes:** 368r13

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	11/16/22	Tamara Rentz	11/16/22
Stephen C. Ede	10/31/23	Tamara Rentz	10/31/23

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Tamara Rentz 2022.11.21 06:36:18 -09'00'

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Summary of Changes from Previous Revision:

- Edited section 12.3 and attachment B to reflect current MB corrective actions.
- Section 8.4 was updated to clarify LCS standard preparation.
- Minor grammatical changes throughout
- Minor grammatical changes throughout.

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1.0. OBJECTIVE:

This method is for the determination of sulfide in aqueous samples. Sulfide is reacted with N, N-dimethyl-p-phenylenediamine sulfate to produce methylene blue. The intensity of the color is proportional to the sulfide concentration.

2.0. SCOPE AND APPLICATION:

2.1. This method is for the determination of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes in concentrations up to 20 mg/L.

2.2. Acid insoluble sulfides are not measured by this method. Copper sulfide is the only common sulfide in this class.

3.0. DEVIATIONS FROM REFERENCE METHOD:

There are no deviations from the reference method.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP, and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Strong reducing substances such as sulfite, thiosulfate and hydrosulfite interfere by reducing the blue color or preventing its development.

5.2. High concentrations of sulfide may inhibit full color development and require sample dilution.

5.3. For turbid samples, a sulfide-free blank can be prepared and used in place of the deionized water blank in the procedure.

6.0. SAMPLE HANDLING:

6.1 Sample Matrix – Drinking, surface, and saline waters, and domestic and industrial wastes.

6.2 Sample Collection – The samples must be collected so that headspace is minimized. The sample should be unaltered and unopened prior to analysis.

6.3 Sample Size – Samples are collected in 250 mL inert plastic containers.

6.4 Sample Preservation – Samples are preserved with 2 mL 2N zinc acetate per liter, and sufficient NaOH to keep the pH above 9. Samples require refrigeration at 0-6°C.

6.5 Holding Times – Samples must be analyzed within 7 days of collection.

6.6 If samples have a pH below 9, are not stored at 0-6ºC, or if samples have broken hold time, approval from Project Manager (PM) must first be obtained before proceeding with analysis.

7.0. APPARATUS:

7.1 Hach DR/890 (or equivalent).

7.2 Hach sample cells with 25 mL marks

7.3 Pipettes (Eppendorf)

7.4 25 mL Class A graduated cylinder.

8.0. REAGENTS:

8.1. **Deionized (DI) water**

8.2. **Sulfide 1 Reagent:** Commercially purchased (unit number 1816-32). Contains demineralized water and sulfuric acid.

8.3. **Sulfide 2 Reagent:** Commercially purchased (unit number 1817-32). Contains Potassium Dichromate and demineralized water.

8.4. **Stock Sulfide LCS Standard:** Pat dry a sulfide crystal and crush or use sodium sulfide as is from the reagent container if not in crystal form. Dissolve 0.748 g of $Na_2S{\cdot}9H_2O$ into 80 mL of DI water in a 100-mL volumetric flask. Then, dilute to mark. Transfer to 2 mL vials with no headspace and store in a box at 4$\pm$2ºC. The concentration is 998 mg/L. Make fresh every three months or when the analysis indicates that the standard has degraded. Vials should not be re-capped once standard has been removed. Dispose of any remaining standard from an opened vial.

8.4.1. Working Sulfide standard (49.9 mg/L). Dilute 0.5 mL of stock sulfide standard to 10 mL DI.

8.5. **Bromine Water:** Sulfide reagent purchased from Hach.

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8.6. **Phenol Solution:** Sulfide reagent purchased from Hach

9.0. EXTRACTION:

N/A

10.0. CALIBRATION:

The HACH DR/890 Spectrometer is pre-calibrated for sulfide analysis. Calibration is verified by successfully analyzing a laboratory control sample and a low point calibration verification sample. If calibration becomes necessary, follow the manufacturer's guidelines.

11.0. ANALYSIS:

11.1. Using pH paper, confirm and document that the pH of the sample is greater than 9. Check bottle and/or the cap for a notation indicating the sample is preserved with zinc acetate. If the sample is not properly preserved, contact the Project Manager, and make a note in the sample preparation log.

11.2. Pipette 25 mL of sample into a clean sample cell.

11.3. Prepare a method blank for each batch of 20 samples or less by adding 25 mL of DI water to a sample cell.

11.4. Create a laboratory control standard (LCS) at 0.5 mg/L with each batch of 20 field samples or less. Pipette 25 mL of DI water into a sample cell and add 250 µL working sulfide standard (8.4.1.).

11.5. Prepare a low point calibration verification sample (LPCV) at 0.1 mg/L with each batch of 20 samples or less. Pipet 200 µL of working sulfide standard (8.4.1) into 100 mL flask and fill to the mark with deionized water, mix then pipette 25 mL of the LPCV into a sample cell.

11.6. Prepare a matrix spike (MS) and matrix spike duplicate (MSD) sample for every batch of 20 paying samples or less (5%), with a concentration of 0.5 mg/L. Pipette 250 µL of working sulfide standard (8.4.1.) into 25 mL of the samples designated as the MS/MSD.

11.7. Load the sulfide program by pressing **PRGM**. Once the display shows **PRGM ?** press **93 ENTER** and the display will show the concentration units and zero prompt.

11.8. For turbid samples, use a sulfide free blank in place of the DI blank. Prepare as follows:

11.8.1. Measure 25 mL of sample into a 50 mL Erlenmeyer flask.
11.8.2. Add bromine water dropwise with constant swirling until a permanent yellow color just appears.
11.8.3. Add phenol solution dropwise until the yellow color just disappears. Use this solution in place of the DI blank.

11.9. If a lower detection limit is requested by the client, the following procedure can be used to concentrate the sample:

11.9.1. To concentrate a sample or to remove interferences, add zinc acetate and NaOH to 100 mL of sample, mix & allow to settle. Decant and discard supernatant. Use DI to restore the sample to original volume or a portion of original volume if concentration is desired.

11.10. Once the samples and blank have been prepared in the sample cells add 1.0 mL of Sulfide 1 Reagent to each cell and swirl to mix. Then add 1.0 mL of Sulfide 2 Reagent to each cell and immediately swirl to mix. A pink color will develop and the solution will turn blue if sulfide is present.

11.11. Press **TIMER ENTER**. A 5-minute reaction period will begin.

11.12. Clean the outside of the blank vial with a Kim Wipe and place the vial into the holder. Press **ZERO**.

11.13. After the timer beeps, clean the outside of the sample vial with a Kim Wipe. Place vial into adapter and press **READ**. Record the concentration of samples.

12.0. QUALITY CONTROL:

12.1. Laboratory Control Standard (LCS) will be prepared with every prep batch of 20 samples or less. The acceptance criterion is +/- 25%. LCS failure action: re-extract the batch. A LCS duplicate (LCSD) will be required if the sample duplicate fails to meet QC limits. Re-extraction and reanalysis may be required if, after one rerun, the LCS/LCSD RPD is greater than 25.

12.2. A matrix spike (MS/MSD) is prepared for each matrix in a batch at a frequency of 5% for every 20 samples or less. The recovery limit for the MS / MSD is +/- 25%. If the MS or MSD fails these criteria, flag the parent sample and use the LCS for the batch QC. The relative percent difference (RPD) between the MS and the MSD must be less than or equal to 25. If the RPD is outside this limit, the RPD of a LCS /LCSD can be used and the sample is flagged. Both the LCS and the LCSD must be reported, the LCSD is only evaluated for RPD.

12.3. A method blank (MB) is required for each matrix at a frequency of one (1) for every 20 samples or less. The acceptance criterion is less than the LOQ, some clients require an acceptance criteria of less than or equal to ½ the LOQ. In the event of MB contamination, samples with concentrations greater than or equal to 5 times the blank contamination do not require reanalysis and can be reported. Samples with concentrations less than 10 times the blank contamination must be reanalyzed. Samples with results less than the LOQ do not require reanalysis.

12.4. A Low Point Calibration Verification sample (LPCV) will be prepared at the LOQ and analyzed with every batch. The acceptance criteria is ± 50%.

12.5. Any corrective action required to address a QC outlier or technical problem which is not listed in this SOP requires the prior approval of the QA Manager or Technical Director.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 Concentration is given in mg/L, to two decimal places.

13.2 Equations

13.2.1 LCS/LCSD Recovery Calculation

%Recovery = (Vo/Ve) x 100

where Vo = observed value
Ve = expected value

13.2.2 Matrix Spike Recovery Calculation

%Recovery = (Vo-Sc)/Ve x 100

where Vo = observed value of spiked sample
Sc = sample concentration
Ve = expected value of spike

13.2.3. MS/MSD Relative Percent Difference (RPD) Calculation

RPD = (Sc - Dc)/{(Sc + Dc) /2} x 100 Where Sc = observed sample concentration
Dc = observed duplicate sample concentration

14.0. HEALTH AND SAFETY:

14.1. Sulfide 1 reagent is extremely corrosive, and Sulfide 2 reagent is a carcinogen. Only handle the reagents under a fume hood. Use extreme caution in avoiding contact and inhalation.

14.2. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.3. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.4. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.5. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.6. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

15.1 SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

15.2 Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium at a concentration regulated as a hazardous waste. Dispose of samples containing the Sulfide 2 reagent in a specified waste container kept in the flammables room.

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

DL studies are performed when a significant change in instrument is required. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

18.0. LIMIT OF DETECTION (LOD):

The LOD is ½ of the established LOQ.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP 116. LOQ verification shall be performed quarterly according to the schedule set by the QC office. The LOQ for this method is currently 0.10 mg/L.

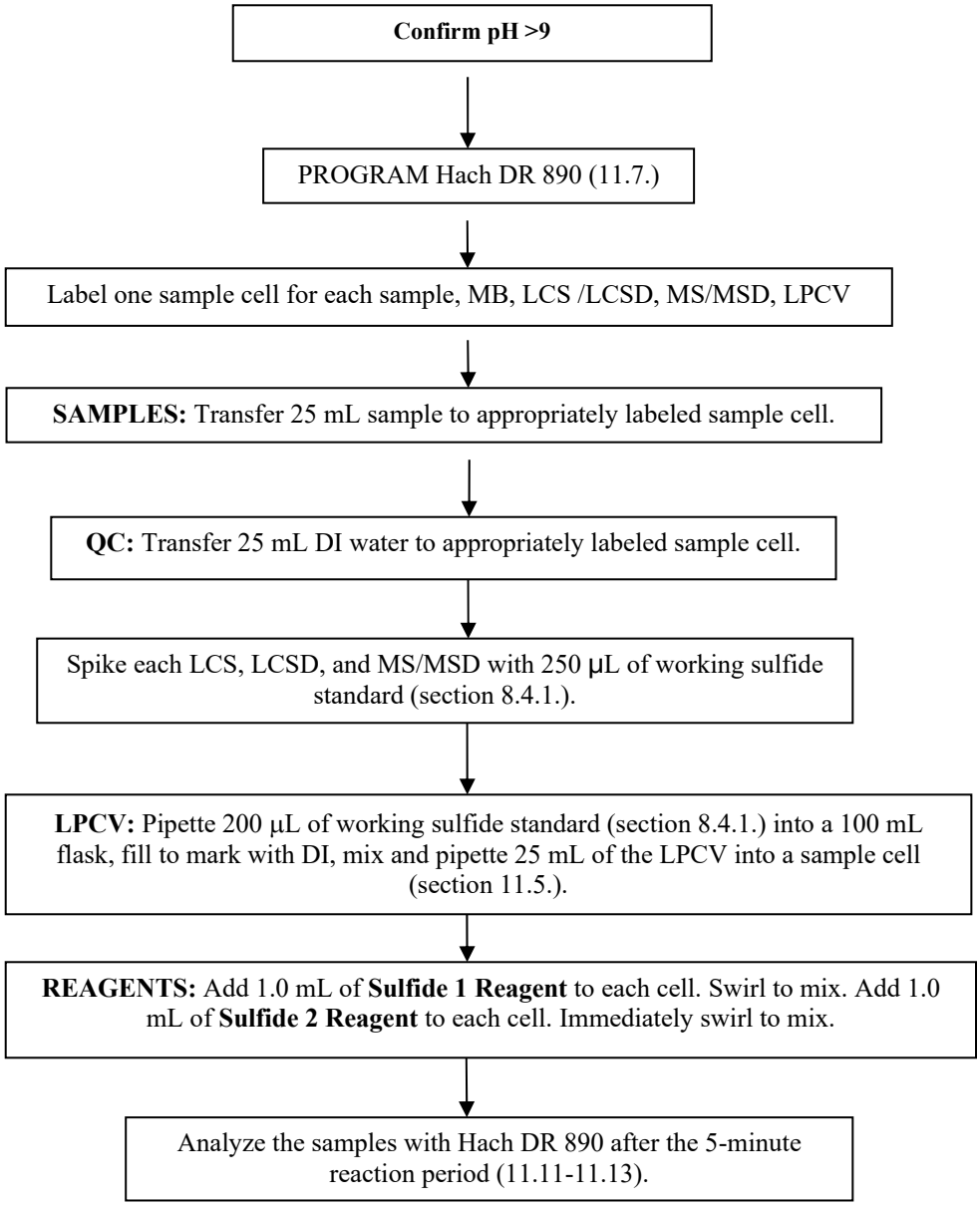
20.0. REFERENCES:

EPA Method 376.2
Standard Method 4500S^{2-}-D: Methylene Blue Method, 23rd edition.
HACH Method 8131

21.0. ATTACHMENTS:

Attachment A: Flow Chart
Attachment B: Corrective Action Table

ATTACHMENT A: Flow Chart



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ATTACHMENT B: Corrective Action Table

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
4500S; 376.2	QC Sample (LCS)	1 per batch of ≤ 20 samples.	± 25%	1. Batch must be reanalyzed.
	Method Blank	1 per batch of ≤ 20 samples.	< LOQ	1. Evaluate samples: concentration > 5x MB contamination or < LOQ are ok to report. 2. Reanalyze all samples not fitting the criteria above.
	Matrix Spike (MS) Matrix Spike Duplicate (MSD)	1 per batch of ≤ 20 samples.	Recovery ± 25%	1. Use LCS QC and flag the sample for matrix interference.
	Matrix Spike / Matrix Spike Duplicate (MS/MSD)	1 per batch of ≤ 20 samples.	RPD ≤ 25	1. Use LCS, LCSD RPD and flag the sample.
	Laboratory Control Sample (LCS) Laboratory Control Duplicate (LCSD)	1 per batch of ≤ 20 samples	Recovery ± 25% RPD ± 25	1. Repeat analysis once. 2. Reanalyze the batch. 1. Use to verify batch if sample duplicate is outside limits.
	Low Point Calibration Verification	1 per batch of ≤ 20 samples.	Recovery ± 50 %	1. See QA Manager or Technical Director.

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S.O.P. Title: Total Recoverable Metals Low Level Preparation	**Revision Date**: April 2022
Method No: EPA 200.2 / MM.LL	**SOP No**: 375r07
Page: 1 of 11	**Supersedes**: 375r06

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Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	4/12/22	Jamaica Reuter	4/12/22

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Each staff member responsible for this SOP will print & sign this coverage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Controlled copy distributed to Susan Chapnick with VHB on 04/12/2022.

Summary of Changes from Previous Revision:

- Added section 8.5
- Updated section 9.6 to reflect change from MS/MSD to 10% MS batch QC criteria.
- Edited section 9.9 to replace prep logbook with electronic laboratory notebook and remove redundant language.
- Added capping digestion vessels with Milli-Q water rinsed cap to section 9.10.
- Updated sections 9.9, 12.3, and Attachment A to reflect new MS criteria.
- Minor grammar throughout.

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1.0. OBJECTIVE:

This document outlines a procedure of sample digestion for total recoverable *low level* metal analytes.

2.0. SCOPE AND APPLICATION:

This SOP is applicable to the preparation of drinking waters, ground waters, surface waters, waste waters (industrial and domestic) and storm water using acid digestion for analysis by Inductively Coupled Plasma - Mass Spectroscopy (200.8). Specifically, this method differs from the reference by concentrating metal ions present in low quantities.

3.0. DEVIATIONS FROM REFERENCE METHOD:

The reference method (EPA.200.2) is designed to accommodate large reagent volumes, glassware, and hardware. The digestion times and reagent amounts have been reduced for this method because the hot blocks and digestion vessels used are smaller than those of the reference method.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

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4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

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4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1 Samples containing large amounts of suspended or dissolved solids should be carefully monitored because splattering may occur causing contamination and/or loss of analyte.

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5.2 To avoid contamination, all equipment coming into contact with the sample should be thoroughly cleaned and exposure of the digestates to the environment limited.

5.3 This method is suitable for the preparation of samples containing < 0.1 mg/L silver. For samples containing silver at concentrations > 0.1 mg/L, notify the supervisor or project manager. A sample may need to be prepared a second time or diluted in an approved manner.

5.4 When boron and silica are to be determined, only plastic, Teflon, or quartz lab ware should be used for collection and preparation.

5.5 This method will dissolve and hold in solution only minimal amounts of barium in the presence of free sulfate. For the analysis of barium in samples having unknown amounts of sulfate, the analysis should be performed as soon as possible after preparation.

6.0. SAMPLE HANDLING:

6.1. Aqueous samples that require total recoverable metal analyses should not be filtered. They should be preserved to a pH of < 2 with nitric acid.

6.1.1. Samples should be field preserved, using nitric acid, to a pH < 2.

6.1.2. If samples are not field preserved, they must be preserved upon receipt at the laboratory. Following acidification, the sample should be mixed, tagged with a "pH adjusted: wait 24 hours" sticker and held for 24 hours prior to digestion. The pH of samples preserved in the lab should be tested by the analyst immediately prior to taking an aliquot. The time, date, and initials from both the initial preservation and the 24 hour pH check will be recorded in the Metals pH Verification logbook. If the sample has not equilibrated to pH < 2, then this step must be repeated.

6.2. The holding time for preserved samples is six months. The samples are stored at room temperature. Digests are held for one month after analysis.

6.3. Samples should be field preserved, using nitric acid, to a pH < 2. Acid preservation to pH < 2 will be verified upon sample receipt. If samples are not field preserved, additional HNO3 will be added to samples with pH > 2. (As per SOP#118, 2-ml of 1:3 HNO3 will be added to 250-ml containers). Following acidification, the sample should be mixed and held for 24 hours prior to sample preparation or analysis.

7.0. APPARATUS:

7.1 Pipette: Eppendorf (or equivalent) variable volume pipettes:10 -100 µg/mL and 100 - 1000 µg/mL.

7.2 Hot Block: Environmental Express (or equivalent) digestion block.

7.3 SCP Science digestion vessels (or equivalent polypropylene vessels) with graduations at 10 mL, 25 mL and 50 mL.

7.4 Polypropylene ribbed watch glasses

8.0. REAGENTS:

8.1. Milli-Q filtered ultra-pure water.

8.2. Concentrated nitric acid (HNO_3), reagent grade.

8.3. Concentrated hydrochloric acid (HCl), reagent grade

8.4. Spiking solutions:

8.4.1. Low Level Matrix Spike 1: following table lists the concentration of each analyte in the spike solution.

Aluminum	10 µg/mL	Cobalt	2.5 µg/mL	Potassium	500 µg/mL
Arsenic	5 µg/mL	Copper	5 µg/mL	Selenium	5 µg/mL
Barium	5 µg/mL	Iron	100 µg/mL	Silver	1 µg/mL
Beryllium	2.5 µg/mL	Lead	1 µg/mL	Sodium	500 µg/mL
Bismuth	2.5 µg/mL	Magnesium	500 µg/mL	Thallium	0.5 µg/mL
Cadmium	2.5 µg/mL	Manganese	10 µg/mL	Vanadium	5 µg/mL
Calcium	500 µg/mL	Nickel	2.5 µg/mL	Zinc	10 µg/mL
Chromium	2.5 µg/mL				

8.4.2. Preparation of the Low Level Matrix Spike 1.

8.4.2.1. In a 100 mL Class A volumetric flask, add 5 mL of concentrated HNO_3. Add 10 mL of ICal custom standard (5000 µg/mL Ca, K, Mg, Na).

8.4.2.2. Add the following:

- 1 mL 1000 µg/mL Al stock solution,
- 1 mL 1000 µg/mL Mn stock solution,
- 1 mL 1000 µg/mL Zn stock solution,
- 0.5 mL 1000 µg/mL As stock solution,
- 0.5 mL 1000 µg/mL Cu stock solution,
- 0.5 mL 1000 µg/mL Se stock solution,
- 0.5 mL 1000 µg/mL V stock solution,
- 0.25 mL 1000 µg/mL Be stock solution,
- 0.25 mL 1000 µg/mL Bi stock solution,
- 0.25 mL 1000 µg/mL Cd stock solution,
- 0.25 mL 1000 µg/mL Co stock solution,
- 0.25 mL 1000 µg/mL Cr stock solution,
- 0.25 mL 1000 µg/mL Ni stock solution,
- 0.1 mL 1000 µg/mL Pb stock solution,
- 0.05 mL 1000 µg/mL Tl stock solution,
- 0.5 mL 1000 µg/mL Ba stock solution,
- 0.1 mL 1000 µg/mL Ag stock solution,
- 1 mL 10000 µg/mL Fe stock solution.

8.4.2.3. Dilute to 100 mL using deionized water and transfer to a 100 mL plastic Nalgene bottle that has been covered in tape.

8.4.3. Low Level Matrix Spike 2: following table lists the concentration of each analyte in the spike solution.

Antimony	1 µg/mL
Boron	10 µg/mL
Molybdenum	2.5 µg/mL
Silicon	500 µg/mL

Tin	2.5 µg/mL
Titanium	5 µg/mL
Phosphorus	100 µg/mL

8.4.4. Preparation of the Low Level Matrix Spike 2.

8.4.4.1. In a 100 mL Class A volumetric flask, add 5 mL of HNO_3.

8.4.4.2. Add the following:

5.0 mL 10000 µg/mL Si
0.1 ml 1000 µg/mL Sb
0.1ml 10000 µg/mL B
1.0 ml 10000 µg/mL P
0.025 ml 10000 µg/mL Mo
0.5 ml 1000 µg/mL Ti
0.025 ml 10000 µg/mL Sn

8.4.4.3. Dilute to 100 mL using deionized water and transfer to a 100 mL plastic Nalgene bottle covered in tape.

8.5. All reagents must be labeled using the criteria in SOP#112. Expiration dates for stock HCl and HNO_3 are 5 years from the date opened. The expiration dates for spike solutions are one year from the date made or the earliest expiration date from the stock standards used to make the spike solution, whichever is earlier.

9.0. EXTRACTION:

9.1 Label plastic digestion vessels with work order and sample number, including QC samples.

9.2 Rinse digestion vessels completely with Milli-Q water.

9.3 Adequately mix sample. Transfer 50 mL of sample to a labeled digestion vessel. If greater than 1/8-inch of sediment is present in bottle, contact Project Manager for guidance on how to proceed.

9.4 Method Blank (MB): Add 50 mL of Milli-Q water to a digestion vessel. One method blank is required for each batch of ≤ 20 samples.

9.5 Laboratory Control Sample (LCS): One LCS is required for each batch of ≤ 20 samples.

9.5.1 Add 50 mL of Milli-Q water to a digestion vessel.

9.5.2 Add 0.25 mL of Low Level Matrix Spike 1 (section 8.4.2.) and 0.25 mL of Low Level Spike 2 (section 8.4.4.).

9.6 Matrix Spike (MS): An MS is required at a 10% frequency. One MS is required for every ten samples.

9.6.1 Choose a random sample within the batch to be the MS. Homogenize sample by shaking and add 50-mL to a labeled digestion vessel.

9.6.2 Add 0.25 mL of Low Level Matrix Spike 1 (section 8.4.2) and 0.25 mL of Low Level Matrix Spike 2 (section 8.4.4).

9.7 Billable Bench Spike Digested (BBND): A client may specify a BBND on a particular sample. The sample will be issued two laboratory HSN numbers. The BBND is digested separately, but it is spiked at the bench. Add 50 mL of sample to two digestion vessels. The parent sample will have a lower sample number and the BBND will have a higher sample number.

9.8 Add 0.2 mL of HNO_3 and 0.1 mL of HCl to each digestion vessel. **NOTE: When spiking the LCS or MS samples, be sure to add the spiking solution *before* the addition of digest acids.**

9.9 Place all samples and quality control (MB, LCS, MS, and BBND) into the Hot Block, preheated to 95 ± 5°C. Allow sample volume to reduce to < **10 mL** (do not boil) for **6-7 hours**. Rinse the ribbed watch glasses with Milli-Q water and place on top of the digestion vessels. Reflux for 30 minutes. The temperature of the hot block is monitored using a thermometer. The thermometer is placed in a digestion vessel with a hole in the cap. The thermometer must be suspended above the bottom of the vessel and cannot touch the side of the walls. Any space in the hole of the cap should be sealed to ensure that the water does not evaporate.

The position of the thermometer must be placed in a different cell of the hot block each day of use in order to track the temperature of the entire hot block. A grid is used to identify the location of each cell in the hot block, where letters (i.e. A-I) read from left to right and numbers (i.e. 1-6) read from top to bottom, this is illustrated in figure 1 below.

Figure 1

	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
1	1A	1B	1C	1D	1E	1F	1G	1H	1I
2	2A	2B	2C	2D	2E	2F	2G	2H	2I
3	3A	3B	3C	3D	3E	3F	3G	3H	3I
4	4A	4B	4C	4D	4E	4F	4G	4H	4I
5	5A	5B	5C	5D	5E	5F	5G	5H	5I
6	6A	6B	6C	6D	6E	6F	6G	6H	6I

Figure 1. Hot block Grid

The temperature and position will be monitored and recorded in the electronic laboratory notebook for a different cell during each day of use. The thermometer will move from top to bottom and left to right (i.e. 1A→2A and 6A→1B).

9.10 Remove from Hot Block and allow the samples to cool. Rinse digestion vessel caps thoroughly with Milli-Q water. Dilute with Milli-Q water to 10 mL and cap the digestion vessels with the rinsed caps.

9.11 Post, label and give to analyst.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1 A laboratory method blank (MB) must be prepared with each batch of 20 samples or less.

12.2 A laboratory control sample (LCS) must be prepared with each batch of no more than 20 samples. See section 9.5 for the preparation procedure.

12.3 Matrix spike (MS) samples must be prepared at a 10% interval.

12.4 Billable Bench Spike Digested (BBND) is only required when specified by the client.

13.0. CALCULATIONS, REVIEW AND REPORTING:

N/A

14.0. HEALTH AND SAFETY:

14.1. The reagent solutions and chemicals used during this procedure have varying degrees of toxicity. The material safety data sheet for each chemical is located in the quality control room. Before performing any part of the procedure, the pertinent SDS should be read.

14.2. Personal protective equipment (PPE) must be worn during handling of samples, reagents, and spiking solutions. The PPE worn should include a laboratory coat, eye protection (goggles) and gloves. In addition, when handling concentrated acid or base preservative, a face shield and apron must be worn.

14.3. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.4. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized per SGS SOP#134. Elementary neutralization may be performed at the bench or using the Elementary Neutralization Hood located in GC Prep. The Sample Custodian may accomplish performance of this step.

14.5. All surplus reagent acids shall be neutralized on a daily basis. Elementary neutralization may be performed at the bench or using the Elementary Neutralization Hood located in GC Prep.

14.6. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session. This may be performed in conjunction with elementary neutralization.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

17.1 Detection Limit (DL) studies are performed annually for each EPA 200.8 analyst, when a new operator is trained for drinking water analyses, when a significant change in instrument response is observed, or when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this

method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

18.0. LIMIT OF DETECTION (LOD):

The LOD is as defined in the current version of SGS SOP#116.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is as defined in the current version of SGS SOP#116.

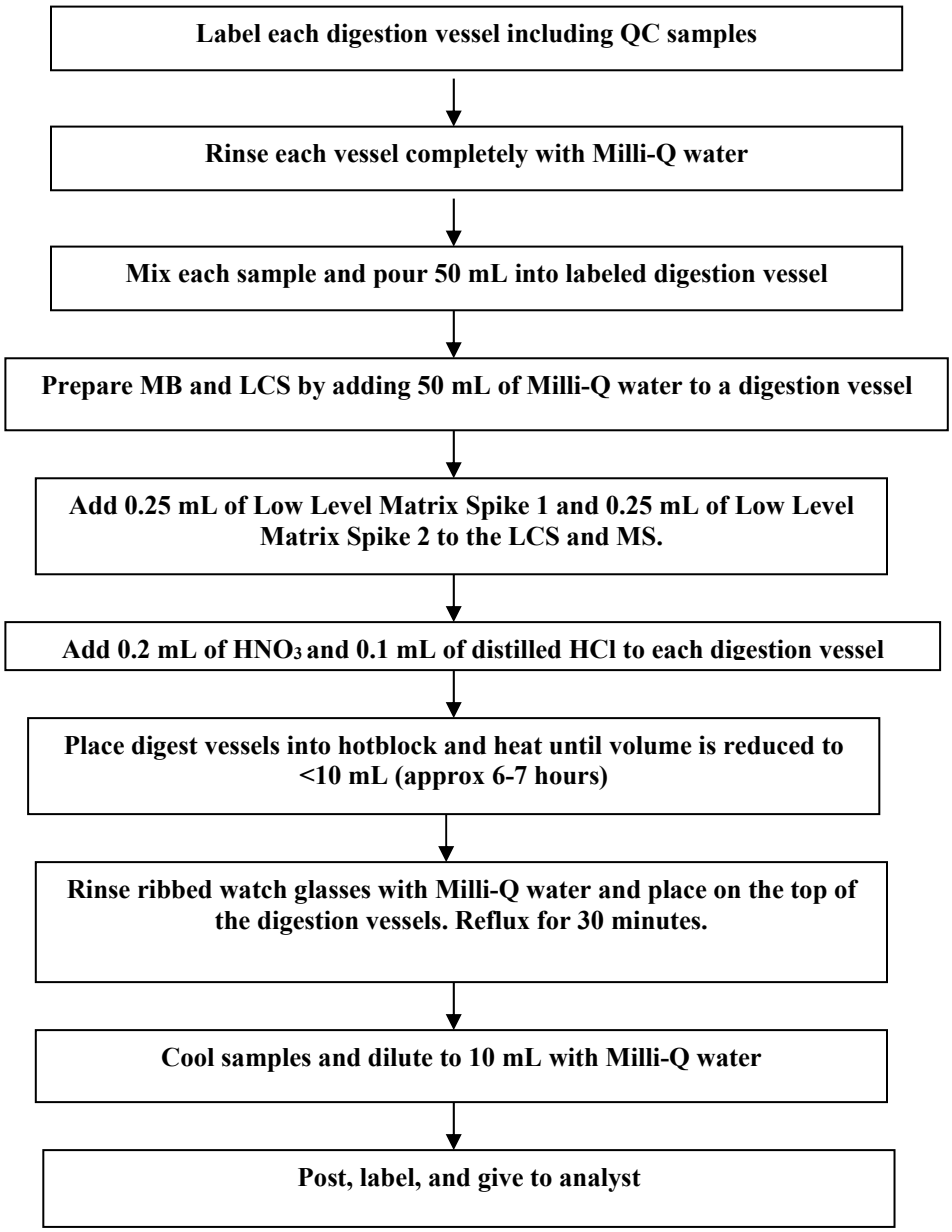
20.0. REFERENCES:

EPA 600/R-94-111, Method 200.2, May 1994, and SOP 367.

21.0. ATTACHMENTS:

Attachment A: Flow Chart

Attachment A: FLOW CHART



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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Determination of Metals in Drinking Water and Wastewater by ICP-MS (Low Level Method)	**Revision Date**: September 2023
Method No: EPA 200.8 (Low Level)	**SOP No**: 385r10
Page: 1 of 25	**Supersedes:** 385r09

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/15/23	Samara Rentty	9/15/23

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This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- Updated section 8.12 to reference the correct section.
- Updated section 12.5, 12.13, and Attachment A from “10 times” to “5 times” contamination level for MB and CB to reflect current practices.
- Updated Attachment C - Element Isotopes for Monitoring Only to include Krypton 83 and Palladium 105

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1.0. OBJECTIVE:

This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples according to EPA 200.8[1] using the NexION 300 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) or Agilent 7800 ICP-MS.

2.0. SCOPE AND APPLICATION:

2.1 This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples according to U.S. EPA Method 200.8 Rev 5.4, 1994.

2.2 This method is applicable to ground waters, surface waters, and drinking waters.

2.3 Routine operation and maintenance procedures for the NexION 300 ICP-MS may be found in the ICP-MS Training Manual for the 300 provided by the instrument manufacturer.

2.4 Routine operation and maintenance procedure for the Agilent 7800 ICP-MS may be found in the training provided by the instrument manufacturer.

2.5 Detailed information regarding requirements by U.S. EPA Method 200.8 may be found in version 5.4 of Method 200.8.

2.6 This SOP is designed for use with EPA Method 200.8 for the NexION 300 and Agilent 7800 instruments.

2.7 Aqueous sample, digestates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into an RF plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that house a quadrapole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector.

2.8 Initial Performance Data: It is the responsibility of the user of this SOP to generate the method specific performance data on the user's specific instrument before the analysis of any samples.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. Standards are matrix matched at 2% nitric acid 0.4% hydrochloric acid as opposed to 1% nitric acid as stated in Method 200.8.

3.2. Correction equations for polyatomic interferences are those recommended in Table 5 of Method 200.8 and are not established daily. For elements requiring correction equations that are not listed in Method 200.8, equations from the instrument manufacturer are used.

3.3. Internal standards typically used are Sc, Ge, In, and Ir as opposed to the mix of Sc, Y, In, Tb, and Bi as stated in 200.8.

3.4. Method 200.8 states in section 3.5 that the IDL is three times the standard deviation of a series of ten replicate measurements of a calibration blank. This laboratory is currently using the SW846 protocol to determine the IDL so that the IDL can be utilized for both methods. SW 846 criteria are more detailed and must meet more stringent criteria (see section 11.1.1. of this SOP).

3.5. The NexION 300 instrument is tuned to Be, Mg, In, U, and **Ce** rather than to Be, Mg, Co, In, and Pb as stated in Method. The Agilent 7800 instrument does not deviate from the EPA 200.8 Method.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1 Isobaric Interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98). Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interferences are already present as default interference equations in the NexION and Agilent software.

Note: Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

5.2 Molecular interferences are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.) Predictions about the type of molecular interferences may be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $Ar^{40}Cl^{35}$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($Ar^{40}Cl^{37}$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl^{37} to Cl^{35}.

5.3 This instrument method contains correction equations to compensate for the above-mentioned interferences. All samples analyzed by this method are subject to correction, the extent of which is based on sample

matrix. It is the responsibility of the analyst to identify possible matrix problems and produce data of known quality. All data users should be aware that these corrections are possible without explicit notification.

5.4 This method is suitable for the preparation of samples containing < 0.1 mg/L silver. For samples containing silver at concentrations > 0.1 mg/L, notify the supervisor or project manager. A sample may need to be prepared a second time or diluted in an approved manner.

6.0. SAMPLE HANDLING:

Refer to SOP 367 & 375 (Total and Dissolved Metals Preparation and Low-Level Metals Preparation, respectively) for sample preservation. Digests are stored for one month prior to disposal. Refer to SOP 108 for disposal guidance.

7.0. APPARATUS:

7.1 NexION 300D

7.1.1 Perkin-Elmer NexION 300 ICP-MS system or equivalent, includes the NexION 300 instrument, computer system, ELAN NT software, printer, and auto sampler. Instrument capable of scanning the mass range 5-250 amu with a minimum resolution of 1 amu peak width at 5% peak weight.

7.1.2 SC-2 Autosampler (ESI no. SC-8102-1341-54) or equivalent.

7.1.3 Peristaltic pump tubing:

Tab colors	I.D. mm	Purpose
Black – Black	0.75	Sample introduction
Green – Orange	0.51	Internal standard introduction
Grey – Grey	1.3	Spray chamber drain

7.2 Agilent 7800 ICP-MS

7.2.1 Agilent 7800 ICP-MS includes the instrument, computer system, software, and autosampler. Instrument capable of scanning the mass range 5-250 amu with a minimum resolution of 1 amu peak width at 5% peak weight.

7.2.2 Recommended peristaltic pump tubing (other sizes may be used to enhance performance or reduce pulsations produced by the pump):

7.2.2.1 White/White -1.02 mm i.d. (for carrier and tuning solution).

7.2.2.2 Yellow/Blue - 1.52 mm i.d. (for drain).

7.3 Instrument maintenance logs must be maintained per SGS SOP 111 and 500.

7.4 Calibrated mechanical pipettes: 20-200 µL, 100-1000 µL, 1000-5000 µL

7.5 Metal-free plastic pipette tips (for the pipettes specified in 7.4).

7.6 14.0 mL disposable Autosampler tubes. (VWR # 60818-614 or equivalent)

7.7 50-mL freestanding graduated digestion vessels for standards (VWR # 21008-480 or equivalent).

7.7.1 100-mL freestanding graduated digestion vessels are used for the Agilent instrument.

7.8 Class A volumetric labware for the preparation of calibration standards.

7.9 Argon gas: High purity grade (99.99%)

8.0. REAGENTS:

8.1. All stock solutions/chemicals/reagents must be traceable back to documented written records. Refer to SOP 500 and SOP 112 for proper documentation procedures. The expiration dates of all reagents must be recorded and written on the container. Where applicable, this information must be entered into the LIMS system.

8.2. Nitric acid, concentrated. (JT Baker "Instra-Analyzed" # 9598-34 or equivalent)

8.3. Hydrochloric acid, (JT Baker "Instra-Analyzed" # 9530-33) or equivalent

8.4. Reagent water equivalent to ASTM Type I water (ASTM D 1193).

8.5. Single element or multi-element stock solutions of the following elements as needed for Standards, Tuning Solutions, and Performance Solutions.

Li, Be, B, Al, Sc, V, Cr, Mn, Co, Ni, Cu, Zn, Ge, As, Se, Au, Hg, Sr, Mo, Ag,
Cd, In, Sn, Sb, Ba, Ir, Tl, Pb, Bi, Th, U, Na, Mg, Si, P, K, Ca, Ti, Fe, Ce.

8.6. Agilent Stock Tuning Solution (Agilent #5184-3566): 10ug/L Li, Y, Ce, Tl, and Co in 2% HNO3.

8.6.1. Agilent Daily Start-up Configuration Solution: Prepare by pipetting 5mL of Agilent Stock Tuning Solution with approximately 20mL of reagent water and 1.0 mL concentrated nitric acid. Dilute to 50mL and mix well.

8.7. NexIon Stock Solutions:

8.7.1. Intermediate FE Solution: Add 2.5mL of concentrated nitric acid to approximately 20 mL of reagent water in a 50 mL digestion vessel. Pipette the appropriate amount of Fe stock solution. Dilute to 50 mL with reagent water. Refer to CX-0019 for the current preparation.

8.7.2. NexIon Daily Tuning Stock Solution: Add 5mL concentrated nitric acid to approximately 30 mL of reagent water in a 100 mL Teflon volumetric flask. Prepare as per CX-0019.

8.8. Daily Tuning solution:

8.8.1. For NexIon: Prepare by pipetting 50 µL of NexIon Daily Tuning Stock Solution (Section 8.7.2.) into a 50-mL digestion vessel filled with approximately 20 mL of reagent water and 1.0 mL of concentrated nitric acid. Dilute to 50 mL with reagent water and mix well.

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8.8.2. For Agilent 7800: prepare by adding 500µL of the NexIon Daily Tuning Stock Solution (section 8.7.2), which includes Co, to a 50-mL digestion vessel filled with approximately 20-mL of reagent water and 1 mL of nitric acid. Dilute to 50 mL with reagent water and mix well.

8.9. Internal Standard Solution containing Sc, In, Ge, and Ir.

Prepare by pipetting the following elements at listed volumes to a 4-liter container with approximately 1,000 mL of reagent water, 40 mL of nitric acid and 4 mL of a 10,000 µg/mL gold standard for a final concentration of 10 µg/mL gold. Dilute to 4 liters with reagent water.

Element	Stock conc. (µg/mL)	Vol. (mL)
Sc	1000	7.5
In	1000	0.5
Ge	10,000	0.4
Ir	1000	1

8.10. Stock calibration standard solutions: While this SOP gives directions for making specific concentrations, the analyst may, at times, be directed to prepare alternate concentrations for "fit of purpose." Standards may be prepared from single element solutions or a custom blend solution that has been purchased. Charts are provided below for preparing each of the stock solutions from single element standards. All solutions for each stock standard are pipetted into a 100 ml class A Teflon volumetric flask with 30ml of reagent water and 5ml nitric acid. After all elements for each solution have been added, fill the flask to the mark with reagent water.

200.8LL Cal 1

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
As	10,000	0.1	Mn	10,000	0.2
Be	10,000	0.05	Ni	10,000	0.05
Bi	10,000	0.05	Pb	10,000	0.02
Cd	10,000	0.05	Se	10,000	0.1
Co	10,000	0.05	U	1000	0.2
Cr	10,000	0.05	V	10,000	0.1
Cu	10,000	0.1	Zn	10,000	0.2
Li	10,000	0.04	Th	10,000	0.05

200.8LL Cal 2

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Mo	10,000	0.05	Si	10,000	5.0
B	10,000	0.2	Sn	10,000	0.05
Sb	10.000	0.02	Ti	10,000	0.1

200.8LL Cal 3

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Ag	1000	0.2	Tl	1000	0.1
Ba	10,000	0.1			

200.8LL Cal 4

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Al	10,000	0.2	K	10,000	5.0
Ca	10,000	5.0	Mg	10,000	5.0
Fe	10,000	2.0	Na	10,000	5.0
Sr	10,000	0.5			

8.11. Quality Control Sample (QCS) stock - the QCS stock MUST BE from a source independent of that used for calibration standards. The concentrations of the analytes in the QCS are prepared near the mid-point of the calibration range and a concentration not used for calibration.

200.8LL QCS 1

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
As	1,000	0.5	Mn	1,000	1.0
Be	1,000	0.25	Ni	1,000	0.25
Bi	1,000	0.25	Pb	1,000	0.1
Cd	1,000	0.25	Se	1,000	0.5
Co	1,000	0.25	U	1,000	0.1
Cr	1,000	0.25	V	1,000	1.0
Cu	1,000	0.5	Zn	1,000	1.0
Li	1,000	0.2	Th	1,000	0.25

200.8LL QCS 2

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Mo	1,000	0.25	Si	10,000	2.5
B	1,000	1.0	Sn	1,000	0.25
Sb	1.000	0.1	Ti	1,000	0.5

200.8LL QCS 3

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Ag	1,000	0.1	Tl	1,000	0.05
Ba	1,000	0.5			

200.8LL QCS 4

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Al	1,000	1.0	K	5,000	5.0
Ca	5,000	5.0	Mg	5,000	5.0
Fe	10,000	1.0	Na	5,000	5.0
Sr	1,000	2.5			

8.12. Post Digestion Spike (BND): Pipette 50 µL of each QCS 1,2,3,4 stock (8.11), 2 mL of original sample and 2.8 mL sample diluent (8.17) for a final volume of 5.0 mL. Remember to match the sample dilution of the original sample.

8.13. Low Level Cd/Se Spike (BBND) stock:

8.13.1. Intermediate BBND stock: Prepare by adding 2 mL concentrated nitric acid to a 100 mL class A PFA volumetric flask. Pipette 0.05 mL of 1,000 mg/L single element Cd and 2.5 mL of 1000 mg/L single element Se and bring up to volume with reagent water.

8.13.2. Working BBND stock: Prepare by adding 1.0 mL concentrated nitric acid to a 50 mL class A PFA volumetric flask. Pipette 0.5 mL of Intermediate BBND stock (8.12.1) and bring up to volume with reagent water.

8.14. Low Level BBND: Pipette 0.2 mL of Working BBND stock (8.12.2), 2 mL of original sample and 2.8 mL of sample diluent (8.17) for a final volume of 5.0 mL. Remember to match the sample dilution of the original sample.

8.15. Working Calibration

8.15.1. Working calibration scheme for **Agilent**: Prepare fresh calibration standards daily in Class A volumetric labware as is appropriate for the analysis. Prepare the calibration standards according to the chart below.

A/S Position	Solution	Ingredients	Acids	Final vol.
1	Blank	Reagent Water	2.0mL HNO_3/400µL HCl	100mL
5	Standard 4	1.0mL each of Cal 1, 2, 3, 4	2.0mL HNO3/400µL HCl	100mL
4	Standard 3	10mL of Standard 4	1.8mL HNO_3/360µL HCl	100mL
3	Standard 2	10mL of Standard 3	1.8mL HNO3/360µL HCl	100mL
2	Standard 1	10mL of Standard 2	1.8mL HNO3/360µL HCl	100mL
5	ICV/CCV	5X auto dilution of Standard 4	N/A	N/A
9	QCS	1.0 ml each of stock QCS 1, 2, 3, 4	2.0mL HNO3/400µL HCl	100mL

8.15.2. Working calibration scheme for **NexIon**: Prepare fresh calibration standards daily in Class A volumetric labware as is appropriate for the analysis. Prepare the calibration standards according to the chart below.

A/S Position	Solution	Ingredients	Acids	Final vol. mL
1	Blank	Reagent water	1.0 mL HNO_3 /200µL HCl	50
6	Standard 4	500 µL each of Cal 1,2,3,4	1.0 mLHNO_3 /200µL HCl	50
5	Standard 3	5 mL of Standard 4	0.9 mL HNO_3 /180µL HCl	50
4	Standard 2	5 mL of Standard 3	0.9 mL HNO_3 /180µL HCl	50
3	Standard 1	5 mL of Standard 2	0.9 mL HNO_3 /180µL HCl	50
7	ICV / CCV	100 µL each of stock Cal 1,2,3,4	1.0 mL HNO_3 /200µL HCl	50
8	QCS	500 µL each of stock QCS 1,2,3,4	1.0 mL HNO_3 /200µL HCl	50

8.16. Dual Detector Solution is for normalization between the pulse and analog stages of the detector. Fill a 1-L Nalgene bottle with approximately 400 ml of reagent water. Add 20 ml of concentrated HNO_3. Pipette the following single element standards into the bottle and dilute to 1 L with reagent water.

Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml	Element	Stock conc mg/L	Vol. ml
Al	1000	0.25	Fe	10,000	0.5	Sb	1000	0.25
Ag	1000	0.1	Ge	10,000	0.1	Sc	1000	0.4

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As	10,000	0.15	In	1000	0.1	Se	10,000	1.5
B	10,000	0.3	K	1000	0.2	Si	10,000	0.1
Ba	1000	0.5	Mg	1000	0.3	Sn	1000	0.3
Be	10,000	0.3	Mn	1000	0.15	Th	1000	0.1
Bi	1000	0.05	Mo	1000	0.75	Ti	1000	0.3
Cd	1000	0.4	Na	1000	0.25	Tl	1000	0.075
Co	1000	0.2	Ni	1000	0.5	V	1000	0.2
Cr	1000	0.25	P	10,000	0.75	Zn	10,000	0.2
Cu	1000	0.5	Pb	1000	0.1	U	1000	0.05

8.17. Sample Diluent: 2% (v/v) nitric acid. Volumes may be made as necessary at a ratio of 20.0 mL of nitric acid per 1000 mL of reagent water.

8.18. Rinse: containing 4.0% (v/v) hydrochloric acid, 2.0% (v/v) nitric acid and 1 µg/mL Au. Volumes may be made as necessary at a ratio of 80.0 mL of HNO_3, 160.0 mL of HCL, and 0.4 mL of 10,000 µg/mL Au per 4,000 mL of reagent water.

9.0. EXTRACTION:

Refer to SOP 375 (Total Recoverable Metals Low Level Preparation) for sample preparation.

10.0. CALIBRATION:

10.1 The instrument must be calibrated before analysis of any samples. Multipoint calibration (default calibration method). The instrument must be calibrated before analysis of any samples with a blank and four calibration standards. If less than four calibration standards are used, refer to the SGS SOP 500 for the criteria used in dropping calibration points. **Note:** Perkin-Elmer's software documentation refers to "blank subtraction;" however, this is actually a zeroing of the instrument to the blank response before calibration is initiated. No blank subtraction is performed following calibration. The manufacturer describes the calibration technique as "force through zero." This technique has been shown to be linear at the blank response. Weighted calibration is allowed with approval of the QA for select elements.

10.2 The concentrations of the standards (ppb) used should be entered into the calibration page of the analytical method in the instrument software according to the following values:

Element	Std #1	Std #2	Std #3	Std #4
Be	0.0500	0.500	5.000	50.00
B		2.000	20.00	200.0
Al		2.000	20.00	200.0
V		1.000	10.00	100.0
Cr		0.500	5.000	50.00
Mn	0.2000	2.000	20.00	200.0
Co	0.0500	0.500	5.000	50.00
Ni		0.500	5.000	50.00
Cu	0.1000	1.000	10.00	100.0
Zn		2.000	20.00	200.0
As		1.000	10.00	100.0
Se		1.000	10.00	100.0
Sr	1.000	10.00	100.0	1000
Mo	0.0500	0.500	5.000	50.00

Ag	0.020	0.200	2.000	20.00
Cd	0.0500	0.500	5.000	50.00
Sn		0.500	5.000	50.00
Sb		0.200	2.000	20.00
Ba	0.1000	1.000	10.00	100.0
Tl	0.010	0.100	1.000	10.00
Pb	0.020	0.200	2.000	20.00
U	0.020	0.200	2.000	20.00
Th	0.050	0.500	5.000	50.00
Bi	0.0500	0.500	5.000	50.00
Na		50.00	500.0	5000
Mg		50.00	500.0	5000
Si		50.00	500.0	5000
K		50.00	500.0	5000
Ca		50.00	500.0	5000
Fe		20.00	200.0	2000
Li		0.400	4.000	40.00
Ti		1.000	10.00	100.0

10.3 The first standard run should be the lowest level standard containing analyte and be equal to or less than the LOQ, followed by standards of increasing concentration in order to minimize cross-contamination and carryover.

10.4 The ICP-MS software labels the calibration solution as follows: Blank, Standard 1, Standard 2, Standard 3, and Standard 4.

10.5 The R-value (linear regression correlation coefficient) for each element must be greater than or equal to 0.995.

10.6 The average of three integrations, for calibration, instrument QC, preparation QC, and samples, will be used.

11.0. ANALYSIS:

11.1 Initiate the plasma and allow a warm-up of at least 30-60 minutes. The tuning procedures may be carried out during warm-up. (When turning on Agilent 7800 ICP-MS, transfer the carrier probe to the Start-up configuration solution right away. The instrument will automatically run its start-up configuration process once the required plasma temperature is met).

11.2 Before calibration and analysis of samples, the instrument must undergo a series of performance checks to ensure that the instrument is operating properly.

11.2.1 For the NexIon 300D

11.2.1.1 Open the “SGS SmartTune Daily.wrk” workspace.

11.2.1.2 While using the Dual Detector Solution (Section 8.16), select the “Dual Detector Calibration” tab, right click and select “Quick Optimize”. When calibration is complete, open the interactive window, review the graph and print it to archive with the tune package. Run twice. It is a good practice to do the dual detector calibration every

day, but it only needs to be done once a week or whenever detector voltages are changed.

11.2.1.3 Switch to the Daily Tune Solution (Section 8.8.1) after rinsing the lines. Select the "Mass Calibration and Resolution" tab, right click, and select "Quick Optimize". The software will automatically check the limits, make adjustments, and rerun if there is a failure. After each run the results will print out.

11.2.1.4 The measured mass for each analyte must be +/- .05 AMU of the exact mass.

11.2.1.5 The measured peak width must be 0.7 amu, +/- 0.1 amu at 10% peak height (per Perkin Elmer recommendations). EPA 200.8 states 0.75 amu at 5% peak height. The peak width of 10% at 0.7 amu will be less than 5% at 0.75 amu. The decrease in peak width is accounted for by the increase in resolution from 0.75 to 0.7 amu.

11.2.1.6 Select the "Autolens" tab, right click and select "Quick Optimize". When calibration is complete, open the interactive window, review the graph, and print it to archive with the tune package.

11.2.1.7 Select the "Daily Performance Check" tab, right click, and select "Quick Optimize". The software will automatically check the limits except for the RSD. Make adjustments and rerun if there is a failure. After each run the results will print out.

11.2.1.8 The RSDs for the five replicates of Be, Mg, In and U need to be less than or equal to 5%.

11.2.1.9 Monitor daily performance measures for Be sensitivity, Mg sensitivity, In sensitivity, U sensitivity, background, % double charged and % oxide levels.

Background at mass 220	< 5 cps
Ce++ double charged	≤3%
CeO/Ce Oxide	≤2.5%
Be intensity	> 2,000 CPS
Mg intensity	> 15,000 CPS
In intensity	> 40,000 CPS
U intensity	>30,000 CPS

NOTE: These performance requirements must be achieved before any analysis is performed. Oxides and double charged levels can be adjusted by slightly varying the nebulizer flow rate. **This performance report must be printed and submitted with each analytical batch.

11.2.2 For Agilent 7800-ICP-MS:

11.2.2.1 Open ICP-MS Instrument Control (Online ICP-MS Mass Hunter) software. Click the "Batch" dropdown and select 200.8LL.icpms.template. Rename the folder using the date (MM.DD.YYYY.P7). Click the "Batch" icon, Acq Method, Tune, and select Start Auto Tune. Monitor the sensitivity and Resolution/Axis of Be, Mg, Co, In, and Pb

Analyte and Mass	Sensitivity RSD%	Axis	W-5%
Be, 9	<5.000%	8.90-9.10	<0.9000%
Mg, 24	<5.000%	23.90-24.10	<0.9000%

Co, 59	<5.000%	58.90-59.10	<0.9000%
In, 115	<5.000%	114.90-115.10	<0.9000%
Pb, 208	<5.000%	207-90-208.10	<0.9000%

11.3 Set up the analytical run.

11.3.1 For NexIon 300D

11.3.1.1 Open the "SGS 200.8 Low Level.wrk" workspace that contains the method and sample worksheet for analysis.

11.3.1.2 Calibration standards and QC solution prepared in Section 8.15 are to be placed into their assigned autosampler (A/S) positions.

11.3.1.3 Edit the Sample window under the batch analysis to update the schedule with new sample information and autosampler locations. Ensure that the proper dilution factor is entered for each sample. Save the sample window with the date of analysis.

11.3.1.4 Pour all digested samples into 14 mL test tubes and place in the appropriate autosampler position. Further dilutions can be made if the sample exhibits a difficult matrix or has proven to exceed the linear range for an element of interest.

11.3.1.5 The choice can be made to automate CCV/CB rates at every 10 samples analyzed or specify CCV/CB analysis at particular times during the run (not to exceed an interval of 10 samples) within the method under the QC tab / QC Frequency sub-tab. The default is automated at 10 samples.

11.3.1.6 Select the samples to be analyzed in the sample window by highlighting the rows to be analyzed. Make sure that row 1 containing the calibration command is highlighted or that calibration is performed prior to the analysis of any samples.

11.3.1.7 Press the Analyze Batch button.

11.3.2 For Agilent ICP-MS.

11.3.2.1 Click "Batch".

11.3.2.2 Calibration standards and QC solutions prepared in Section 8.15 are to be placed into their assigned autosampler (A/S) positions.

11.3.2.3 Check "Calibration Standards" and "Check Samples 1" to make sure there are no "Skip" boxes checked. Click "Add to Queue". (This step is meant to run the calibration and instrument QC first before entering sample information.) A new window, Online ICP-MS Data Analysis, will pop out once the calibration begins.

11.3.2.4 Once the calibration is done and instrument QCs meet the criteria, go back to "Online ICP-MS MassHunter", click "Batch" icon and "Start Editing Mode". Click "Unknown Samples" and enter the necessary sample information. If prepFAST dilution is utilized, enter the appropriate dilution under "prepFAST Dilution". The "Total Dil." column will automatically be filled out. If bench dilution is done, the "prepFAST Dilution" and "Total Dil." columns will remain as 1.00. Add the bench dilution in the "Comment"

section. Go to "Check Samples 2" and uncheck the "Skip" boxes for CCV and CB. Save the batch information and Click "Add to Queue" to start the analysis.

11.4 All samples containing target analytes above linear range shall be diluted and re-analyzed.

11.5 At the end of the analytical run, print out the following Calibration summary and instrument runlog and include them with the raw data.

11.5.1 For Nexion: Open the "report option window."

11.5.1.1 Choose "CAL REPORT.rop" file. Print the report view.

11.5.1.2 Open "RUN-COVER.rop" file. Print the report view.

11.5.1.3 Click back on the "report option window."

11.5.1.4 Open "SGS.rop" file to reset.

11.5.2 For Agilent 7800: Go back to "Online ICP-MS Data Analysis" window.

11.5.2.1 Click the "Report" dropdown and choose "Quick Batch Report". A new window, containing the instrument runlog, will appear. Click "Print" to save a PDF copy.

11.5.2.2 Right Click any graph in the "Calibration Curve" section. Choose "Print Calibration" and "Simple". A new window, containing the calibration summary, will appear. Click "Print" to save a PDF copy.

12.0. QUALITY CONTROL:

12.1. Initial Demonstration of Laboratory Performance - The following items must be completed before the analysis of any samples is performed by each laboratory using this method.

12.1.1. Instrument Detection Limits (IDLs) - IDLs must be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and or/ at a frequency designated by the project. IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. This procedure states that the IDLs be estimated by calculating the sum of the standard deviations of the three runs on three nonconsecutive days from the analysis of a reagent blank solution with seven consecutive measurements. Each measurement must be performed as though it was a separate sample (i.e., with rinsing in between). The IDL must be less than or equal to the DL. Refer to SOP 500 for further guidance.

12.1.2. Detection Limits (DLs) - In order to determine the DL in a matrix, the analytes should be spiked into the matrix of interest (e.g. 200.2 sample preparation blank, ground water matrix, or other sample preparation procedure matrix) at a level that is two to five times the estimated DL. The spiked matrix is then carried through the entire sample preparation procedure. Each replicate result must be calculated the same as if it were a final client sample result. The DL is calculated by multiplying the standard deviation obtained from a minimum of seven replicates by the one-sided 99% confidence level t-statistic. Refer to SOP 116 for further guidance.

12.2. Linear Dynamic Ranges

12.2.1 As defined by EPA Method 200.8, the linear range "…should be an observed signal no more than 10% below the level extrapolated from lower standards." Analyte concentrations that are greater than 90% of the determined Linear Range must be diluted and reanalyzed.

12.2.2 Calibrate the instrument, as described in Sections 9 and 10 and run a series of standards that increase in concentration until the measured value deviates 10% from the expected value. The upper limit is determined to be the value of the highest standard that is within 10% of the expected value.

NOTE: The Linear Dynamic Range should be re-evaluated on the following basis:

- whenever new PA tube is installed in the RF generator.
- upon any significant change to the instrument (i.e. new detector or alternate sample introduction system)

12.3 Initial Calibration Verification (ICV): the ICV is required after calibration and before any sample analysis. The ICV must be within ± 10% of the expected value. If the 10% criteria is not met, the ICV may be analyzed a second time. If the ICV is still outside of acceptance criteria, the cause of the error must be determined, and a recalibration must be performed.

12.4 Quality Control Sample (QCS): the QCS is analyzed immediately following the ICV to verify the accuracy of the calibration curve. The QCS is prepared from a source that is independent of the calibration standards. It is at a concentration not used for calibration, at or near the mid-point of the calibration curve. All analytes of interest must be within 10% of the expected value. If the 10% criteria is not met, the QCS may be analyzed a second time. If the QCS is still outside of acceptance criteria, the cause of the error must be determined, and a recalibration must be performed.

12.5 Continuing Calibration Blank (CB): the CB is analyzed following QCS and CCV samples. In the CB all analytes of interest should be below the established LOQ. If the CB cannot be brought below the LOQ for the analytes of interest, there are several options. If an analyte is detected in the CB but is not detected in the sample, or if the analyte is greater than five times the level detected in the CB, the data can be reported with appropriate comments. All samples that do not meet those criteria must be reanalyzed after the instrument has been recalibrated. If a CB failure occurs consistently with one or more elements, then a re-evaluation of the IDL and DL for those analytes may be required.

12.6 Continuing Calibration Verification (CCV): the CCV is analyzed at a rate of not greater than every 10 samples and at the end of an analytical batch. All analytes of interest must be within 15% of the expected results. If the 15% criteria is not met, the CCV may be analyzed a second time. If the CCV is still outside of acceptance criteria, the cause of the error must be determined, and a recalibration must be performed.

12.7 Internal Standards: the internal standards must be monitored in all solutions to correct for instrumental drift. The NexION software will report the % recovery for each internal standard being used. The intensities of internal standards in all solutions analyzed must be within 60-125% of the levels in the blank used for calibration.

12.7.1 Samples of significantly different matrix may cause the internal standards to fall outside tolerance levels. These samples may be serially diluted by additional factors of 5 until the internal standards fall within limits.

12.7.2 Persistent failure of internal standards indicates that the instrument has drifted outside of acceptable limits relative to the initial calibration and requires the instrument to be recalibrated and to re-analyze all affected samples.

12.8 Matrix Spike (MS): the MS is prepared and analyzed to determine the accuracy of the analytical system. At least 10% of the samples within an analytical batch must be spiked with known concentrations of all analytes of interest and evaluated for spike recovery. Samples are spiked at a level equal to that of the LCS. Spike recoveries must be between 70% and 130% for all analytes of interest in which the sample concentration is not greater than 4x the spike concentration. If any analyte does not meet recovery criteria in the matrix spike but that analyte is in control in the laboratory control sample, the spike recovery in the sample is judged to be matrix induced and data should be flagged as such.

12.9 Matrix Spike Duplicate (MSD): the MSD is performed only on a client request basis and is used to gauge the precision of the analytical system. Spike recoveries must be between 70% and 130% for all analytes of interest in which the sample concentration is not greater than 4x the spike concentration. The relative percent difference (RPD) between the MS and MSD must be 20 or less. A sample with a MS/MSD RPD outside 20 should be flagged appropriately.

12.10 Post Digestion Spike (BND): the BND is performed only on spiked samples that do not meet MS evaluation criteria. An aliquot of digested sample at standard or inflated dilution is spiked with a known concentration of analyte. The concentration of spike should be relative to the naturally occurring concentration in the sample. Recoveries calculated on the BND should be between 70% and 130% of the expected values. If the spike is not recovered within the acceptance limits and the lab performance for that analyte is in control, then the recovery problem is judged matrix related. The data will be flagged as suspect.

12.11 Low Level Cd/Se Spike (BBND): the BBND is performed only on a client request basis and is used as a cadmium and selenium low level check. An aliquot of digested sample at standard or inflated dilution is spiked with a known concentration of analyte. Recoveries calculated on the BBND should be between 50% and 150% of the expected values. If the spike is not recovered within the acceptance limits and the lab performance for that analyte is in control, then the recovery problem is judged matrix related and the data will be flagged as suspect.

12.12 Laboratory Control Sample (LCS): the LCS is reagent water that has been spiked with known concentrations of analytes which is processed and analyzed identically to unknown samples to assess system accuracy. One LCS will be prepared and analyzed for each sample batch of 20 samples or less. Results for the LCS must fall within 15% of expected concentrations for all analytes of interest. If an analyte of interest in the LCS is greater than 115% of the expected value and the associated sample results for that analyte are below the LOQ, then those samples may be reported with appropriate qualifiers. If an analyte of interest in the LCS is less than 85% of the expected value, then those samples affected must be re-processed through the digestion procedure.

12.13 Method Blank (MB): the MB is analyte-free reagent water that has been processed and analyzed identically to an unknown sample to assess systematic contamination. One MB will be prepared and analyzed for each sample batch of 20 samples or less. Results for analytes of interest in the MB must be less than LOQ values. If results for analytes of interest exceed the LOQ, then all associated samples must be re-processed through the digestion procedure unless the associated samples contain concentrations of analyte greater than 5x the MB contamination. All samples reported with MB contamination above the LOQ must be flagged with appropriate qualifiers.

12.14 See Section 21 for specific corrective actions. Any corrective action needed to address a QC outlier that is not listed in this SOP requires the written approval of the QA Manager or Technical Director.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1. The ICP-MS software performs all calculations necessary to convert raw data (ion counts/second). The calculated quantities are selected by choosing the desired options in the Report Options screen. The default report option for the NexION 300 200.8 Method is CTE1.rop. If the user desires, this format can be edited and saved under a new name.

13.2. All calculations performed in the ICP-MS software are based on the ratio of the analyte intensity (cps) to the internal standard intensity (cps). In all calculations where internal standards are used the ratio of the analyte intensity to internal standard intensity is taken before any other calculation is performed.

13.3. Quality Control Sample results may be checked using the QC Checking features in the ICP-MS software. All values entered in the default ICP-MS Method 200.8 should be checked and edited to match the true values used by the laboratory. The modified method can then be saved under a different name.

13.4. The recoveries for standard solutions (both digested standards and the periodic quality control standards) are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{Vo}{Ve} \times 100 = \% \text{ recovery} \qquad \text{EQUATION 1}$$

Vo = the observed value
Ve = the expected value

13.5. The calculation for spike recoveries requires the subtraction of the sample contribution from the response of the spiked sample, the division of this result by the expected value of the spike, and the multiplication by 100 yields percent recovery.

$$\frac{Vo-Sc}{Ve} \times 100 = \% \text{ recovery} \qquad \text{EQUATION 2}$$

Vo = the observed value of the spiked sample
Sc = the observed value of the sample
Ve = the expected value of the spike

13.6. The relative percent difference (RPD) between duplicates is calculated as the absolute difference between the sample and duplicate, divided by the average of the sample and duplicate, all multiplied by 100.

EQUATION 3

$$\frac{|Sc - Dc|}{\{(Sc + Dc) / 2\}} \times 100 = \text{Duplicate Relative Percent Difference (Dup RPD)}$$

Sc = observed sample concentration
Dc = observed duplicate sample concentration

13.7. The post digestion spike (BND) spike recovery is calculated as the difference of the BND concentration minus the sample concentration all divided by the expected concentration of the BND. The resultant value is multiplied by 100 to give percent recovery.

$$\frac{BSc - Sc}{BSe} \times 100 = \% \text{ recovery} \qquad \text{EQUATION 4}$$

BSc = observed value of post-digestion spike
Sc = observed value of sample
BSe = expected value of post-digestion spike

13.8. Samples that have been digested using concentration procedures must have the concentrations of the elements multiplied by the appropriate factor.

13.9. Data Archiving: The raw data files produced by the ICP-MS software are moved over to the network on a monthly basis. The archive location is:
\\usfs700\ANK_Instrument_Data\METALS\ICPMS_Archive\DATA

14.0. HEALTH AND SAFETY:

14.1. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices should be followed at all times.

14.2. The reagents, solutions, and chemicals that are utilized throughout this method have varying degrees of toxicity. The material safety data sheet (MSDS) for each chemical is located online and in the safety office. Before performing any part of the procedure, the pertinent MSDSs should be read.

14.3. Safety glasses should be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

14.4. A lab coat should be worn whenever handling samples, digests or reagents in the laboratory.

14.5. Gloves should be worn at all times when handling samples and reagents.

14.6. Liquid argon represents a potential cryogenic and asphyxiation hazard. Safe-handling procedures should be used when handling liquid argon tanks at all times.

14.7. The ELAN 6100 is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the ELAN if any safety interlock is known to be disabled or malfunctioning. Spilled samples, reagents, and water should be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills, the acid should be neutralized with sodium bicarbonate solution before cleanup.

14.8. Samples are to be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.9. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized. Elementary neutralization may be performed at the bench or using the Elementary Neutralization Hood located in GC Prep. The sample custodian may accomplish this step.

14.10. All surplus reagent acids shall be neutralized on a daily basis. Elementary neutralization may be performed at the bench or using the Elementary Neutralization Hood located in GC Prep.

14.11. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session. This may be performed in conjunction with elementary neutralization.

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15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

DL studies are performed when a significant change in instrument response is observed, when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

18.0. LIMIT OF DETECTION (LOD):

The LOD is defined per SOP 116.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP 116. LOQs may be adjusted for sample dilution or the presence of matrix interferences. LOQs are established based on experimental DL studies and comparison to program and project-specific reporting limit requirements**.** LOQ information is only provided to give the reader an idea of the reporting capabilities of the method as it is implemented at SGS. LOQs may change at the laboratory's discretion.

Element	LOQ ug/L
Li	0.8
Ag	0.04
Al	4
As	1
B	10
Ba	0.1
Be	0.1
Bi	0.1
Ca	100
Cd	0.1
Co	0.04
Cr	0.4
Cu	1
Fe	40

K	100
Mg	40
Mn	0.1
Mo	0.1
Na	200
Ni	1.24
P	80
Pb	0.2
Sb	0.1
Se	2
Si	200
Sn	0.4
Sr	10
Tl	0.04
V	2
Zn	6.2
U	0.08

20.0. REFERENCES:

20.1. Methods for the Determination of Metals in Environmental Samples - Supplement 1", EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942.

20.2. ICP-MS 300 Training Manual, 2009, Perkin-Elmer Corporation.

20.3. EPA 200.8 Rev.5.4: "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma- Mass Spectrometry", 1994.

20.4. Agilent 7800/7900 ICP-MS Hardware Maintenance Manual, 2019, Agilent Technologies, Inc.

21.0. ATTACHMENTS:

Attachment A: Corrective Action Table
Attachment B: Method Nomenclature
Attachment C: Primary and Secondary Isotopes

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Attachment A: CORRECTIVE ACTION TABLE

Internal Quality Control Procedures EPA200.8 (Low-Level)

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.8	Calibration	Daily before each method.	Correlation coefficient acceptance criterion ≥ 0.995.	1. Find source of problem and correct. 2. Repeat calibration.
EPA200.8	Instrument Performance	Daily before each method.	See Sections 11.2.1 and 11.2.2 for parameters.	1. Fix problem and recalibrate.
EPA200.8	Calibration Blank (CB)	After calibration, 1 per 10 sample readings, and at the end of the analytical run.	Concentration < LOQ	1. Rinse with acid and repeat. 2. Evaluate samples; if N.D. or concentration > 5 times blank contamination level, ok to report. 3. Apply B flag if results reported without compliant blank. 4. Re-digest and reanalyze samples with concentration of contaminant > LOQ 5. Recalibrate 6. Reanalyze all affected samples.
EPA200.8	Calibration Verification Std. (CCV)	1 per 10 sample readings and at the end of the analytical run.	± 15%	1. Repeat analysis once. 2. Fix problem. 3. Recalibrate. 4. Reanalyze all affected samples.
EPA200.8	Initial Calibration Verification Std. (ICV)	After Calibration	± 10%	1. Repeat analysis once. 2. Fix problem. 3. Recalibrate.
EPA200.8	Quality Control Standard (QCS) Second Source	After ICV	± 10%	1. Repeat analysis once. 2. Fix problem. 3. Recalibrate
EPA200.8	Method Blank (initial Calibration Blank) (MB)	1 per batch of 20 or less	Concentration < LOQ	1. Repeat analysis once. 2. Evaluate samples; if N.D. or sample concentration > 5 times blank contamination level, ok to report. 3. Apply B flag if results reported without compliant blank. 4. Re-digest and reanalyze samples with concentration of contaminant > LOQ.

EPA200.8	Laboratory Control Sample (LCS)	1 per batch of 20 or less	Recovery ± 15% Or lab control limits	1. Repeat analysis once. 2. Evaluate samples; if LCS is high but samples are ND for that analyte, ok to report, but flag sample. 3. Re-digest and reanalyze samples with concentration of contaminant > LOQ. 4. Re-digest and reanalyze samples for all failed analytes, if the LCS fails low.
EPA200.8	Matrix Spike (MS)	1 per 10 samples Digested	70-130% if analyte concentrations are <4times the spike.	1. If MS recovery is not within limits and LCS is in control, flag data as matrix interference suspected.
EPA200.8	Matrix Spike Dup. (MSD)	Per client request	70-130% if analyte concentrations are <4times the spike. RPD ≤ 20	1. If MS recovery is not within limits and LCS is in control, flag data as matrix interference suspected. 2. Also flag data as possible matrix interference if RPD is not within limits.
EPA200.8	Post Digest Spike (BND)	When required	70 – 130%	1. Flag Data as suspected matrix interference.
EPA200.8	Low Level Cd/SE Spike (BBND)	Per client request	50-150%	1. Flag Data as suspected matrix interference.
EPA200.8	Internal Standards	In all solutions	In CCV/CB: 60-125% original cal blank intensity. --- In Samples ICS stds.: 60 – 125% original cal blank intensity.	1. Terminate analysis. 2. Correct problem. --- 1. Dilute the sample 5x and reanalyze.

ATTACHMENT B: METHOD NOMENCLATURE

METHOD SYMBOL	SGS SYMBOL	METHOD NAME	SGS NAME
CAL	ICV, CCV	Calibration Standard	Continuing Calibration Verification
LD1, LD2	DUP	Laboratory Duplicates	Duplicates
LFB	LCS	Laboratory Fortified Blank	Laboratory Control Sample
LFM	MS/MSD	Laboratory Fortified Matrix	Matrix Spike/ Matrix Spike Duplicate
LRB	MB	Laboratory Reagent Blank	Method Blank
QCS	QCS	Quality Control Sample	Quality Control Sample

Attachment C: ICP/MS Isotopes

The following isotopes should be monitored for ICP/MS analyses.
Determination of the primary isotope will be based on user knowledge and matrix. The tables below are to be used only as a general guideline. Interferences generally result in a biased high isotope. In most cases, an attempt should be made to report the lowest recovering isotope if there is a significant difference between isotopes.

Element Isotopes for Monitoring Only

Analyte		Isotope	Notes
Tungsten	**W**	**184, 186**	
Chlorine	**Cl**	**37**	
Carbon	**C**	**13**	
Scandium	**Sc**	**45**	Internal Standard
Chromium	**Cr**	**53**	
Nickel	**Ni**	**58, 62**	
Zinc	**Zn**	**67**	
Germanium	**Ge**	**74**	Internal Standard
Selenium	**Se**	**77**	
Krypton	**Kr**	**83**	
Molybdenum	**Mo**	**97**	
Ruthenium	**Ru**	**99**	
Palladium	**Pd**	**105**	
Cadmium	**Cd**	**106, 108**	
Gold	**Au**	**197**	
Indium	**In**	**115**	Internal Standard
Iridium	**Ir**	**193**	Internal Standard
Lead	**Pb**	**204**	
Titanium	**Ti**	**48**	
Iron	**Fe**	**56**	

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Reportable Element Isotopes

Analyte		Preferred	Alternate	Notes
Lithium	**Li**	**7**		
Beryllium	**Be**	**9**		
Boron	**B**	**11**		
Aluminum	**Al**	**27**		
Vanadium	**V**	**51**		
Chromium	**Cr**	**52**		
Manganese	**Mn**	**55**		
Cobalt	**Co**	**59**		
Nickel	**Ni**	**60**		
Copper	**Cu**	**65**	63	Use 63 with high Ca
Zinc	**Zn**	**66**	68	
Arsenic	**As**	**75**		
Selenium	**Se**	**82**	78	Use 78 only for method 3051
Strontium	**Sr**	**88**		
Molybdenum	**Mo**	**95**	98	Isotopes are essentially equal
Silver	**Ag**	**107**	109	Isotopes are essentially equal
Cadmium	**Cd**	**114**	111	Use 111 with high Mo
Tin	**Sn**	**118**		
Antimony	**Sb**	**121**	123	Isotopes are essentially equal
Barium	**Ba**	**135**	137	Isotopes are essentially equal
Mercury	**Hg**	**202**	201	If W is present; use lowest recovering isotope
Thallium	**Tl**	**203**	205	Isotopes are essentially equal
Lead	**Pb**	**206, 207, 208**		The reported value for lead is the sum of the three isotopes.
Bismuth	**Bi**	**209**		
Uranium	**U**	**238**		
Sodium	**Na**	**23**		
Magnesium	**Mg**	**24**	25	
Silicon	**Si**	**28**	29	
Phosphorus	**P**	**31**		
Potassium	**K**	**39**		
Calcium	**Ca**	**43**		
Titanium	**Ti**	**47**		
Iron	**Fe**	**54**	57	

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Alkalinity, Acidity, pH, Free CO_2 & Conductivity by Auto-titrator	**Revision Date**: April 2023
Method No: SM2320B, SM2310B, SM4500H-B, 4500-CO_2, SM2510A and B	**SOP No**: 394r010
Page: 1 of 22	**Supersedes:** 394r09

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	4/13/23	Samara Beatty	4/13/23

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- Section 10.3.1 corrected to current number of standards used for conductivity calibration check.
- Minor grammatical and format changes made throughout.
- Note added to section 11.4 regarding pH analysis requiring no dilutions.

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1.0. OBJECTIVE:

This method outlines a procedure for determining the pH, conductivity, alkalinity and/or acidity of aqueous or solid material by auto-titrator.

2.0. SCOPE AND APPLICATION:

2.1 **Conductivity:** Conductivity is a numerical expression of the ability of an aqueous solution to conduct electric current. This ability depends on the presence of ions; their concentrations, mobility and valence; and on the temperature of the measurement. This method is used to determine the conductivity and resistivity of drinking, surface, saline waters, domestic and industrial wastes, and soils. Laboratory conductivity measurements are used for various purposes such as estimating total dissolved solids in a sample, assessing degree of mineralization or estimating sample size to be used for common chemical determinations.

2.2 **pH:** A pH electrode and a thermistor temperature sensor or a combination electrode is used to determine the pH of drinking water, surface water, and aqueous industrial waste samples.

2.3 **Alkalinity:** This operating procedure is used to determine the alkalinity of surface and groundwater, effluents, and industrial waters by means of an auto-titrator. An unaltered sample is titrated to a predetermined pH of 4.5 using 0.02N H_2SO_4. The end point is determined with a pH meter and results are reported as milligrams per liter. If a lower detection limit is needed the titrator will then titrate the sample to a pH of 4.2. To distinguish different forms of alkalinity the titrator will titrate to a pH of 8.3 when applicable.

2.4 **Acidity:** This operating procedure is used to determine the acidity of drinking water, surface and sewer waters, industrial waters, particularly mine drainage and receiving streams, and other water free of ferrous iron or other polyvalent cations in a reduced state by means of an auto-titrator. An unaltered sample is titrated to a predetermined pH of 8.3 using 0.02N carbonate-free NaOH solution. The end point is determined with a pH meter and results are reported as milligrams per liter.

2.4.1 This operating procedure is also used to determine the acidity of samples containing hydrolysable metal ions and reduced forms of polyvalent cations via hot peroxide treatment.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 The conductivity meter used at SGS incorporates a temperature probe. The cell constant is not adjusted. Calibration is confirmed at the beginning of each batch by reading high and low, NIST traceable, standards.

3.2 Conductivity samples are run at room temperature and are not warmed to 25ºC. PT studies demonstrate that this is an acceptable deviation from the reference method.

3.3 Acidity samples have a 14-day regulatory holding time.

3.4 Acidity and Alkalinity samples are analyzed at 50 mL. PT studies demonstrate that this is an acceptable deviation from the reference method.

3.5 The pH probe is a temperature compensating probe and, therefore, the temperature is not recorded in the run log. The temperature is, however, available in the raw data printed by the auto-titrator.

3.6 pH buffers will be used within the listed expiration date unless they fail to produce a valid calibration. If a buffer begins to respond erratically it will be replaced with an unopened container.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 An electronic copy of this SOP and any prior versions of this SOP are maintained on the computer network in a "read only" file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

4.6 It is the responsibility of the analyst to monitor the waste and reagent levels in order to avoid spills and false results.

5.0. INTERFERENCES:

5.1 **Conductivity:**

5.1.1 Temperature variations and corrections represent the largest source of potential error. The probe includes an automatic temperature compensation (ATC) probe.

5.1.2 Conductivity cell must be kept clean. See instrument manuals for recommended cleaning procedures.

5.1.3 Samples containing mineral acids may also contribute to potential errors.

5.2 **pH:**

5.2.1 Samples containing large amounts of particulate or oily films may cause problems with electrode response. Removal of the interfering matter may be accomplished by wiping the electrode, gentle washing with a detergent followed by a thorough rinse with DI water, treatment with dilute hydrochloric acid, or a combination of the above.

5.2.2 The temperature of the sample may interfere with the pH analysis. The temperature could affect the electrode itself, or the sample pH could be temperature dependent. pH analysis should be performed on samples and buffers at room temperature.

5.3 **Alkalinity:**

5.3.1 Response time can become sluggish if the sample is oily or coats pH probe.

5.3.2 If the sample contains inorganic acids or salts of a weak acid, they can cause interferences with the electrometric pH meter.

5.3.3 Bubbles in the titrant dispensing line may cause incorrect dispensing volumes. To remove bubbles, follow the steps outlined in section 11.2.10.

5.3.4 If the sample contains mineral acids, it may be necessary to change the end point to a pH of 3.9.

5.4 **Acidity:**

5.4.1 Samples may be subject to microbial action and to loss or gain of CO_2 or other gases when exposed to air. Sample agitation and prolonged exposure to air should be avoided.

5.4.2 Suspended matter present in the sample, or precipitates formed during the titration may cause sluggish electrode response. This may be offset by a slow drop wise addition of titrant as the endpoint pH is approached.

5.4.3 Bubbles in the titrant dispensing line may cause incorrect dispensing volumes. To remove bubbles, follow the steps outlined in section 11.2.10.

5.4.4 Hydrolysable metal ions and reduced forms of polyvalent cations such as acid mine drainage may cause interference. These samples must be prepared using the hot peroxide treatment method.

6.0. SAMPLE HANDLING:

6.1 Sample Matrix – Groundwater, surface and sewer waters and industrial wastes.

6.2 Sample Collection –The samples should be collected in 500mL plastic containers and stored at 0-6ºC.

6.3 Sample Size – The 110mL tubes should be approximately ¾ of the way filled with sample (approximately 80mL). The titrator will take up 50 mL to analyze. Conductivity, pH and alkalinity can be performed with one 110mL tube, but Acidity will need its own 110mL tube for analysis.

6.4 Sample Preservation – Unpreserved.

6.5 Holding Times:

6.5.1 Conductivity samples are to be analyzed within 28 days of collection. If analysis is not completed within 28 days, filter through a 0.45 µm filter. Filter and apparatus should be washed with DI water and pre-rinsed with sample before use.

6.5.2 The pH of samples should be taken as soon after collection as possible. Analysis in the field would be optimal.

6.5.3 Alkalinity and Acidity analysis should take place as soon as possible after collection. The maximum hold time is 14 calendar days from collection.

6.6 Criteria for Acceptance/Rejection of Samples – If samples are not stored at 0-6ºC or if samples have broken hold time, approval from Project Manager (PM) should first be obtained before proceeding with analysis.

7.0. APPARATUS:

7.1 Metrohm 855 Robotic Titrosampler or equivalent. Both the conductivity probe and pH probe are temperature-compensating probes.

7.2 Tiamo titration software or equivalent.

7.3 110 mL plastic tubes or equivalent.

7.4 Autotitrator maintenance

7.4.1 Ensure the stopper on the pH probe is removed when running and is put back in place as soon as the run has ended.
7.4.2 Rinse probe with tap water after each run and put back in storage solution.
7.4.3 Ensure storage solution is kept full and the probe is placed back into this solution after every run.
7.4.4 Empty, clean out and refill the storage solution periodically.

7.5 For further instrument maintenance refer to the manual. For computer hardware/software, refer to IT.

8.0. REAGENTS:

8.1 DI H_2O.

8.2 Conductivity standards:

8.2.1 Inorganic Ventures (or equivalent) 5 µmhos/cm standard (CAT#CON5-25)

8.2.2 Fisher (or equivalent) 10 microsiemens standard (CAT#09-328-1)

8.2.3 Fisher (or equivalent) 100 microsiemens m standard (CAT#09-328-2)

8.2.4 Fisher (or equivalent) 1000 microsiemens standard (CAT#09-328-3)

8.2.4.1 These are NIST Traceable Calibration Standards. Standards are stable for 1 year from date of preparation, or until the expiration date listed on the standard, whichever is first.

8.3 pH 4 & 7 Guide 34 buffers are purchased from Inorganic Ventures. Certified pH 10 buffer is purchased from Fisher Scientific. or other equivalent vendors.

8.4 Electrode Filling Solution and electrode storage solution, 3M KCL.

8.5 Standard H_2SO_4 titrant (0.02N): This reagent is purchased commercially from Fisher, or equivalent.

8.6 Alkalinity Quality Control (QC) Standard, Hach: 25,000 mg/L. Dilute 10 mL (one ampule) to 1,000 mL in a volumetric flask to yield a final concentration of 250 mg/L. The concentration may vary from lot to lot so check the certificate of analysis for the current concentration.

8.7 Acidity QC Standard, Potassium Hydrogen Phthalate. Dissolve 0.15 g in 1,000 mL of DI water to yield a final concentration of 37 mg/L as $CaCO_3$.

8.8 Standard NaOH titrant (0.02N): This reagent is purchased commercially from Fisher, or equivalent.

8.9 Hot peroxide reagents:

8.9.1 Sulfuric acid (0.02N): This reagent is purchased commercially from Fisher, or equivalent.

8.9.2 Hydrogen Peroxide (30%).

9.0. EXTRACTION:

9.1 **Conductivity:**

9.1.1 For soils, a 1:1 extraction with DI water is performed before analysis, if enough soil is available. Weigh 60 grams of soil into a titrator tube or vessel large enough to handle extraction. Add 60 mL of DI water. Swirl the tube for 3-5 minutes and then centrifuge or let soil settle for at least 1 hour or centrifuge. The amount of soil and water can be adjusted according to the needs of the instrument being used, as long as a 1:1 ratio is maintained. Calibration and standard checks should be done as in section 10.0. A duplicate should be prepared for every 10 samples. Analyze the aqueous layer in the same manner as described in section 11.0.

9.2 **Alkalinity:**

9.2.1 For soils, a 1:1 extraction with DI water is performed before analysis. Weigh 50 grams of soil into a titrator tube or a vessel large enough to handle extraction. Add 50 mL of DI water. Swirl the tube for 3-5 minutes and then centrifuge or let soil settle for at least 1 hour. The amount of soil and water can be adjusted according to the needs of the instrument being used, as long as a 1:1 ratio is maintained. Use Teflon chips as the soil matrix for the method blank and LCS. Use DI water for the method blank and the alkalinity standard as the liquid matrix in the LCS. Be sure to use a 1:1 ratio for both. Analyze the aqueous layer in the same manner as described in section 11.0.

9.3 **Acidity:**

9.3.1 Most samples received by SGS are free from hydrolysable metal ions and reduced forms of polyvalent cations. These samples do not require any treatment.

9.3.2 Samples containing, or suspected of containing, hydrolysable metal ions or reduced forms of polyvalent cations must be flagged as such by the PM. These samples must be treated with hot peroxide.

9.3.2.1 *Hot peroxide treatment:* Aliquot a sample into a beaker and measure the pH. If the pH is greater than 4.0 add 5mL increments of 0.02N sulfuric acid until the pH is below 4.0. Carefully note the amount of acid used. Add 5 drops of 30% H_2O_2 and boil for 2 to 5 minutes.

10.0. CALIBRATION:

Conductivity:

10.1 A one-point calibration is performed for conductivity using the 100 µmhos/cm standard. Calibration must be done weekly.

10.2 **Setting Up and Running a Conductivity Calibration:**

10.2.1 Select the workplace icon.

10.2.2 Insert new line in sample data chart.

10.2.3 Select *Conductivity Cal SGS* method from the drop-down menu.

10.2.4 Enter sample position of 100µmhos/cm buffer.

10.2.5 Name sample *Conductivity Cal*.

10.2.6 Set sample size to 1.0 mL.

10.2.7 Press *Apply* and close dialogue box.

10.2.8 Press *Start* to run conductivity calibration.

10.3 **Checking a Conductivity Calibration:**

10.3.1 Pour out approximately 70-80 mL of the four conductivity standards into four different plastic 110mL tube or equivalent.

10.3.2 Insert new line in sample data chart.

10.3.3 Select *Conductivity STD Check SGS* method from drop down menu.

10.3.4 Enter sample position of 5 µmhos/cm buffer.

10.3.5 Name sample the true value of the buffer.

10.3.6 Set sample size to 50.0 mL.

10.3.7 Press the arrow that points to the right, immediately beside the *Line* number near the bottom of the dialogue box.

10.3.8 Repeat steps 10.3.3-10.3.7 for the remaining conductivity standards.

10.3.9 Press *Start* to run conductivity calibration check.

10.3.10 Press the Database icon to review the calibration check data. The QC recovery should be within ±10% of the true value for the standards >5.0 µmhos/cm. The 5.0 µmhos/cm should be within ±50% of the true value. The 10 µmhos/cm and 1000 µmhos/cm are posted into LIMS as the LCS and CVS, respectively. The remaining standards are verified that they meet QC criteria before continuing with analyzing samples, but they are not posted.

10.4 A three-point calibration is performed for pH using buffers with pH values 4, 7, and 10. Calibration must be done weekly.

10.5 **Setting Up and Running a pH Calibration:**

10.5.1 Insert new line in sample data chart.

10.5.2 Select *pH Cal – 3buffers SGS* method from the drop-down menu.

10.5.3 Input sample position as 1.

10.5.4 Name sample *pH Cal*.

10.5.5 Set sample size to 1.0mL.

10.5.6 Press *Apply* and close dialogue box.

10.5.7 Fill three 110mL sample tubes with ph 4, 7, and 10 buffers. Set these buffers into positions 55, 56, and 57 respectively. (*Note: Buffers MUST be placed in these positions, no matter what position is set as the sample position.)*

10.5.8 Press *Start* to run pH calibration.

10.6 **Checking a pH Calibration:**

10.6.1 Fill three different plastic 110 mL tubes approximately ¾ of the way with the three pH buffers.

10.6.2 Insert new line in sample data chart.

10.6.3 Select *pH SGS* method from drop down menu.

10.6.4 Enter sample position of pH 4 buffer.

10.6.5 Name sample the true value of the buffer.

10.6.6 Set sample size to 50.0mL.

10.6.7 Press the arrow that points to the right, immediately beside the *Line* number near the bottom of the dialogue box.

10.6.8 Repeat steps 10.6.3-10.6.7 for the other two pH standards.

10.6.9 Press *Start* to run pH calibration check.

10.6.10 Press the *Database* icon to review the data. The passing criterion is ± 0.05 pH points from the true standard value.

11.0. ANALYSIS:

11.1 **Setting Up the Instrument:**

11.1.1 Inspect all reagent containers to assure that the levels are appropriate *(**Note:** This is the area that is most prone to mistakes)*. Empty waste container if necessary, make sure both water tanks are filled with DI H_2O and make sure there are appropriate levels of titrant.

11.1.2 Prepare samples for analysis. All samples are analyzed at room temperature. Shake the sample to homogenize and pour sample out filling the 110mL tubes approximately ¾ of the way full.

11.1.3 Put two containers of DI H_2O in the last two positions. These will be the rinses.

11.1.4 Ensure stopper on pH probe is removed.

11.2 **Setting Up the Software:**

11.2.1 Open the Tiamo software and open the *Workplace* tab.

11.2.2 Insert new line in the *Sample data* chart.

11.2.3 Select appropriate method(s) for what the particular sample needs to be analyzed for.

11.2.4 Enter the sample position for the current sample.

11.2.5 Label with appropriate HSN.

11.2.6 Enter sample size of 50.0mL. (***Note:** Limited volume samples may not have a high enough volume for the full 50.0mL. In these cases, assign a sample size that is 10.0mL less than what is in the tube. This is to make sure the titrator does not draw up air.)*

11.2.7 Press the arrow that points to the right, immediately beside the Line number near the bottom of the dialogue box.

11.2.8 Repeat steps 11.2.3 – 11.2.7 for the remainder of the samples.

11.2.9 The maximum number of samples that can be analyzed at a time is 54. The last five sample positions are reserved for the rinses and pH calibration.

11.2.10 It may be necessary to flush the titrant lines before analysis if a new titrant is used in the cartridge or if bubbles develop in the titrant line. In Tiamo click the *Manual* icon. Choose the appropriate dosing device, click the *Prepare* tab and press *Start*.

11.3 Starting the instrument – press the *Start* icon.

11.4 The instrument will stop when the *Sample data* chart is complete. New samples can be added at any time during the process. The instrument will analyze the samples in the order of the *Sample data* chart and NOT in order of sample positions.

Note: *The pH must be analyzed at a 1X and cannot be analyzed at a dilution. If the sample cannot be run at 1X on the Autotitrator, for example if the matrix is a dense fish sample or leachate, email front counter and Project Manager (PM) to have pH analysis rescheduled to SW9040.*

12.0. QUALITY CONTROL:

12.1 Precision Measurements – Run one sample duplicate for every ten samples.

12.1.1 **Conductivity:** Duplicate should have a Relative Percent Difference (RPD) of ≤ 20 or a difference of less than 1.0 µmhos/cm for low-level samples. Low level samples are samples with a conductivity result below 10 µmhos/cm. If duplicate fails re-homogenize the sample by thorough agitation, rinse cell and repeat analysis.

12.1.2 **pH:** Duplicate readings must agree within 0.1 pH units. If duplicate fails re-analyze sample. Samples that fail this criterion should be flagged as non-homogeneous.

12.1.3 **Alkalinity:** Duplicate should have an RPD of ≤25. If the RPD fails, shake the sample well to ensure that the sample is well mixed. Reanalyze the sample and duplicate.

12.1.4 **Acidity:** Duplicate should have an RPD of ≤25. If the RPD fails, shake the sample well to ensure that the sample is well mixed. Reanalyze the sample and duplicate.

12.2 Accuracy Measurements – Run a QC sample for every twenty samples.

12.2.1 **Conductivity:** Run the 10 µmhos/cm and 1000 µmhos/cm standards as calibration checks. The QC recovery should be within ±10% of true value. If QC sample is not within criteria, recheck once. If it is still outside QC criteria, then re-calibrate and repeat analysis. This applies to waters and soils.

12.2.2 **pH:** Measure the pH of the 7 buffer solution. The pH buffer must be within 0.05 pH units. If QC sample is not within 0.05 pH units, re-calibrate and repeat analysis.

12.2.3 **Alkalinity:** Run a 250 mg/L Laboratory Control Sample (LCS) that has been prepared in the lab (section 8.5). Control limits for QC recovery are ± 15%. If the QC sample recovers outside control limits, check the accuracy of the titrant concentration and reanalyze.

12.2.4 **Acidity:** Run a 37 mg/L LCS that has been prepared in the lab (section 8.7). Control limits for QC recovery are ±15%. If the QC sample recovers outside control limits, check the accuracy of the titrant concentration and reanalyze.

12.3 Calibration Criteria – Calibration checks are needed for both conductivity and pH readings.

12.3.1 **Conductivity:** Calibrate the titrator with the 100 µmhos/cm standard (***Note:*** *Be sure that the true value of the standard is the one that is entered into the* Conductivity Cal SGS *method).* The EPA Manual for the Certification of Drinking Water Laboratories (5th edition) states that conductivity probes are to be calibrated monthly. SGS North America, Inc. – Alaska Division performs the calibration weekly. A calibration check is then performed using 5 µmhos/cm, 10µmhos/cm, 100µmhos/cm, and 1000µmhos/cm. The QC recovery should be within ±10% of true value for the standards >5.0 µmhos/cm. The 5.0 µmhos/cm should be within ±50% of the true value. If QC sample is not within QC criteria, re-calibrate and repeat analysis.

12.3.2. **pH:** A three-point calibration is used with pH 4, 7, and 10 buffers. A calibration check is then done with pH 4, 7, and 10 buffers. The pH buffer readings must be within 0.05 pH units of the true value. If QC sample is not within 0.05 pH units, re-calibrate and repeat analysis. pH calibrations are to be performed weekly.

12.3.3. Conductivity, Alkalinity, & Acidity, Blank Criteria – Run a method blank (MB) (DI water) at the beginning of each run of 20 samples. For soils, add 60 mL of DI water to 60 g of Teflon chips, prepare as in section 9.1-9.2 and analyze. The acceptable range for blanks is less than the LOQ. If the blank value is outside acceptable control limits, the quality of deionized water should be examined. Samples having concentration greater than 10 times the blank value may be reported with comments. Samples having less than 10 times the blank value must be reanalyzed.

12.3.3.1. Alkalinity Blanks for DOD clients and drinking water samples must be evaluated at ½ the LOQ.

12.4. Alkalinity analysis (drinking water): an additional QC sample (LLQ): low level quantitation) must be analyzed at the LOQ.

12.5. Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 Units/Significant Figure:

13.1.1 **Conductivity:** Units are µmho/cm reported to two decimal places.

13.1.2 **pH:** Units are pH and are reported to one decimal place for samples and two decimal places for calibration.

13.1.3 **Alkalinity:** Units are mg/L and are reported to two decimal places.

13.1.4 **Acidity:** Units are mg/L and are reported to two decimal places.

13.2 Equations:

13.2.1 **Resistivity:**

13.2.1.1 Resistivity is calculated using the measured conductivity as follows:

$$\text{Resistivity (ohm-m)} = \frac{1}{(A/10{,}000)}$$

Where: A = Conductivity, µmho/cm

13.2.2 **Alkalinity:**

13.2.2.1 Alkalinity can be reported as four different types, depending on the pH values and amount of titrant used to obtain the different pH endpoints. The most common form is alkalinity as $CaCO_3$ (Total alkalinity) and it is calculated as follows:

$$\text{Alkalinity (mg/L } CaCO_3) = \frac{A*B*50{,}000}{C}$$

Where: A = mL of titrant to pH 4.5
B = H_2SO_4 normality, N
C = Sample volume, mL

13.2.2.2 If the initial pH is greater than 8.3, the auto titrator will titrate to pH 8.3 and record the volume of titrant used (expressed as P). The titrator will then continue titrating to a pH of 4.5 (The total volume is expressed as T). Use equation described in 13.2.2.1 and Table listed in 13.2.2.3 to determine the alkalinity relationships.

13.2.2.3 Types of Alkalinity

P & T Relationship	**OH^- Alkalinity**	**CO_3 Alkalinity**	**HCO_3 Alkalinity**
P=0	0	0	T
P < ½ T	0	2P	T – 2P
P = ½ T	0	2P	0
P > ½ T	2P - T	2(T – P)	0
P = T	T	0	0

P = Phenolphthalein alkalinity (calculated from volume of titrant to pH 8.3)
T = Total alkalinity (calculated from volume of titrant to pH 4.5)

13.2.2.4 If the calculated total alkalinity value is less than 20 mg/L, the auto-titrator will titrate the sample to a pH of 4.2. **Low-level alkalinity is posted in place of total alkalinity, except for the method blank**. Low-level alkalinity is calculated as follows:

$$\text{Low Level Alkalinity (mg/L, } CaCO_3) = \frac{(2A - D)*B*50{,}000}{C}$$

Where: A = Titrant volume to pH 4.5, mL
B = H_2SO_4 normality, N
C = Sample volume, mL
D = Titrant volume to pH 4.2, mL

13.2.3 **Acidity:**

13.2.3.1 The Acidity of a sample is calculated as follows:

$$\text{Acidity (mg/L)} = \frac{A*B*50{,}000}{C}$$

Where: A = Titrant volume to pH 8.3, mL
B = NaOH normality, N
C = Sample volume, mL

13.2.4. **Free Carbon Dioxide using 4500-CO_2: Nomograph for evaluation (See Attachment F):**

13.2.4.1. Aline temperature (scale 1) & Total Dissolved solids (scale 3) which determines Point **P1**

13.2.4.2. Aline pH (scale 4) & bicarbonate Alkalinity (scale 7) which determines Point **P2**

13.2.4.3. Aline **P1 & P2** to determine **free CO_2 (scale 5)**

Note: Temperature data found in the auto sampler instrument runlog
TDS, pH, and alkalinity are found in LIMs. (Inquiry by sample number - Show results tab)

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be own at all times. Proper PPE when handling samples includes a lab coat, gloves, and safety glasses. In addition, when handling concentrated acids or bases, a face shield and apron must be worn

15.0. POLLUTION PREVENTION:

15.1. SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

15.2. Pollution prevention techniques utilized in this method are smaller sample sizes to reduce the amount of waste and titrant. The conductivity calibration has been changed from a four-point calibration to a one-point calibration. Because the calibration check is only needed at the high and low levels, we have been able to eliminate the use of two conductivity standards.

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

17.1 The DL study is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

17.1.1 **Conductivity:** DL studies are not applicable to conductivity analysis.

17.1.2 **pH:** DL studies are not applicable to pH analysis.

17.1.3 **Alkalinity:** Per ADEC drinking water requirements DL studies are verified annually at a minimum. DL studies are performed when a new operator is trained, when a new instrument is purchased for analysis, or when a major modification to the methodology is made. The suggested DL spiking level for alkalinity is 2.0 mg/L.

17.1.4 **Acidity:** A DL study is performed initially, when a new instrument is purchased for analysis or when a major modification to the methodology is made. The suggested spiking level for acidity is 3.7 mg/L.

18.0. LIMIT OF DETECTION (LOD):

Refer to SOP 116 for further guidance.

19.0. LIMIT OF QUANTITATION(LOQ):

19.1 **Conductivity:** The current LOQ for this method is 5.0 µmho/cm.

19.2 **pH:** There is no LOQ available for this method.

19.3 **Alkalinity:** The current LOQ for this method is 10 mg/L.

19.4 **Acidity:** The current LOQ for this method is 10 mg/L. An LOD/LOQ verification needs to be performed quarterly for acidity.

19.5 *Note: LOQs are subject to change. A change to the LOQ does not necessitate a revision to this SOP.*

20.0. REFERENCES:

20.1 Standard Methods for the Examination of Water and Wastewater, 23rd edition online.

21.0. ATTACHMENTS:

Attachment A: Flow Chart
Attachment B: Corrective Action Table – Conductivity
Attachment C: Corrective Action Table – pH

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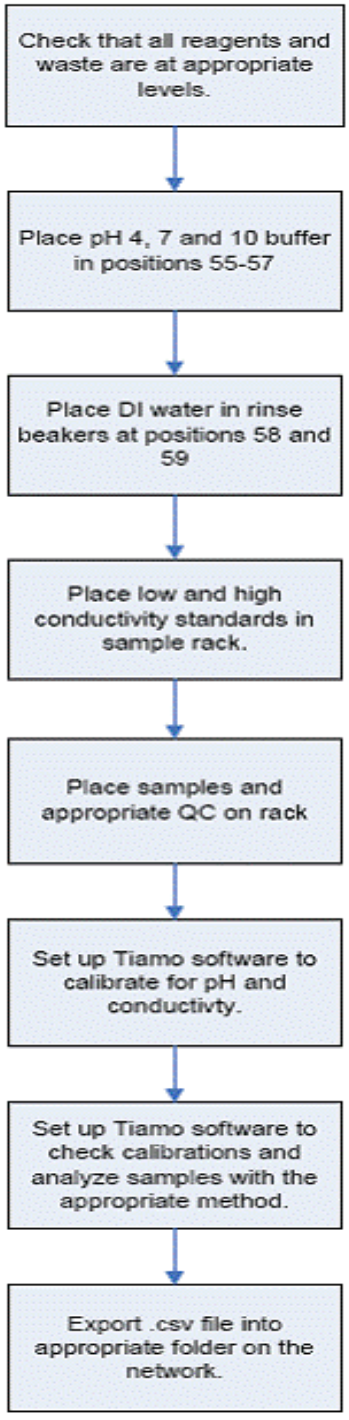
Attachment D: Corrective Action Table – Alkalinity
Attachment E: Corrective Action Table – Acidity
Attachment F: pH Best Practices
Attachment G: Free Carbon Dioxide Nomograph

ATTACHMENT A – Flow Chart



ATTACHMENT B: Corrective Action Table – Conductivity

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM 2510 A, B	Calibration: Confirm all points 5, 10, 100, and 1000	Weekly	± 50% for low point ± 10% for all other points	1. Find source of problem and correct. 2. Repeat calibration.
	LCS, CVS (two-point calibration verification)	1 per batch ≤ 20 samples	± 10%	1. Take a new homogenous portion of standard, rinse the cell and reanalyze. 2. Check instrument calibration or recalibrate.
	Duplicate (DUP) Sample	1 per ≤ 10 samples	RPD ≤ 20 or a difference of <1.0 µmho/cm for low-level samples	1. Take a new homogenous portion of sample, rinse the cell and reanalyze.
	Method Blank (MB)	One per batch of ≤ 20 samples	< LOQ	1. Re-rinse sample cell and reanalyze blank. 2. Check quality of DI system and operation.

ATTACHMENT C: Corrective Action Table – pH

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM4500H-B	Calibration: Three-point calibration	Weekly	Calibration Verification must pass	3. Find source of problem and correct. 4. Repeat calibration.
	Calibration Verification (read back buffers 4, 7, and 10). Buffer 7 is used for the LCS.	After calibration	Readback must be within 0.05 pH units of the known pH	1. Find source of problem and correct. 2. Repeat calibration.
	QC Sample (LCS) Buffer 7	1 per batch of ≤ 20 samples	Result must agree within 0.05 pH units of known pH	Recalibrate
	Matrix DUP	Minimum of 1 per batch, or 1 per ≤ 10 samples	Result must agree within 0.1 pH units	1. Reanalyze sample. 2. Samples that fail this criterion should be flagged.

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ATTACHMENT D: Corrective Action Table – Alkalinity

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM 2320B	Calibration: Three-point calibration	Weekly	Calibration Verification must pass	1. Find source of problem and correct. 2. Repeat calibration.
	Calibration Verification (read back buffers 4, 7, and 10)	After calibration	Read back must be within 0.05 pH units of the known pH	1. Find source of problem and correct. 2. Repeat calibration.
	MB **MB for Drinking Water Samples**	1 per batch of ≤ 20 samples	Less than the LOQ **Evaluate at ½ LOQ**	1. Evaluate samples. Samples having a concentration of greater than 10 times the blank value may be reported. Samples having a concentration of less than 10 times the LOQ must be reanalyzed. 2. Evaluate the DI water. 3. Recalibrate and reanalyze all affected samples.
	LCS	1 per batch of ≤ 20 samples	Recovery ± 15%	1. Check accuracy of titrant concentration. 2. Reanalyze.
	DUP sample	1 per ≤ 10 samples	RPD ≤ 25	1. Ensure that sample is well mixed 2. Reanalyze.

ATTACHMENT E: Corrective Action Table – Acidity

Analytical Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM 2310B	Calibration: Three-point calibration	Weekly	Calibration Verification must pass	1. Find source of problem and correct. 2. Repeat calibration.
	Calibration Verification (read back buffers 4, 7, and 10)	After calibration	Readback must be within 0.05 pH units of the known pH	1. Find source of problem and correct. 2. Repeat calibration.
	MB	1 per batch of ≤ 20 samples	Less than the LOQ	1. Evaluate samples. Samples having a concentration of greater than 10 times the blank value may be reported. Samples having a concentration of less than 10 times the LOQ must be reanalyzed. 2. Evaluate the DI water. 3. Recalibrate and reanalyze all affected samples.
	LCS	1 per batch of ≤ 20 samples	Recovery ± 15%	1. Check accuracy of titrant concentration. 2. Reanalyze.
	DUP sample	1 per ≤ 10 samples	RPD ≤ 25	1. Ensure that sample is well mixed 2. Reanalyze.

Attachment F: pH Best Practices

Keep the electrode hydrated

Why– Drying out the electrode leads to drifting pH values, slow response times, and incorrect measurements.

Fix – "Revive" a dry electrode by submerging the bulb and junction in pH storage solution for at least one hour.

Rinse, do not wipe your electrode

Why– Wiping the pH glass can produce a static charge which interferes with the pH reading of the electrode.

Fix – Simply rinse the electrode with tap water. Blot (do not rub) with a lint-free paper towel (e.g. Kimwipes®) to remove excess moisture.

Store your electrode in storage solution

Why– Storing in deionized water (DI) causes ions to leach from the glass membrane and reference electrolyte resulting in a slow and sluggish response.

Fix – Store your electrode in storage solution.

Clean your electrode regularly

Why– Deposits can form on the electrode during use, coating the sensing glass. This can lead to erroneous calibrations and readings.

Fix – Clean the electrode using a specially formulated cleaning solution for pH electrodes - ideally one that's developed for your application.

Calibrate often

Why – All pH electrodes need to be calibrated often for best accuracy.

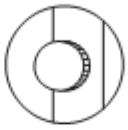
Fix – The frequency of calibration depends on how accurate you want to be - daily calibration is ideal.

Pick the right electrode for your sample

Why – General purpose electrodes are functional for a wide variety of applications but not ideal for all samples.

Fix – Based on your sample you may require an electrode designed for food, high/low temp, non-aqueous, or other types of samples.

Open or loosen the fill hole cap



Why– A closed electrode fill hole may lead to slower stabilization times.

Fix – Loosen or remove the fill hole cap. Remember to put it back when storing the electrode. (Not applicable for non-refillable electrodes)

Keep the electrolyte level full

Why– Electrolyte flows out from the reference junction over time. Low electrolyte levels may cause erratic readings.
(Not applicable for non-refillable electrodes)

Fix – Ensure that your electrode fill solution level is no less than one-half inch from the fill hole cap.

Properly submerge your electrode

Why– Both the pH sensing glass and reference junction need to be completely immersed in order to function properly.

Fix – Add enough sample to submerge both the junction and sensing glass.

Inspect your electrode

Why– Over time, the sensing portion of the glass becomes less responsive and will eventually fail. Damage from use is also possible. This will cause erroneous readings.

Fix – Check your electrode for damage and perform a slope and offset calculation. Reference blog for instructions.

Attachment G: Free Carbon Dioxide Nomograph

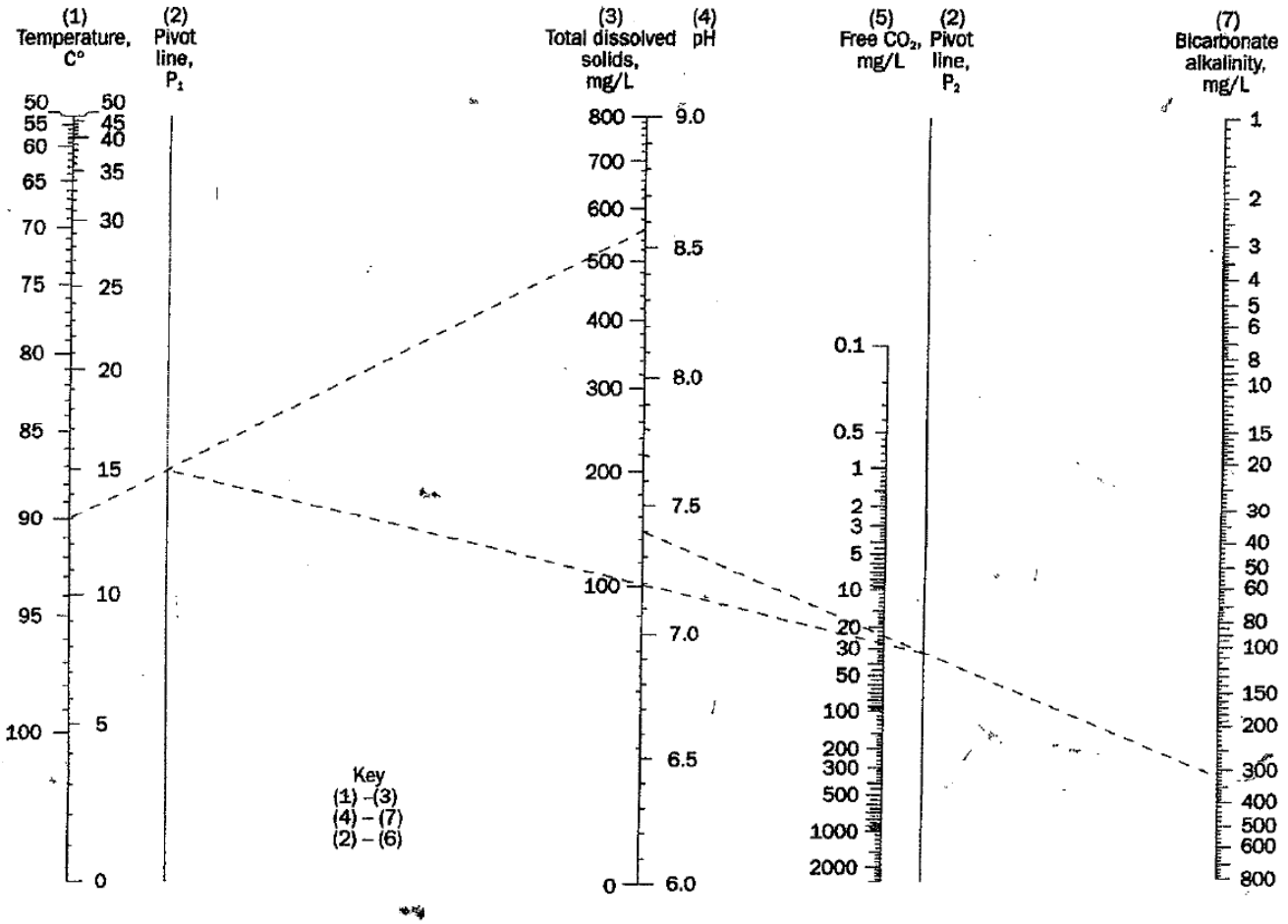


Figure 4500-CO_2:4. Nomograph for evaluation of free carbon dioxide content. To use: Align temperature (Scale 1) and total dissolved solids (Scale 3), which determines Point P_1 on Line 2; align pH (Scale 4) and bicarbonate alkalinity (Scale 7), which determines Point P_2 on Line 6; align P_1 and P_2 and read free carbon dioxide on Scale 5. (Example: For 13°C temperature, 560 mg total dissolved solids/L, pH 7.4, and 320 mg alkalinity/L, the free carbon dioxide content is found to be 28 mg/L.)

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Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	2/24/22	Samara Rentz	2/24/22

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Summary of Changes from Previous Revision:

- Incorporated all addenda
- Updated section 2.0 to enhance clarity.
- Updated section 7.1 to reflect current practices.
- Updated section 7.2 to reflect current practices.
- Updated section 8.7 to reflect current practices.
- Updated section 8.8 to reflect current practices.
- Removed section 10.4.
- Removed Table 3 and renumbered remaining tables and references.

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1.0. OBJECTIVE:

This procedure is used to determine the concentration of semi-volatile organic compounds in prepared extracts and is applicable to several types of matrices including soil, water, liquids, and solids.

Internal Standards

1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12

Surrogates

Acid Surrogates: 2-Fluorophenol, Phenol-d6, 2,4,6-Tribromophenol
Base/Neutral Surrogates: Nitrobenzene-d5, 2-Fluorobiphenyl, p-Terphenyl-d14

Target Compounds

N-Nitrosodimethylamine	Pyridine	Aniline	Phenol
Bis(2-chloroethyl)ether	2-Chlorophenol	1,3-Dichlorobenzene	1,4-Dichlorobenzene
Benzyl alcohol	1,2-Dichlorobenzene	2-Methylphenol	Bis(2-chloro-isopropyl)ether
3&4-Methylphenol	N-Nitroso-di-n-propylamine	Hexachloroethane	Nitrobenzene
Isophorone	2-Nitrophenol	2,4-Dimethylphenol	Benzoic Acid
Bis(2-chloroethoxy)methane	2,4-Dichlorophenol	1,2,4-Trichlorobenzene	Naphthalene
2,6-Dichlorophenol	4-Chloroaniline	Hexachlorobutadiene	4-Chloro-3-methyl-phenol
1-Methylnaphtalene	2-Methylnaphtalene	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
2,4,5-Trichlorophenol	2-Chloronaphthalene	1-Chloronaphthalene	2-Nitroaniline
Dimethylphthalate	2,6-Dinitrotoluene	Acenaphthylene	3-Nitroaniline
Acenaphthene	2,4-Dinitrophenol	4-Nitrophenol	2,4-Dinitrotoluene
Dibenzofuran	Diethylphthalate	Fluorene	4-Chlorophenylphenylether
4-Nitroaniline	4,6-Dinitro-2-methylphenol	N-Nitrosodiphenylamine	Azobenzene 4-
Bromophenylphenyl-ether	Hexachlorobenzene	Pentachlorophenol	Phenanthrene Anthracene
Di-n-Butylphthalate	Fluoranthene	Pyrene	Butylbenzylphthalate
3,3'-Dichlorobenzidine	Benzo(a)anthracene	Chrysene	Bis(2-ethylhexyl)-phthalate
Di-n-octylphthalate	Benzo[b]fluoranthene	Benzo[k]fluoranthene	Benzo[a]pyrene
Indeno(1,2,3-c,d)pyrene	Dibenzo[a,h]anthracene	Benzo[g,h,i]perylene	

Custom Compounds

Carbazole

2.0. SCOPE AND APPLICATION:

A measured weight or volume of sample is extracted with methylene chloride using method 3520C, 3550C or 3580A. The extracts are concentrated to a volume of 1 mL and analyzed by GCMS. Qualitative identification of the analytes is achieved using the relative abundance of their characteristic fragmentation patterns (m/z). Quantitative analysis is performed using the internal standard technique with a single quantitation ion.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. Method 8270D indicates that matrix spike/matrix spike duplicate (MS/MSD) are part of the minimum batch method QC. SGS performs this QC set only if sufficient sample volume is received; otherwise, an LCS/LCSD is performed.

3.2. Method 8270D states that the relative retention times of each analyte in each calibration standard should agree within 0.06 RRT units. SGS requires each calibration standard to agree within 0.06 RRT units from the mid point of the calibration.

3.3. The ICV recovery goal will be ±20 %, as per DOD QSM. Recoveries greater than ±20 %, but less than ±30% will be allowed for non-DOD samples.

3.4. Internal standard areas are monitored only from the mid point of the most recent calibration.

3.5. The surrogates and internal standards used do not appear in Table 8 of method 625.

3.6. The tune criteria is not the same as Table 9 in method 625, but is taken from Table 3 in method 8270D.

3.7. Tailing criteria is taken from method 8270D, not method 625.

3.8. Abundance of characteristic ions is taken from method 8270D, not method 625.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Solvents, reagents, glassware and other samples processing hardware may yield discrete artifacts that affect the quantitation of samples. All materials involved in the analysis of the samples must be demonstrated to be free from interferences under the conditions of the analysis by running method and instrument blanks.

5.2. Interferences by phthalate esters may pose a problem with analysis. Minimizing contact with any type of plastic material will help avoid this problem.

5.3. Matrix interference may be caused by co-extraction of contaminants from the sample. The interference varies depending on matrix and source of the environment being sampled.

5.4. Samples with high concentrations of acids or glycols may cause damage to the column, which may lead to reduced responses for some analytes. Clipping the column and/or recalibration may be necessary after such samples.

6.0. SAMPLE HANDLING:

6.1 This procedure is applicable to several types of matrices including soil, water, liquids, solids, and oils.

6.2 The samples should be collected in accordance with the project QAP.

6.3 The usual requested sample size is two 1-liter amber jars for water samples and one 4-ounce amber jar for soil samples.

6.4 After collection, the samples must be protected from light and stored at 0-6°C until extraction.

6.5 The holding time for water samples is seven days from collection. The holding time for soils is 14 days from collection. The extraction must occur within the listed hold time. The hold time for the sample extract is 40 days after extraction.

6.6 Criteria for Acceptance/Rejection of Samples – In addition to the above criteria, samples are compromised if they have been preserved with acid.

7.0. APPARATUS:

7.1. An Agilent 6890/7890 (or equivalent) gas chromatograph with a split/splitless injector and a 5973/5977 mass selective detector with ionization energy of 70 eV and capable of scanning 35 to 500 amu every second or less.

7.2. A Restek Rxi-5Sil MS (Restek, P/N 12738-124 or equivalent) 20 m x 0.18 mm x 0.36 µm column is used in the analysis of the extracts.

7.3. Syringes: 5 µL Autosampler (Hamilton part # 87990, or equivalent). Micro syringes: 5 µL, 10 µL, 25 µL, 100 µL, 2.5 mL, 5.0 mL (Supelco, Hamilton 1000 series (or equivalent), gas tight, high performance).

7.4. Glassware: 5.0 mL, 10.0 mL, 25.0 mL, 50.0 mL, 100.0 mL glass volumetric, class A (Kimax, or equivalent).

7.5. Autosampler vials: 2.0-mL amber crimp vial (HP part # 5181-3376, or equivalent), with autosampler crimp caps (Kimble part #73825-11, or equivalent).

7.6. For instrument maintenance and troubleshooting, refer to the instrument manual. For computer hardware and/or software, refer to IT.

8.0. REAGENTS:

8.1 Refer to SOP 112 (Standards Labeling and Traceability) for guidance on standard and reagent labeling.

8.2 Methylene Chloride ($MeCl_2$), GC grade (EM Science), or equivalent.

8.3 Stock standards are prepared by diluting purchased standard solutions in methylene chloride. Stock standards are stored at ≤ -10°C. All standards are disposed of as chlorinated waste.

8.4 Initial Calibration Standards: Allow stock standards to come to room temperature. Sonicate any that appear to have precipitated. A minimum of five calibration levels for each analyte is needed. Using volumetric syringes mix appropriate volumes to yield the working calibration standards. See Table 1 for dilution instructions and part numbers. Internal standard must be added to every standard and sample.

8.5 Continuous Calibration Verification (CCV): The CCV standard is made at 50 µg/mL (per Table 1). Compounds with higher LOQs are present at a greater concentration than 50 µg/mL. The calibration verification is made from the same source as the initial calibration. The calibration verification must be checked frequently for signs of evaporation or degradation. The calibration verification must be stored in the freezer when not in use. The calibration verification standard is made frequently to limit the effects of evaporation and degradation.

8.6 Surrogate Standard: The surrogate standard is a mixture of three acid surrogates and three base surrogates. The surrogate working standard is diluted from a certified stock to yield 200 µg/mL for the acid surrogates and 100 µg/mL for the base surrogates in the final extract. The certified surrogate stock standard is diluted for addition to the working calibration standards. Prep working surrogate: **Dilute 10 mL of Restek #33028 and 10 mL of Restek # 33029 to 500 mL with methylene chloride.**

8.7 Laboratory Control Standard (LCS): The laboratory control standard is a mixture of all reported compounds. The working LCS is diluted from certified stocks. The working LCS is made to yield a concentration of 100 µg/mL for most compounds in an extracted sample. Compounds with higher LOQs are present at a greater concentration than 100 µg/mL. The working LCS is also used for spiking client samples.

8.8 Initial Calibration Verification (ICV): The ICV standard is made at 100 µg/mL. Compounds with higher LOQs are present at a greater concentration than 100 µg/mL. The initial calibration verification is made from a different source than the initial calibration.

8.9 Tuning Standard: The working tuning standard is diluted from a certified stock. The tuning standard includes Pentachlorophenol, DFTPP, benzidine, and 4,4'-DDT. The working tuning standard has a concentration of 50 µg/mL for all compounds. Tune standard – Ultra GCM-150 1,000 µg/mL of Pentachlorophenol, DFTPP, Benzidine, and 4-4'-DDT. 50 µL is added to 950 µL of methylene chloride to yield a 50 µg/mL working solution.

8.10 Internal Standard: The working internal standard solution is purchased from Ultra scientific (US 108). 10 µl of internal standard is added to all samples and standards. If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard solution must be added to the diluted extract to maintain the same concentration as in the calibration standards. Internal Standard: Ultra US-108 4000 µg/mL.

9.0. EXTRACTION:

See Appropriate Preparation SOP

10.0. CALIBRATION:

10.1 A tune evaluation is run to begin the analytical shift. The peaks in the tune evaluation should show minimal tailing. The tailing factor for Benzidine and for Pentachlorophenol must be ≤ 2, DDT must not show breakdown over 20% (see formulas below for determining DDT breakdown). DFTPP must meet the tune criteria in Table 2. Use the autofind tune macro (average of apex, apex+1, and apex-1 and minus a background scan not within the DFTPP peak). If the autofind macro does not locate the correct peak or if the autofind is not using the appropriate scans (due to peak shape), the analyst will use a single scan located within one scan number of the apex, and background subtraction is included only to eliminate column bleed or instrument background ions. Alternatively, using the average of the scans for the peak with appropriate background subtraction is allowed. Identical mass spectrometer settings that were used to analyze the DFTPP tune criteria are used for all subsequent injections; see section 13.2.5 for equation.

10.2 Calibration is established using the internal standard technique. The area of the characteristic ion for each compound is tabulated vs. the concentration for each standard. A calibration curve is constructed from the response factors, using the calibration standards outlined in Table 1. The calibration is considered acceptable if the following criteria are met:

10.2.1 All compounds have met the recommended minimum response factor for the target analytes, as noted in Table 3.

10.2.2 The retention time of all analytes is input into a controlled spreadsheet (CS-0004_RRT_8270_Calibration_Check) to determine if all analytes are within the 0.06 RRT units

10.2.3 Linearity of Target Analytes: If the RSD of any target analyte is 15% or less, then the relative response factor is assumed to be constant over the calibration range and linearity through the origin can be assumed. The average response factor may then be used for quantitation. The average RF should not be used for compounds that have a RSD greater than 15% unless the concentration is reported as estimated. **If the RSD of any target analyte is greater than 15% than one of the following options will be used:**

- Linear (first order) least squares regression. This option requires a minimum r^2 value of 0.99 (or r value of 0.995). The linearity through the origin is assumed not forced through the origin.

- Non-linear (2^{nd} or 3^{rd} order) least squares regression. This option requires a minimum r^2 value of 0.99 (or r value of 0.995). Analyst employs a regression equation that does not pass through the origin.

10.2.4 The use of equal weighted least squares regression may be used. The deciding factor in the use of these techniques will be the residuals for the calibration points (which fit best matches the points). The residual is the difference between the known concentration of the calibration point and the calculated value based on the calibration curve. The analyst should strive to reduce the residual error at each point in the calibration to less than 20%.

10.3 Analyte retention times and qualifier ion responses are updated with every calibration using the 50 mg/L standard. The reference spectra will be updated as needed.

11.0. ANALYSIS:

11.1. **Analyte identification:** The qualitative identification of compounds in a sample is based on retention time and comparison of mass spectrum with characteristic ions in a reference spectrum after background subtraction. The reference spectrum must be generated using the same conditions as the calibration. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

11.1.1. All extracts and standards are allowed to come to room temperature. The autosampler is set to inject a 1 µL aliquot onto the chromatographic column.

11.1.2. Positive identification of target compounds is confirmed when the following criteria are met:

- The compound component should elute within 0.06 RRT units of the retention time of the authentic compound (or alkyl homologue) grouping determined by the midpoint of the initial calibration or the current continuing calibration verification.

- The relative intensities of the characteristic ions should agree within 30% from the reference spectrum. Analyst judgment should be used when interferences are observed.

11.1.3. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. The identification criteria may be met, but each compound spectrum will contain extraneous ions. Appropriate selection of compound spectra and background spectra is important.

11.1.4. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The most recent versions of the NIST library should be available. See SOP 500 for guidance.

11.1.5. The software will not always correctly identify or properly integrate the analyte peaks. When this occurs, manual integration will be necessary to accurately identify and quantitate target analytes. See SOP 144 for manual integration guidance.

11.2. Quantitation:

11.2.1. Once a target compound has been identified, the quantitation of a compound is based on the integrated abundance from the extracted ion profile (EIP) of the primary characteristic ion.

11.2.2. Internal standards are used for quantitation and generally the ISs are assigned as indicated in Method 8270D. Exceptions may occur depending on the chromatography of specific columns and/or instruments. IS assignments are generally based on retention time proximity (0.80-1.20). Exceptions to this rule must be obtained from the Technical Director.

11.2.3. If the response of the quantitation ion exceeds the calibration range, the sample extract must be diluted and reanalyzed. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Internal standards must be added so that the concentration of the IS in the dilution matches that of the undiluted samples and calibration standards.

11.2.4. If there is interference with the primary quantitation ion of the internal standard, a secondary ion can be used for quantitation.

12.0. QUALITY CONTROL:

12.1 Accuracy Measurements:

12.1.1 A laboratory control sample is analyzed with every soil and water extraction batch consisting of up to 20 samples to demonstrate that the extraction procedure is in control for the specific matrix type. Performance based limits are used to determine LCS acceptance criteria. Any LCS failure may be re-analyzed once to confirm the failure. If the failure is confirmed and the bias is high, any samples below LOQ for the affected analytes can be reported. If the failing analytes are detected above the LOQ, or if the failing analytes are biased low, the batch must be re-extracted.

12.1.2 A matrix spike and matrix spike duplicate are analyzed with every extraction batch assuming sufficient sample volume is available for generating a spike and spike duplicate set. Performance based limits are used to generate matrix spike recovery criteria. If the MS/MSD is biased high or low and the LCS meets QC criteria note in LIMS.

12.1.3 The continuous calibration verification standard (CCV) is analyzed at the beginning of each 12 hour analytical shift after the tune verification. The CCV is analyzed near the mid-point of the calibration curve (typically 50 µg/mL). The CCV is considered acceptable if the recoveries are within ±20% and the minimum response factors for each compound are met. If the CCV is outside of criteria check the system, correct the source of any problems and re-analyze the CCV. If the failure persists, any analytes that are biased high in the CCV but not detected above the LOQ in the samples may be reported. If the analytes are detected above the LOQ, or if the CCV bias is low, the affected samples must be re-analyzed with a valid CCV.

12.1.4 For DOD samples – The continuous calibration verification standard (CCV) is analyzed at the beginning of each 12 hour analytical shift after the tune verification. The CCV is analyzed near the mid-point of the calibration curve (typically 50 µg/mL). The CCV is considered acceptable if the recoveries are within ±20% and the minimum response factors for each compound are met. If the CCV is outside of criteria, check the system, the analyst may immediately (within 1 hour and before any additional samples have been acquired) reanalyze 2 successive CCV's. If both are within QC criteria for all analytes, then the samples that have already acquired may be reported and the sequence may continue. A closing CCV is required at the end of the analytical sequence and must meet ±50% recovery criteria for the analytes of interest. If any analytes are biased high in the closing CCV but are not detected above the LOQ in the samples they may be reported. If the analytes are detected above the LOQ, or the CCV is biased low, the affected samples must be re-analyzed with valid bracketing CCV's.

12.2 Precision Measurements:

12.2.1 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Spike two sample aliquots with the primary solution. The analytes should be within the LCS lab generated control limits for each analyte and the relative percent difference (RPD) criteria is 20%. MS recoveries are not used to control the acceptability of a batch. A footnote is to be generated discussing any outliers. If there is insufficient sample volume to perform a MS/MSD then a LCSD must be analyzed as part of the extraction batch.

12.2.2 RPD criteria for the LCS/LCSD is 20%. If the RPD fails QC criteria and the affected analytes are not detected above the LOQ then the analytes may be reported. If the analytes are detected above the LOQ, then the affected samples must be re-extracted.

12.3 Calibration Quality Control: Once the criteria for calibration of this SOP have been met, the following quality control must be met.

12.3.1 The initial calibration verification (ICV) must be analyzed after the successful calibration and before sample analysis. The initial calibration verification is considered acceptable if the recoveries are within ±20%, ±30% is allowed for non-DOD samples.

12.4 Blank Criteria:

12.4.1 A method blank is analyzed with every soil and water extraction batch consisting of 20 samples or less to demonstrate that the extraction procedure is free of contamination and interferences. The method blank is carried through all stages of preparation and analysis. No analytes may be detected above the LOQ (1/2 LOQ for DOD). If the MB is outside of QC criteria, re-analyze the sample to confirm. Samples with the affected analytes less than the LOQ, or greater than ten times the blank contamination, may be reported (see SOP #102). Other samples must be re-extracted.

12.4.2 An instrument blank is analyzed during each analytical shift to demonstrate that the analytical system is free from interferences or contamination. The instrument blank is made from methylene chloride fortified with the internal standards and surrogates. All target analytes in the instrument blank must be below the LOQ. The instrument blank can be run at any time during the analytical shift, especially after high concentration samples. If the IB is outside of QC criteria re-analyze the sample to confirm. Samples with the affected analytes less than the LOQ or greater than ten times the blank contamination may be reported. Samples with analytes that are detected above the LOQ but less than ten times the blank contamination must be re-analyzed once the instrument is free of contamination.

12.5 Tune Criteria: The tune criteria in section 10.1 must be met prior to every analytical run and every twelve hours.

12.6 Internal Standard (IS) Criteria: Internal standard responses must be within 50-200% of the mid point of the most recent calibration. If the IS responses are outside of QC criteria, check the chromatography for any indication that a matrix component may be affecting the IS responses. Re-analysis of the sample is required. If review of the chromatogram indicates an obvious matrix effect, the re-analysis should be performed at a dilution. If the IS responses are non-compliant in the re-analysis, further dilutions must be made. If the IS responses are compliant in the re-analysis, report the data from the re-analysis. No analytes may be reported with failing internal standard responses.

12.7 Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

12.8 Spike and surrogate standards for sample prep will be tested prior to use by the analyst. The recoveries must meet the second source calibration verification criteria ± 20% for the LCS solution, and ± 20% for the surrogate solution.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 Units:

Soil: mg/Kg
Water: mg/L
Oil: mg/Kg
TCLP: mg/L

13.2 Equations:

13.2.1 Response factor:

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

Where As = area of the standard and Ais = area of the internal standard.
Where Cs = concentration of the standard and Cis = concentration of the internal standard

13.2.2 Relative Standard Deviation:

$$RSD = \frac{SD}{Ave.} \times 100$$

Where SD = standard deviation and ave = average.

13.2.3 Linear regression equation derived for the internal standard technique:

$$\frac{AsCis}{Ais} = aCs + b$$

Where As = area of the standard and Ais = area of the internal standard.
Where Cs = concentration of the standard and Cis = concentration of the internal standard.
Where a = slope of the line and b = y intercept.

13.2.4 Peak Tailing Factors:

Peak Tailing Factor = BC/AB
10% Peak Height = BD
Peak Height = DE
Peak Width at 10% Peak Height = AC

13.2.5 DDT Breakdown:

$$\text{DDT Breakdown} = \frac{\text{Total DDT degradation peak area (DDD + DDE)}}{\text{Total degradation peak area (DDD+DDE+DDT)}} \times 100$$

13.2.6 Relative Retention Times:

$$RRT = \frac{\text{Retention time of analyte}}{\text{Retention time of IS}}$$

13.3 Review/Peer Review Steps: Peer review will be done in accordance with SOP 101.

13.4 Data Qualifiers/Flags: Data will be appropriately flagged in accordance to SOP 136.

13.5 Archive Policy (hardcopy and electronic): Data will be archived in accordance with SOP 142.

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

14.6. Many of the solvents and target analytes used in this procedure are known or suspected carcinogens. The analyst must take the responsibility to know the potential risks and use adequate protection. All extracts and standards should be diluted in the fume hood. The use of a personal respirator mask is suggested when work must be done outside of a fume hood. Frequent washing of hands is also recommended.

15.0. POLLUTION PREVENTION:

15.1 SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

15.2 Pollution prevention techniques utilized in this method are the consolidation of all sample extracts, standards, and waste material as chlorinated waste, which is disposed of by trained personnel in accordance with SOP 108.

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

17.1 DL studies are performed when a significant change in instrument response is observed or when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

17.2 The suggested DL spiking levels are: 5 µg/L, 10 µg/L, and 50 µg/L for waters and 0.222 mg/Kg and 0.444 mg/Kg for soils. **Note - The DL for samples is highly dependent on sample matrix. Matrix effects can cause the DL to be elevated.**

18.0. LIMIT OF DETECTION (LOD):

18.1 The LOD is defined per SOP 116; LOD verification shall be performed quarterly according to the schedule set by the QA Office.

19.0. LIMIT OF QUANTITATION (LOQ):

19.1 The LOQ is defined per SOP 116; LOQ verification shall be performed quarterly according to the schedule set by the QA Office.

19.2 Suggested spiking guidance for quarterly LOQ verification is as follows:

Analysis	Matrix	LOQ Spike Amount
8270	soil	50 µL
		150 µL
		250 µL
		500 µL
8270	water	100 µL
		200 µL
		300 µL
		500 µL

20.0. REFERENCES:

SW846 rev. 5 July 2014 8270D
EPA 625
DOD QSM (current version)

21.0. ATTACHMENTS:

Table 1: Standards Preparation
Table 2: Tune Criteria
Table 3: Minimum response factor criteria
Table 4: Corrective Action Table
Table 5: Analyte List and Associated Target Ions

TABLE 1 - 8270 Calibration Standards

	INDV. STD VOL*	TOTAL STD VOL	INT STD VOL	MECL2 VOL	FINAL VOL	FINAL CONC
SGS lot #	µL	µL	µL	µL		
STD 1	5	20	10	970	1 mL	5 ng/µL
STD 2	10	40	10	950	1 mL	10 ng/µL
STD 3	20	80	10	910	1 mL	20 ng/µL
STD 4	50	200	10	790	1 mL	50 ng/µL
STD 5	80	320	10	670	1 mL	80 ng/µL
STD 6	100	400	10	590	1 mL	100 ng/µL
STD 7	160	640	10	350	1 mL	160 ng/µL
STD 8 IB**	50	50	10	940	1 mL	0 ng/µL

*STANDARDS USED= 8270-2, 8270-3, 8270-4, 8270-5

**STANDARD USED= 8270-1

8270-1	Amount	Final Conc.
Acid Surrogates	400 µL	4000
Base Neutral Surrogates	400 µL	2000
Mecl2	200 µL	
	1 mL	

8270-2	Amount	Final Conc.
SVW14-2-9	500 µL	
3,3'-Dichlorobenzidine	500 µL	1000
	1 mL	

8270-3	
MegaMix	500/1000

8270-4	
Benzoic Acid	2000

8270-5	
Custom standard	1000/2000

Acid Surrogates = Restek #33029 10000 µg/mL
Base/Neutral Surrogates = Restek #3308 5000 µg/mL
MegaMix = Restek #31850 500/1000 µg/mL
Internal Standard = Ultra Scientific #US-108N 4000 µg/mL
3,3-Dichlorobenzidine = Restek #31835 2000 µg/mL
Benzoic Acid = Restek #31879 2000 µg/mL
Custom STD = Restek #565054 1000/2000 µg/mL

TABLE 2 - Tune Criteria

Mass	Ion Abundance Criteria
51	10% to 80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10% to 80% of mass 198
197	< 2% of mass of 198
198	Base peak, or>50% of Mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	> 1% of mass 198
441	Present but less than 24% of mass 442
442	> 50% of mass 198
443	15% to 24% of mass 442

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TABLE 3

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400

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TABLE 3: (continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TABLE 4: CORRECTIVE ACTION TABLE

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration for all analytes	Periodic	See Table 3 minimum response factor criteria for analytes and SPCCs % RSD Analytes ≤ 15% or r^2 ≥ 0.99 or r ≥ 0.995	1. Correct source of problem. 2. Repeat analysis. 3. Recalibrate if needed.
Second Source Cal. Verification. (ICV)	Once per initial calibration	All analytes within ±20% ±30% allowed for non-DOD projects	1. Correct source of problem. 2. Repeat analysis once. 3. Recalibrate if needed.
Continuous Calibration Verification (CCV) **DOD**	Before sample analysis, Immediately after tuner Every 12 hours **Closing CCV**	Table 3 minimum response factor criteria and all analytes ±20% recovery **±50% recovery**	1. Evaluate the data. 2. Correct source of problem. 3. Repeat analysis if needed. 4. Recalibrate if needed 5. **DOD** – Re-analyze 2 successive CCV's
Method Blank (MB)	One MB per extraction batch	Analytes < LOQ **For DOD: < ½ LOQ**	1. Evaluate the data/prep info. 2. See section 12.4.1
LCS/LCSD	One LCS per extraction batch; If insufficient volume for MS/MSD, then run LCSD	Performance-based limits RPD: 20%	Repeat analysis. If recoveries or RPD is biased high and samples are non-detect, note in LIMS. If analytes are detected or recoveries are biased low, re-extract the prep batch if possible.
MS/MSD	One MS/MSD per 20 client samples; LCSD if insufficient volume	Performance-based limits RPD: 20%	I If MS/MSD is biased high or low and LCS meets QC criteria note in LIMS. If RPD is outside of criteria, note that the results in the parent sample are estimated for any effected analytes.
Surrogate Spike	Every Sample	Performance-based limits	If surrogates are biased high and sample is non-detect note in LIMS. If surrogates are biased low, re-extract. If sample has been analyzed at a dilution and surrogates are biased note in LIMS.
Tune Standard	Prior to Initial Calibration and Calibration Verification Every 12 hours	See Table 2	1. Retune 2. recalibrate if needed 3. Re-analyze samples after 4. failing tune
Internal Standard	Every sample post extraction	See section 12.6	Evaluate the data. Correct source of problem. Dilute samples if needed
Instrument Blank (IB)	Every 12 hours	Analytes < LOQ	See section 12.4.2
Demonstration of Capability	Once per analyst initially And repeated annually	Performance-based limits	Repeat analysis or re-extract if needed.

Table 5: Analyte List and Associated Target Ions

Analyte	Primary Ion	Secondary Ion	Tertiary Ion	Quaternary Ion
1,4-Dichlorobenzene-d4	152	150	115	
N-nitrosodimethylamine	74	42	43	41
Pyridine	79	52	51	50
Aniline	66	93	65	
2-Fluorophenol	112	64	92	
Phenol-d6	99	71		
Phenol	94	66		
Bis(2-chloroethyl)ether	63	93	95	
2-Chlorophenol	128	130		
1,3-Dichlorobenzene	146	148	111	
1,4-Dichlorobenzene	146	148	111	
Benzyl alcohol	79	108	107	77
1,2-Dichlorobenzene	146	148	111	
2-methylphenol	108	107	77	
Bis(2-chloroisopropyl)ether	45	41	121	
Acetophenone	105	120		
3&4-Methylphenol	107	108	77	51
N-nitrosodi-n-propylamine	70	42	101	130
Hexachloroethane	117	201	199	166
Naphthalene-d8	136	108		
Nitrobenzene-d5	82	128	54	
Nitrobenzene	77	123	65	
Isophorone	82	95	138	
2-Nitrophenol	139	65	109	
2,4-Dimethylphenol	107	122	121	
Benzoic acid	105	122	77	
Bis(2-chloroethoxy)methane	93	95	123	
2,4-Dichlorophenol	162	164	98	
1,2,4-Trichlorobenzene	180	182	145	
Naphthalene	128	129	127	
2,6-Dichlorophenol	162	164	98	
4-Chloroaniline	65	92		
Hexachlorobutadiene	225	223	227	
4-Chloro-3-methylphenol	107	77	142	
2-Methylnaphthalene	142	141	115	
1-Methylnaphthalene	142	141	115	

Acenaphthene-d10	164	162	80	
Hexachlorocyclopentadiene	237	235	272	
2,4,6-Trichlorophenol	196	198	132	
2,4,5-Trichlorophenol	196	198	97	132
2-Chloronaphthalene	162	164	127	
1-Chloronaphthalene	162	164	127	
2-Fluorobiphenyl	172	171	170	
2-Nitroaniline	138	65	92	
Dimethylphthalate	163	194	164	
2,6-Dinitrotoluene	165	89	121	
Acenaphthylene	152	151	153	
3-Nitroaniline	138	92	65	
Acenaphthene	153	154	152	
2,4-Dinitrophenol	184	91	107	
4-Nitrophenol	65	139	109	
2,4-Dinitrotoluene	165	63	182	
Dibenzofuran	168	139		
Diethylphthalate	149	177	150	
Fluorene	166	165	167	
4-Chlorophenyl-phenyl-ether	204	206	141	77
4-Nitroaniline	138	108	92	65
Phenanthrene-d10	188	489		
4,6-Dinitro-2-methylphenol	198	105	121	
N-nitrosodiphenylamine	169	168	167	83
Azobenzene	77	182	105	51
2,4,6-Tribromophenol	330	332	328	
4-Bromophenyl-phenyl-ether	248	250	141	
Hexachlorobenzene	284	142	249	
Pentachlorophenol	266	264	268	
Phenanthrene	178	179	176	
Antracene	178	179	176	
Carbazole	167	166	168	139
Di-n-butylphthalate	149	150	104	
Fluoranthene	202	101	100	
Chrysene-d12	240	236	120	
Pyrene	202	101	100	
Terphenyl-d14	244	122	245	
Butylbenzylphthalate	149	91	206	
3,3'-Dichlorobenzidine	252	254	126	

Benzo(a)anthracene	228	226	229	
Chrysene	228	229	226	
Bis(2-ethylhexyl)phthalate	149	167	279	
Di-n-octylphthalate	149	150	279	
Perylene-d12	264	132	130	
Benzo[b]fluoranthene	252	253	125	
Benzo[k]fluoranthene	252	253	125	
Benzo[a]pyrene	252	253	125	
Indeno(1,2,3-c,d)pyrene	276	277	138	137
Dibenzo[a,h]anthracene	278	139	279	
Benzo[g,h,i]perylene	276	138	277	

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure	
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S.O.P. Title: Polynuclear Aromatic Hydrocarbons by GCMS-SIM		**Revision Date**: February 2024
Method No: 8270E-SIM, 625M-SIM		**SOP No**: 727r14
Page: 1 of 17		**Supersedes**: 727r13

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	2/1/24	Jamaica Rentty	2/1/24

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I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

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Summary of Changes from Previous Revision:

- Addendum 1 incorporated to section 8.2.2
- Changed all occurrences of 8270D-SIM to 8270E-SIM.
- Updated section 8.2.3 to current practice.
- Updated Attachment A to reflex 8270E tune criteria.
- Grammar and spelling corrected throughout.

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1.0. OBJECTIVE:

This method describes the quantitative determination of polynuclear aromatic hydrocarbons (PAHs) and their alkylated homologues in extracts of water, sediment, and biological tissues. See below for target compounds and their associated ions:

Target Compounds	**Primary Ion**	**Secondary Ion**	**Tertiary Ion**
Naphthalene	128	127	129
2-Methylnaphthalene	142	141	115
1-Methylnaphthalene	142	141	115
Acenaphthylene	152	151	153
Acenaphthene	154	152	153
Fluorene	166	165	167
Phenanthrene	178	176	179
Anthracene	178	176	179
Fluoranthene	202	101	203
Pyrene	202	200	203
Benzo(a)anthracene	228	226	229
Chrysene	228	226	229
Benzo[b]fluoranthene	252	253	125
Benzo[k]fluoranthene	252	253	125
Benzo[a]pyrene	252	253	125
Indeno[1,2.3-c,d]pyrene	276	277	138
Dibenzo[a,h]anthracene	278	279	139
Benzo[g,h,i]perylene	276	277	138
Compounds not included in Secondary Source	**Primary Ion**	**Secondary Ion**	**Tertiary Ion**
2,6-Dimethylnaphthalene	156	141	
1,6,7-Trimethylnaphthalene	170	155	
Biphenyl	154	153	
1-Methylphenanthrene	192	191	
Benzo(e)pyrene	252	253	125
Internal Standard	**Primary Ion**	**Secondary Ion**	**Tertiary Ion**
Chrysene-d_{12}	240	236	120
Acenaphthene-d_{10}	164	162	160
Surrogates	**Primary Ion**	**Secondary Ion**	**Tertiary Ion**
2-Methylnaphthalene-d_{10}	152	150	122
Fluoranthene-d_{10}	212	106	208

2.0. SCOPE AND APPLICATION:

Quantitation of polynuclear aromatic hydrocarbons (PAHs) is performed by gas chromatography-mass spectrometry (GC/MS) in the selected ion monitoring (SIM) or MS/MS mode. LOQs are at parts per trillion level (ppt, ng/L) in water, and parts per billion (ppb, µg/Kg) in sediment and biological tissues. Alkylated homologues may also be evaluated using this technique.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1 Two internal standards are used (acenaphthene-d_{10} and chrysene-d_{12}).

3.2 Two surrogates are used (fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}).

3.3 The surrogates and internal standards used are not the recommended surrogates and internal standards suggested in Table 8 of Method 625.

3.4 LCS, LCSD, MS, and MSD concentrations are at 500ng/mL.

3.5 Tune criteria is not the same as Table 9 in Method 625 but is taken from Table 3 in Method 8270E.

3.6 Extractions occur at neutral pH only.

3.7 Qualitative ID is not performed using the 3 ions listed in Tables 4 & 5 of Method 625. ID is made per Section 11.1 of this SOP.

3.8 The ICV recovery goal will be ±20 %, as per DOD QSM. Recoveries greater than ±20% but less than ±30% will be allowed for non-DOD.

3.9 Internal standard areas are monitored only from the mid-point of the most recent calibration.

3.10 Method 8270E indicates that matrix spike/matrix spike duplicates are part of the minimum batch method QC. SGS performs this QC set only if sufficient sample volume is received; otherwise an LCS/LCSD is performed.

3.11 Method 8270E states that relative retention times (RRT) of each analyte in each calibration standard should agree within 0.06 RRT units. SGS requires each calibration standard to agree within 0.06 RRT units from the mid-point of the calibration.

3.12 Abundance of characteristic ions is taken from Method 8270E, not Method 625.

3.13 Tailing for benzidine and pentachlorophenol are not evaluated unless requested by client.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1 Contaminants found in reagents, solvents, glassware and other sample/instrument processing hardware may cause discrete artifacts or elevated baselines in the SIM profile. These materials must be demonstrated to be free of interference by analysis of laboratory reagent blanks. Analysis of highly contaminated samples should be followed by instrument blanks to demonstrate carry-over contamination of instrumentation has not occurred.

5.2 Matrix interference may be caused by co-extraction of contaminants from the sample. The interference varies depending on matrix and source of the environment being sampled.

6.0. SAMPLE HANDLING:

6.1 This procedure is applicable to several types of matrices including soils, waters, solids and liquids.

6.2 Sample Collection – Aqueous samples are collected in amber 1-L or 250-mL glass containers and stored at 0-6°C until analysis. Sediment samples are collected in amber 4 - 8 oz. glass sample containers and stored at 0-6°C until analysis.

6.3 Sample Size – The normal sample sizes for water samples is 1 L or 250 mL. The normal sample size for solid samples is 22.5 grams.

6.4 Sample Preservation – There is no preservation of samples during collection except to protect them from light and store them at 0-6°C until extraction.

6.5 Holding Times – Aqueous sample extraction must occur within 7 days of collection. Soil sample extraction must occur within 14 days of collection. The hold time for the sample extract is 40 days.

6.6 Criteria for Acceptance/Rejection of Samples – Samples may not be suitable for analysis if they have not been properly handled. Consult with the Project Manager or the Technical Director concerning the usability of samples.

6.7 Sample extracts must be recapped daily if the septum is punctured.

7.0. APPARATUS:

7.1 Agilent 7890B (or equivalent) gas chromatograph equipped with an auto injection system using 1-µL injections in the split/splitless injection mode.

7.2 Column: A Restek Rxi-5Sil MS (Restek, P/N 12738-124 or equivalent) 30m x 0.25mm x 0.5um column is used in the analysis of the extract.

7.3 Detector: Agilent 5977B (or equivalent) mass selective detector operated in the SIM mode.

7.4 Syringes: 5µL Autosampler (Hamilton part # 87990, or equivalent). Micro syringes: 5-µL, 10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1-mL (Supelco, Hamilton 1000 series (or equivalent), gas tight, high performance).

7.5 Glassware: class A volumetric glassware, volumes ranging from 5.0 mL to 1000.0 mL (Kimax, or equivalent).

7.6 Autosampler vials: 2.0-mL amber crimp vial (Thermo Scientific part# 60180-VT302A, or equivalent), with autosampler crimp caps (Agilent part# 5061-3370, or equivalent).

7.7 For instrument maintenance and troubleshooting see the instrument manual. For computer hardware, software, etc. troubleshooting, refer to IT.

8.0. REAGENTS:

8.1. Methylene Chloride ($MeCl_2$), GC grade Fisher Chemical, (or equivalent).

8.1.1. Methanol (MeOH): Purge and trap grade, Fisher Chemical (or equivalent) High Quality Solvent.

8.2. Stock Standard Solutions: All standards are prepared in $MeCl_2$ or MeOH and are labeled according to SOP#112. Stock standards, sample prep spikes, surrogate solutions, and working standards are stored away from any light source at -10ºC or below. Allow standards to come to room temperature prior to analysis or prior to the preparation of working standards.

8.2.1. **Water surrogate Solution (SU):** Prepare by diluting 1000 µg/mL standard A (Absolute Standards, Inc. part # 20016, or equivalent) and 2000 µg/mL standard B (Restek part# 33913, or equivalent). Dilute 1.0mL of standard A and 0.5 mL of standard B to 10mL with $MeCl_2$ to give an SU stock solution of 100 µg/mL. Dilute 2.5 mL of the 100 µg/mL SU stock to 500 mL with $MeCl_2$ to make the working surrogate standard. The compounds in the SU are 2-fluorobiphenyl, terphenyl-d_{14}, 2-methylnaphthalene-d_{10}, and fluoranthene-d_{10}. Each sample receives a 1.0-mL aliquot of the SU working standard prior to sample extraction to give a final concentration of 500 ng/mL in a 1-mL extract. The surrogate standard is tested by the analyst prior to use and the recovery must be ±20%.

8.2.2. **Soil surrogate Solution (SU):** Use Sigma Chemical Company or equivalent certified product. Use 1000 ug/mL standard A (Absolute Standards, Inc. part# 20016, or equivalent) and 2000 ug/mL standard B (Restek part# 33913, or equivalent). Dissolve 0.250 g of 5α-androstane and 0.250 g of triacontane-d_{62} (DTC) into 5 mL of carbon disulfide and sonicate in a warm water bath. Dilute this solution with methylene chloride to the neck of a 500 mL volumetric flask. Let this solution sit overnight. Add 1.25 mL of standard A and 0.625 mL of standard B to the flask and dilute to a final volume of 500 mL. The compounds in the solution are 2-fluorobiphenyl, terphenyl-d14, 2-methylnaphthalene-d10, fluoranthene-d10, 5α-androstane, and triacontane-d62 (DTC). Each sample receives a 1.0 mL aliquot of the SU standard prior to sample extraction to give a final concentration of 100 µg/mL for 5α-androstane and DTC, and a final concentration of 500 ng/mL for 2-fluorobiphenyl, terphenyl-d14, 2-methylnaphthalene-d10, and fluoranthene-d10 in a 5 mL extract. The surrogate standard is tested by the analyst prior to use and the recovery must be ±20%.

8.2.3. **Initial Calibration Verification (ICV):** Typically prepared by diluting a 2000 µg/mL standard from a secondary source (Restek part #31995 and SupelCo part #48161) to a concentration of 0.5 µg/mL with MeCl2. The ICV is ran immediately following each calibration, and the recovery must be 20% for DOD samples. A recovery of 30% is acceptable for non-DOD sample analysis.

8.2.4. **Laboratory Control Standard Solution (LCS):** Typically prepared by diluting a 1000 µg/mL standard (Absolute Standards part # 93462). This standard is a solution of 23 PAH compounds (see Section 1.0). Dilute 100 µL of the standard to 200 mL with MeOH to give a waters LCS

working solution of 0.50 µg/mL. Dilute 500 µL of the standard to 200 mL with MeOH to give a soil LCS working solution of 2.50 µg/mL. Each LCS receives a 1.0-mL aliquot of the matrix-specific LCS working solution prior to sample extraction to give final concentrations of 0.500 µg/mL for each 1-mL waters extract and 0.500 µg/mL for each 5-mL soils extract. The LCS spiking standard is tested by the analyst prior to use and the recovery must be ±20%.

8.2.4.1. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A matrix spike is defined as a spiked paying (client) sample. A matrix spike and matrix spike duplicate are analyzed with every extraction batch, assuming sufficient sample volume is available for generating a spike and spike duplicate set. Extraction batches with insufficient sample volume for an MS/MSD will have an LCS Duplicate (LCSD) included instead. MS/MSDs are spiked for extraction according to the above section.

8.2.5. **Internal Standard Solution (IS)**: Typically prepared by diluting a 1000 µg/mL standard (Restek part # 31825, or equivalent). Dilute 1.0 mL of the standard to 25 mL with $MeCl_2$ to give an IS working solution of 40.0 µg/mL. Only chrysene-d_{12} and acenaphthene-d_{10} are used as the internal standards for this analysis. Each post-extraction batch QC sample and paying sample receives a 10.0-µL aliquot of the IS solution prior to analysis to give a final concentration of 400 ng/mL in each 1-mL extract.

8.2.6. **Calibration Standard Solution (CSS):** Prepare by diluting a 1000 µg/mL standard (Absolute Standards, Inc. part #93462, or equivalent). Dilute 100 µL of the standard to 25 mL with $MeCl_2$ to give a CSS working solution of 4.0 ng/µL. Appropriate volumes of CSS, SU, and IS are added to prepare 1 mL of each calibration standard at concentrations of 5, 10, 50, 100, 250, 500, 1000, 2000, and 2500 ng/mL. See PAH SIM Calibration Dilution table (Attachment B) for standards' preparations.

8.2.7. **Continuing Calibration Verification (CCV):** The 500 ng/mL calibration standard is used as the CCV. See PAH SIM Calibration Dilution table (Attachment B) for standard preparation.

8.2.8. **Tuning Standard Solution (Tune):** Prepared by diluting a 1000 µg/mL standard (Restek part# 31615, or equivalent). Dilute 50µL of the standard to 1 mL with $MeCl_2$ to give a Tune working solution of 50.0 ng/µL. Internal standard is not added to the tune standard. A 1-µL injection of this standard used to evaluate DFTPP criteria (see Section 10.1).

9.0. EXTRACTION:

See Appropriate Preparation SOP.

10.0. CALIBRATION:

10.1. A tune evaluation is run to begin the analytical shift of 12 hours. DFTPP must meet the tune criteria in Attachment A. Use the autofind tune macro (average of apex, apex +1, and apex -1 and minus a background scan not within the DFTPP peak). If the autofind macro does not locate the correct peak or if the autofind is not using the appropriate scans (due to peak shape), the analyst will use a single scan located within one scan number of the apex, and background subtraction is included only to eliminate column bleed or instrument background ions. Alternatively, using the average of the scans for the peak with appropriate background subtractions is allowed. Identical mass spectrometer settings that were used to analyze the DFTPP tune criteria are used for all subsequent injections. The breakdown of the DDT peak is also evaluated through the tune; analysis

may proceed if the DDT degradation is less than 20%. If the tailing factor for benzidine and for pentachlorophenol is requested by the client, it must be less than 2.

10.2. A calibration (minimum of 5 points) is analyzed to establish the linear range of the detector. Typical standard concentrations are 5, 10, 50, 100, 250, 500, 1000, 2000, and 2500 ng/mL. Calibrate using the internal standard procedure by injecting 1.0 µL of each of the calibration standards and tabulating the area of the quantitation ion against the concentration of each compound and its corresponding internal standard, determined by retention time proximity. See Attachment B for PAH SIM Calibration Dilutions. Verify the accuracy of the curve by analyzing a second source standard, also referred to as the initial calibration verification or ICV (see Section 12.3.1).

10.3. The average relative response factor (RRF) for all analytes is computed relative to the internal standard. The average RRF for all analytes should meet the criteria set for the minimum response factor (RF), listed in Attachment C. A controlled spreadsheet (CS-0005_8270_ICV_Verification_rXXXXX) is used as reference to confirm the minimum average RF for the ICV. The percent relative standard deviation (%RSD) for the RRFs for all calibrated analytes must not exceed 15%, or $r \geq 0.995$ ($r^2 \geq 0.99$), with a 1st or 2nd order fit.

10.4. Relative retention times (RRT) for all calibration standards must agree within 0.06 RRT units from the mid point of the calibration curve. The RRT of all analytes is input into a controlled spreadsheet (CS-0004_RRT_8270_Calibration_Check_XXXXX) to determine if analytes are within the 0.06 RRT criteria. See Section 13.2.2 for the RRT calculation.

10.5. Analyte retention times are updated with every calibration using the 500ng/mL standard. The reference spectra will be updated from this standard as needed.

10.6. The mass spectrometer response for each calibrated compound is verified at the beginning of each 12-hour period at a concentration of 500 ng/mL using the continuing calibration verification (CCV) solution. Daily response factors for each compound are compared to the average response of the initial calibration. If the daily response compared to initial response is within ±20%, analysis may proceed.

11.0. ANALYSIS:

11.1 Qualitative: The extracted ion current profiles of the primary m/z and the confirmatory ion for each analyte must meet the following criteria:

11.1.1 The component elutes within 0.06 RRT units of the retention time of the authentic compound (or alkyl homologue) grouping determined by the midpoint of the calibration.

11.1.2 Primary and secondary ion relative intensities are compared to the relative intensities in the reference spectrum. The intensities must be greater than 10% relative intensity. They should agree within ± 30% of the relative intensities of these masses in a reference mass spectrum. Analyst judgment should be used when interferences are observed. Only the primary ion is used for quantitation.

11.1.3 The software will not always correctly identify or properly integrate the analyte peaks. When this occurs, manual integration will be necessary to accurately identify and quantitate target analytes. See SOP #144 for manual integration guidance.

11.2 Quantitative:

11.2.1 Quantitation of a compound is based on the integrated abundance of the primary characteristic ion and is accomplished by the internal standard technique to generate RRFs. See Section 13.2.1 for the RRF equation.

11.2.2 If the response of the quantitation ion exceeds the calibration range, the sample extract must be diluted and reanalyzed. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Internal standards must be added so that the concentration of the IS in the dilution matches that of the undiluted samples and calibration standards (see Section 8.2.4).

11.2.3 If there is interference with the primary quantitation ion of the internal standard, the extract should be minimally diluted until the interference is minimized.

12.0. QUALITY CONTROL:

12.1 Accuracy Measurements:

12.1.1 A laboratory control sample (LCS) is analyzed with every soil and water extraction batch consisting of up to 20 paying samples to demonstrate that the extraction procedure is in control for the specific matrix type. Performance based limits are used to determine LCS acceptance criteria. Any LCS failure may be re-analyzed once to confirm the failure. If the failure is confirmed and the bias is high, any samples below LOQ for the affected analytes can be reported. If the failing analytes are detected above the LOQ, or if the failing analytes are biased low, the associated samples must be re-extracted.

12.1.2 A matrix spike and matrix spike duplicate (MS/MSD) are analyzed with every extraction batch assuming sufficient sample volume is available for generating a spike and spike duplicate set. If there is insufficient sample volume for the MS/MSD set, an LCSD is extracted instead. Performance based limits are used to generate matrix spike recovery criteria. If MS/MSD is biased high or low and LCS meets QC criteria, comment in LIMS.

12.1.3 For DOD and non-DOD samples – The CCV is run at the beginning of each 12-hour analytical shift in the analytical lot after the tune verification. The CCV is equivalent the mid-point of the calibration curve (typically 500 ng/mL). The CCV is considered acceptable if the recoveries are within ± 20% and the minimum response factors for each compound are met. If the CCV is outside of criteria check the system, correct the source of any problems and re-analyze. If the failure persists, any analytes that are biased high in the CCV but not detected above the LOQ in the samples may be reported. If the analytes are detected above the LOQ, or if the CCV bias is low, the affected samples must be re-analyzed with a valid CCV.

12.1.4 For DOD samples – The criteria listed above must be met. A closing CVC is required at the end of the run and must meet ±50% criteria for analytes of interest. If the CCV or CVC is outside of criteria and no problem source is found, the analyst may immediately (within 1 hour and before any samples have acquired) rerun 2 successive CCVs/CVCs. If both CCVs meet QC criteria for all analytes, then the samples that have already acquired may be reported and the sequence may continue. If both CVCs meet QC criteria, then all DOD samples that have acquired may be reported. Analytes that do not meet the CVC criteria must be re-run with a valid CVC.

12.1.5 Surrogate quality control: The deuterated PAH surrogate compounds (deuterated fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}) are spiked into all paying and quality control samples prior to extraction or analysis. The surrogate compounds measure sample matrix effects associated with sample preparation and analysis. The recovery of the surrogates is monitored in each sample using

the response of the IS (acenaphthene-d_{10} and chrysene-d_{12}) that is added to the final extract. Surrogate failures may be re-analyzed once to confirm the failures. If the failures are confirmed and the bias is high, any analytes below LOQ can be reported. If the analytes are detected above the LOQ, or if the failing surrogates are biased low, the sample must be re-extracted.

12.2 Precision Measurements:

12.2.1 Relative Percent Difference (RPD) criteria for the MS/MSD is a maximum of 20. If the RPD fails QC criteria, comment on the parent sample being non-homogenous and qualify any detectable results as estimated.

12.2.2 If insufficient sample volume is provided for a MS/MSD, then a duplicate laboratory control sample is extracted. RPD criteria for the LCS/LCSD is a maximum of 20. If the RPD fails QC criteria and the affected analytes are not detected above the LOQ then the analytes may be reported. If the analytes are detected above the LOQ, then the affected samples must be re-extracted.

12.3 Calibration Quality Control: Once the criteria for the calibration of this SOP have been met, the following quality control must be met.

12.3.1 The initial calibration verification (ICV) must be analyzed after the successful calibration and before sample analysis. The ICV and associated calibration is considered acceptable if the recoveries are within ±20%. Recoveries of ±30% will be allowed for non-DOD work.

12.4 Blank Criteria:

12.4.1 A method blank (MB) is analyzed with every soil and water extraction batch consisting of 20 samples or less to demonstrate that the extraction procedure is free of contamination and interferences. The MB is carried through all stages of preparation and analysis. No analytes may be detected above the LOQ (1/2 LOQ for DOD). If the MB is outside of QC criteria, re-analyze the MB to confirm. Samples with the affected analytes less than the LOQ, or greater than ten times the blank contamination, may be reported (see SOP#102). Other samples must be re-extracted.

12.4.2 An instrument blank (IB) is analyzed before each analytical shift to demonstrate that the analytical system is free from interferences or contamination. The instrument blank is made from $MeCl_2$ and fortified with the internal standards and surrogates. Target analytes in the IB must be below the LOQ (1/2 LOD for DOD). The surrogate recoveries for the IB must also be within ±20%. If the IB is outside of QC criteria re-analyze the IB to confirm. Samples with the affected analytes less than the LOQ or greater than ten times the blank contamination may be reported. Other samples must be re-extracted. A blank can be run at any time during the analytical shift, especially after high concentration samples.

12.5 Spike and surrogate standards for sample prep are tested prior to use by the analyst. Surrogate recovery criteria and LCS spike recovery criteria is ±20% for every target analyte (see Sections 12.1.1 and 12.1.5). Recovery criteria for surrogates and spikes (excluding IB and CCV solutions) are performance based, respective to the sample matrix. Refer to Horizon-LIMS database for matrix-specific recovery criteria.

12.6 Tune Criteria: The tune criteria in Attachment A must be met prior to every analytical run. The tune criteria must be analyzed every twelve hours; samples analyzed outside of the tune's 12-hour window must be re-analyzed. If tune criteria are not met, the tune can be re-injected once before any samples have acquired. If tune criteria are still not met, maintenance must be performed and prior to re-analysis.

12.7 Internal Standard (IS) Criteria: Internal standard responses must be within 50-200% of the mid point of the most recent calibration. If the IS is outside of QC criteria, check the chromatography for any indication that a matrix component may be affecting the IS response. If review of the chromatogram indicates an obvious matrix effect which cannot be corrected through manual integration, re-analysis should be performed at a dilution. If the IS is non-compliant in the re-analysis, further dilutions must be made. If the IS response is compliant in the re-analysis, report the associated analyte data from the re-analysis. No analytes may be reported with failing internal standards.

12.8 Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1 Units – Soil: ug/Kg, Water: ug/L

13.2 Equations –

13.2.1 Response factor:

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

Where As = area of the standard and Ais = area of the internal standard.
Where Cs = concentration of the standard and Cis = concentration of the internal standard

13.2.2 $$RRT = \frac{\text{Retention time of analyte}}{\text{Retention time of IS}}$$

13.2.3 Relative Standard Deviation

$$RSD = \frac{SD}{Ave.} \times 100$$

Where SD = standard deviation and ave = average

13.3 Review/Peer Review Steps – Peer review will be done in accordance with SOP# 101.

13.4 Data Qualifiers/Flags – Data will be appropriately flagged in accordance to SOP# 136.

13.5 Archive Policy (hardcopy and electronic) – Data will be archived in accordance with SOP# 125.

13.6 DDT Breakdown:

$$\text{DDT Breakdown} = \frac{\text{Total DDT degradation area (DDD+DDE)} \times 100}{\text{Total degradation peak area (DDD+DDE+DDT)}}$$

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP# 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

14.6. Many of the solvents and target analytes used in this procedure are known or suspected carcinogens. The analyst must take the responsibility to know the potential risks and use adequate protection. All extracts and standards should be diluted in the fume hood. The use of a personal respirator mask is suggested when work must be done outside of a fume hood. Frequent washing of hands is also recommended.

15.0. POLLUTION PREVENTION:

15.1. SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

15.2. Pollution prevention techniques utilized in this method are the consolidation of all sample extracts, standards, and waste material as chlorinated waste, which is disposed of by trained personnel in accordance with SOP# 108.

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

17.1. DL studies are performed when a significant change in instrument response is observed or when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP# 116.

17.2. Suggested DL spiking levels are 1.11 ug/Kg for soils and 0.0063 ug/L for waters. Note - The DL for samples is highly dependent on sample matrix. Matrix effects can cause the DL to be elevated.

18.0. LIMIT OF DETECTION (LOD):

The LOD is defined per SOP# 116; LOD verification shall be performed quarterly according to the schedule set by the QA Office.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP# 116; LOQ verification shall be performed quarterly according to the schedule set by the QA Office.

20.0. REFERENCES:

SW846 rev. 6 June 2018 8270E
EPA 625
DOD QSM (current version)

21.0. ATTACHMENTS:

ATTACHMENT A: DFTPP Tuning and Mass Calibration
ATTACHMENT B: PAH SIM Calibration Dilutions
ATTACHMENT C: Minimum Response Factors
ATTACHMENT D: CORRECTIVE ACTION TABLE

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ATTACHMENT A: DFTPP Tuning and Mass Calibration

Mass	Ion Abundance Criteria
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
197	Less than 2.0% of mass 198
198	Base peak, or Present
199	5.0 - 9.0% of mass 198
365	Greater than 1% of Base Peak
441	Less than 150% of mass 443
442	Base peak, or Present
443	15 - 24% of mass 442

ATTACHMENT B: PAH SIM Calibration Dilutions

SGS Lot #		STD* VOL µL	**STD 2*** ** VOL µL	SURR** VOL µL	IS VOL µL	TOTAL STD VOL µL	$MECL_2$ VOL µL	FINAL VOL	FINAL CONC
SVW00-00-01	STD 1	0	500	0	5	505	495	1 mL	5 ng/mL
SVW00-00-02	**STD 2*** **	2.5	0	2.5	10	15	985	1 mL*	10 ng/mL
SVW00-00-03	STD 3	12.5	0	5	10	27.5	972.5	1 mL	50 ng/mL
SVW00-00-04	STD 4	25	0	10	10	45	955	1 mL	100 ng/mL
SVW00-00-05	STD 5	62.5	0	25	10	97.5	902.5	1 mL	250 ng/mL
SVW00-00-06	STD 6	125	0	50	10	185	815	1 mL	500 ng/mL
SVW00-00-07	STD 7	250	0	100	10	360	640	1 mL	1000 ng/mL
SVW00-00-08	STD 8	500	0	200	10	710	290	1 mL	2000 ng/mL
SVW00-00-09	STD 9	625	0	250	10	885	115	1 mL	2500 ng/mL
SVW00-00-10	IB	0	0	50	10	60	940	1 mL	0 ng/mL

ATTACHMENT C: Minimum Response Factors

Semivolatile Compounds	Minimum Response Factor (RF)[1]
Naphthalene	0.700
2-Methylnaphthalene	0.400
Acenaphthylene	0.900
Acenaphthene	0.900
Fluorene	0.900
Phenanthrene	0.700
Anthracene	0.700
Fluoranthene	0.600
Pyrene	0.600
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo[b]fluoranthene	0.700
Benzo[k]fluoranthene	0.700
Benzo[a]pyrene	0.700
Indeno[1,2,3-c,d]pyrene	0.500
Dibenzo[a,h]anthracene	0.400
Benzo[g,h,i]perylene	0.500

(1) SW846, Method 8270E Table 4

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ATTACHMENT D: CORRECTIVE ACTION TABLE

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration for all analytes	Periodic	% RSD ≤ 15% or $r^2 \geq 0.99$ or $r \geq 0.995$ Minimum response factors meet criteria in Attch. C.	Correct source of problem. Repeat analysis. Recalibrate if needed. See Section 10.
Second Source Initial Calibration Verification (ICV)	Once per calibration	All analytes within ±20% for DOD. All analytes within ±30% allowed for non-DOD.	Correct source of problem. Repeat analysis before additional data is acquired. Recalibrate if needed. See Section 12.3.1.
Tune	At the beginning of each 12-hour period in the analytical lot.	See Attch. A. DDT breakdown is <20% for benzidine and pentachlorophenol (less than 2) evaluated upon client request.	Evaluate using background subtraction (see Section 10.1). Re-analyze once. Redo system maintenance and re-analyze (see Section 12.6). Adjust tune and recalibrate if needed.
Continuing Calibration Verification (CCV and CVC)	At the beginning of each 12-hour period in the analytical lot. **For DOD:** a CVC is also required at the end of the run.	All analytes within ±20% **For DOD:** Target analytes within ±50% for closing CVC.	Evaluate the data. Correct source of problem. Repeat analysis if needed. Recalibrate if needed. **For DOD:** Re-run 2 successive CCVs or CVCs. See Sections 12.1.3-12.1.4.
Method blank (MB)	One MB per extraction batch consisting of 20 paying samples or less	Analytes below LOQ **For DOD: < ½ LOQ**	Evaluate the data/prep info. Correct source of problem. See Section 12.4.1.
Laboratory Control Standard (Duplicate) (LCS/LCSD)	One LCS per extraction batch; if insufficient volume for MS/MSD, then LCSD is performed.	Performance based limits. RPD≤20.	Repeat analysis. If recoveries or RPD is biased high and samples are non-detect, note in LIMS. If analytes are detected or recoveries are biased low, re-extract the prep batch if possible.
Matrix Spike (Duplicate) (MS/MSD)	One MS/MSD pair per extraction batch; if insufficient volume for MS/MSD, then LCSD is performed.	Performance based limits. RPD≤20	If MS/MSD is biased high or low and LCS meets QC criteria, note in LIMS. If RPD is outside of criteria, note that the results in the parent sample are estimated for any affected analytes. See Sections 12.1.2, 12.2.1, 12.2.2.
Surrogate Spike	Every Sample.	Performance based limits.	Correct source of problem. Repeat analysis if needed. Re-extract failing samples if needed. See Section 12.1.5.
Internal standards	Every Sample.	50-200% from mid-point of the calibration	Correct source of problem. Adjust tune. Re-calibrate if needed.
Instrument blank	1 per analytical lot	Surrogates within ±20%. Analytes below the LOQ.	See section 12.4.2

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Continuous Liquid-Liquid Extraction for Semi-Volatile Compounds	**Revision Date:** April 2023
Method No: SW846 3520C	**SOP No:** 759r22
Page: 1 of 19	**Supersedes:** 759r21

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	4/17/23	Jamaica Rentty	4/17/23

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Addendum #1 added 6/15/23 by TPR

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Signatures below reflect approval for the following changes to the current SOP. These changes will be incorporated into the SOP during the next review. This addendum will be incorporated into the electronic SOP (i.e., PDF file).

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	6/13/23	Samara Petty	6/13/23

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

The above referenced SOP should be modified as follows:

Change from:

9.6. Assemble stopcocks and add 250mL of methylene chloride to the bottom of each extractor body. Add one Teflon® chip to each jacketed concentrator and ½ inch of granular Na_2SO_4 to all salt chambers. ~~Attach the bottom of the salt chamber to the jacketed concentrator using a Keck clip and the top of the salt chamber to the arm of the extractor body using another clip. Thread the two heated glycol hoses into the jacketed concentrators and tighten the hoses by hand only. The top hose goes to the top threaded stem and the bottom hose goes to the bottom threaded stem.~~

To:

9.6. Assemble stopcocks and secure bodies to the bench. Mark each body with its intended sample number. Add one Teflon® chip to each jacketed concentrator and ½ inch of granular Na2SO4 to all salt chambers.

Change from:

9.11. Verify the sample ID on the container matches what is written on the jacketed concentrator ("bell") before pouring the entire spiked sample into the designated, clean extractor body. Carefully pour the liquid so it does not go into the arm of the extractor body leading to the salt chamber. If the sample volume is significantly greater than 1 liter (e.g., larger than 1-liter sample container) measure 1 liter of sample with a solvent rinsed graduated cylinder. If the volume of the sample to be extracted is significantly less than 1 liter, dilute to 1 liter with de-ionized water. Verify sample ID with backlog to ensure procedure/sample requires lower volume. After pouring the sample into the extractor body rinse the sample container with DI water to remove any sediment, followed by 3 methylene chloride rinses, decanting each rinse into the extractor body. Rinse the cap to each jar with methylene chloride to ensure hydrocarbons present on the Teflon lid are extracted with the sample. **NOTE: if aliquot is taken, the sample container and cap will NOT be rinsed. This may bias the results.** Finally, rinse the neck of the extractor body to remove any sediment impeding the condenser from sealing properly: first with DI water and then with methylene chloride.

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To:

9.11. Remove one body from the bench and clamp it to the apparatus in the hood. Add 250mL of methylene chloride to the bottom of the extractor body. Verify the sample ID on the container matches what is written on the jacketed concentrator ("bell") before pouring the entire spiked sample into the designated, clean extractor body. Carefully pour the liquid so it does not go into the arm of the extractor body leading to the salt chamber. If the sample volume is significantly greater than 1 liter (e.g., larger than 1-liter sample container) measure 1 liter of sample with a solvent rinsed graduated cylinder. If the volume of the sample to be extracted is significantly less than 1 liter, dilute to 1 liter with de-ionized water. Verify sample ID with backlog to ensure procedure/sample requires lower volume. After pouring the sample into the extractor body rinse the sample container with DI water to remove any sediment, followed by 3 methylene chloride rinses, decanting each rinse into the extractor body. Rinse the cap to each jar with methylene chloride to ensure hydrocarbons present on the Teflon lid are extracted with the sample. **NOTE: if aliquot is taken, the sample container and cap will NOT be rinsed. This may bias the results.** Carefully detach the body from the hood apparatus and re-clamp to the bench. Attach the bottom of the salt chamber to the jacketed concentrator using a Keck clip and the top of the salt chamber to the arm of the extractor body using another clip. Thread the two heated glycol hoses into the jacketed concentrators and tighten the hoses by hand only. The top hose goes to the top threaded stem and the bottom hose goes to the bottom threaded stem. Fill the body with DI water until the water level reaches just below the arm. Finally, rinse the neck of the extractor body to remove any sediment impeding the condenser from sealing properly: first with DI water and then with methylene chloride.

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Summary of Changes from Previous Revision:

- Addendum from previous revision incorporated
- Section 2.0 and 3.1 were edited
- Updated Attachment C to current practice
- Minor grammar and formatting edits throughout.

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1.0. OBJECTIVE:

This method is used to isolate organic compounds from aqueous samples. This method is applicable to the isolation and concentration of water soluble and slightly soluble organics in preparation for a variety of chromatographic procedures.

2.0. SCOPE AND APPLICATION:

This SOP is suitable for subsequent analysis with method SW8270D, EPA625, PAH by 8270D-SIMS, 8270D Pest SIMS, 1625B Sulfolane Isotopic Dilution, 8015C, AK102/103 and 8082A. A measured volume of sample, usually 250 mL or 1 liter, is placed into continuous liquid/liquid extractor, adjusted to a specific pH (see extraction summary page), and extracted with organic solvent that has a greater density than the sample, for 4-6 hours or 16 hours. The extracts are then concentrated and analyzed with gas chromatography or gas chromatography/mass spectroscopy.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. The continuous liquid to liquid extraction run time is at least 4 hrs and a maximum of 6 hrs. DRO/RRO and Sulfolane samples will be run for 16 hours. The reference method states that the extraction system may run for 18-24 hrs. Method based performance (e.g. LCS recoveries, PT studies) supports the efficiency of SGS procedure.

3.2. 8270D samples are extracted 2.5 hrs at pH <2 and then for at least 1.5 hrs at pH>11, for a combined time of 4 hrs. Extraction should go no longer than 6 hrs. Method 3520C specifies an extraction period of 18-24 hrs at pH of <2 and at the second pH level of >11 an additional 18-24 hrs extraction is required. Method based performance (e.g. LCS recoveries, PT samples) supports the efficiency of SGS procedure.

3.3. The continuous liquid to liquid extraction currently uses 250mL of methylene chloride to each extractor body. Method 3520C states to add 300-500mL of methylene chloride to each extractor body. Industry standards and best practices use smaller amounts to reduce chlorinated solvent waste.

3.4. The drying column used has a salt ball chamber with fritted glass disc, which is baked at 400°C for a minimum of 3 hrs. The reference method uses a drying column with Pyrex® glass wool at the bottom and a PTFE stopcock.

3.5. The continuous liquid to liquid extraction apparatus, all related glassware, and the technique were custom made for the extraction process.

3.6. The concentration apparatus used is TurboVap by BioTage (or equivalent XcelVap by Horizon Technology) using compressed nitrogen gas. The reference method lists the use of a Kuderna-Danish (K-D) apparatus for sample concentration using a heated water bath with concentric ring covers.

3.7. DRO/RRO samples, for both AK102/103 and 8015C, are collected in HCl-preserved bottles; therefore, associated extraction QC are adjusted to pH <2 with 4.0 mL of HCl.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Samples that contain high hydrocarbon content may emulsify or otherwise interfere with surrogate/spike recovery.

5.2. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH and decrease with shorter extraction time.

5.3. Samples containing a significant amount of solids may interfere with the extraction process.

6.0. SAMPLE HANDLING:

6.1. Sample Collection – Samples should be unaltered prior to extraction. Aqueous samples are stored at 0-6ºC until extraction.

6.2. Sample Size – 1L amber glass jars with Teflon-lined lids. Small volume DRO/RRO AND PAH or samples with a history of high concentrations of the analyte of interest, may be submitted in 250-mL amber glass jars with Teflon-lined lids.

6.3. Sample Preservation – Water samples are stored at 0-6°C until extraction. Prior to extraction water sample collection should include preservation as follows:

Method	Preservative
8270D / EPA625/ Sulfolane	Cool 0-6°C
8270D-SIM	Cool 0-6°C,
8082A /EPA608	Cool 0-6°C,
8015C / AK102 / AK103	Cool 0-6°C, acidic pH (less than 2) by 1:3 HCl

Hexane extracts are refrigerated at 0-6ºC. Methylene chloride extracts are cooled to $\leq$ -10ºC.

6.4. Holding Times – Unpreserved water samples for all analysis except PCB must be extracted within 7 days of sample collection. There is a 1 year holding time for extraction of PCBs. Water samples collected for

AK 102 and/or AK103 that have been acid preserved must be extracted within 14 days of sample collection. Extracts must be analyzed within 40 days.

6.5. Criteria for Acceptance/Rejection of Samples – If samples have broken hold time, were not preserved properly, or were not stored at 0-6ºC, notify the Project Manager (PM) before proceeding with extraction.

7.0 APPARATUS:

7.1. 1500 mL-Glass extractor body with Teflon® stop-cocks

7.2. Coil condensers

7.3. Salt ball chambers ("salt balls")

7.4. Jacketed concentrators ("bells")

7.5. Teflon® chips (for use as boiling chips)

7.6. Pyrex glass concentrator tubes with a 1mL (ungraduated) tip

7.7. Concentrator Apparatus: TurboVap by Biotage (compressed nitrogen gas source), or equivalent.

7.8. Circulating glycol refrigeration unit (used in coil condenser)

7.9. Circulating heated glycol system (used in jacketed concentrators)

7.10. 1mL syringes: Hamilton gastight, certificate of conformance stored in CDS, as stated in SOP 104

7.11. Autosampler vials: Virtuoso, amber and clear, with Teflon®-lined crimp tops

7.12. Graduated cylinders class A.

7.13. Disposable glass pipettes: 2, 5, and 10-mL

7.14. Disposable Pasteur pipettes, 7 and 9 inches

7.15. Permanent, solvent-resistant marker (Sharpie Industrial)

7.16. Teflon wash bottles, 500mL, labeled 'methylene chloride' or 'D.I water' accordingly: FILLED DAILY

7.17. Plastic and metal Keck clips

7.18. Fisher Brand screw top vial, 17X60mm, 2-dram, w/rubber lined cap. (5mL vial)

8.0. REAGENTS:

8.1. Dichloromethane (CH2Cl2 or DCM): Fisher Optima (or equivalent) chromatographic grade.

8.2. n-Nonane: Acros 99% or equivalent (PAH analysis keeper)

8.3. De-ionized water (organic free)

8.4. Granular anhydrous sodium sulfate (Na_2SO_4): JT Baker or Fisher (or equivalent), received and opened dates must be recorded on the side of container. Heat at 400°C for ≥3 hours in a shallow tray, record in the Prep working reagents book. Label jars with ID number, date, and initials.

8.5. Sodium hydroxide solution (10N): Dissolve 400g NaOH in 1000mL organic-free water. Record information in the Prep Working Reagents logbook.

8.6. Hydrochloric acid solution 1:1 HCl: Add 500mL HCl to 500mL organic-free water. Record lot number information in the Prep Working Reagents logbook. (Only applicable to DRO/RRO extraction)

8.7. Sulfuric acid solution 1:1- Add 500mL H_2SO_4 to 500mL organic-free water. Record lot number information in the Prep Working Reagents logbook.

8.8. Hexane (C_6H_{14}): Fisher Optima (or equivalent) high purity Pesticide grade

9.0. EXTRACTION: *(Attachment B: Flow Chart)*

9.1. (#1) Approximately thirty minutes before extraction, turn the red knob on the heater control box to "**on**."

9.2. (#2) Turn the yellow flow control levers under the extraction benches being used to "**extraction**."

9.3. (#3) Check to make sure all green A & B valves under the extraction benches are turned OFF. Turn the yellow flow control handles inside the pump cabinet to "**extraction".**

9.4. (#4) Flip the switch located on the end of the cabinet to "**fill"**.

9.5. (#5) Prepare all glassware, according to the SGS SOP#117 "Glassware Cleaning and Preparation."

9.6. Assemble stopcocks and add 250mL of methylene chloride to the bottom of each extractor body. Add one Teflon® chip to each jacketed concentrator and ½ inch of granular Na_2SO_4 to all salt chambers. Attach the bottom of the salt chamber to the jacketed concentrator using a Keck clip and the top of the salt chamber to the arm of the extractor body using another clip. Thread the two heated glycol hoses into the jacketed concentrators and tighten the hoses by hand only. The top hose goes to the top threaded stem and the bottom hose goes to the bottom threaded stem.

9.7. Pull the batch to be extracted from fridge #7 in GC Prep. Samples are sent in 1L or 250mL amber bottles. Scan the sample number into the ELN to build the batch. Ensure that all samples in a work order are together in the batch, unless the number of samples exceeds 20. Verify that samples are assembled on the bench in order by WO/sample number. Set each jar to the right of the extractor body that will be used for the sample's extraction. Sample IDs should be transcribed onto the jacketed concentrators with a solvent resistant permanent marker.

9.7.1. Sample volumes are used in their entirety unless specified by Project Manager, Technical Director, or Production Manager. If volume used is different from sample volume received for any reason, a comment will be added to that sample in the electronic notebook. Samples that may require a lesser volume may include TCLP leachate or filter samples, samples with previous history of low volume extraction, or samples that appear multi-phasic. Care must be taken to ensure samples are documented at the correct volume.

9.8. All containers will be marked with a solvent resistant permanent marker at the bottom of the meniscus. Then all volumes must be measured to the marked point and recorded in the ELN.

9.9. To prepare the method blank, LCS or LCS Duplicate, fill amber sample jars using de-ionized water (organic free). Add the room temperature spike standard to the QC (Quality Control) sample jars - LCS, LCSD, MS, and MSD -using the designated syringe. Cover with Teflon lined lid. Add the room temperature surrogate standard to all the sample containers, including all QC sample jars. Some sample jars will come filled to the rim. In those situations, decant a portion of the sample into the extractor body, making room to introduce the spiking mixes into the sample in the jar. *Note: refer to Attachment A "Spiking Requirement Table" for method specific spiking requirements.*

9.9.1. Spiking solutions must be introduced into the sample in the original sample container.

9.10. (#6) Turn chiller ON.

9.11. Verify the sample ID on the container matches what is written on the jacketed concentrator ("bell") before pouring the entire spiked sample into the designated, clean extractor body. Carefully pour the liquid so it does not go into the arm of the extractor body leading to the salt chamber. If the sample volume is significantly greater than 1 liter (e.g., larger than 1-liter sample container) measure 1 liter of sample with a solvent rinsed graduated cylinder. If the volume of the sample to be extracted is significantly less than 1 liter, dilute to 1 liter with de-ionized water. Verify sample ID with backlog to ensure procedure/sample requires lower volume. After pouring the sample into the extractor body rinse the sample container with DI water to remove any sediment, followed by 3 methylene chloride rinses, decanting each rinse into the extractor body. Rinse the cap to each jar with methylene chloride to ensure hydrocarbons present on the Teflon lid are extracted with the sample. **NOTE: if aliquot is taken, the sample container and cap will NOT be rinsed. This may bias the results.** Finally, rinse the neck of the extractor body to remove any sediment impeding the condenser from sealing properly: first with DI water and then with methylene chloride.

9.12. SIM PAH: After pouring the spiked sample into the extractor body add 100µl of nonane using a 100 µl syringe.

9.13. Cap the extractor body by lowering the coil condenser into the neck of the extractor body. Be sure that the chiller is approximately 2°C and that cold glycol is circulating through the condensers.

9.14. (#7) Start circulating heated glycol through the jacketed concentrators by rotating the ***TOP (A)*** green valve (underneath the bench) horizontally, followed by opening the ***BOTTOM (B)*** green valve. Reversing this order will cause hot glycol back pressure in the system & glassware. Before starting extraction, check the heater control box to ensure that the glycol temperature is at least 160°F.

9.15. (#8) Open the stopcock to allow solvent to flow through the salt chamber and down into the jacketed concentrator. Be sure not to flood the salt chamber, while maintaining a constant flow to jacketed concentrator. The methylene chloride level should not exceed the neck of the jacketed concentrator or go below the Teflon® chip. Allow the continuous extraction system to cycle for a minimum of 4 hours and a maximum of 6 hours. DRO/RRO and Sulfolane samples will be run for 16 hours.

9.16. Approximately 10 minutes after extraction begins, check all joints on extraction apparatus. This is achieved by checking the stopcock for leaks, verifying the condenser is set securely into the top joint of the body, and all ground glass connections are together. i.e., condenser to body, body to salt ball, salt ball to jacketed concentrator.

9.17. As stated in SW846 3520C – 3, section 7.1. "It is recommended that if high analyte concentrations are anticipated, samples should be collected in smaller sample bottles and the whole sample used."

Note: If there is more than ¼ inch of sediment in the bottom of the container, it is considered to be multi-phasic. If sample is multi-phasic, the Manager or Technical Director and the Project Manager must be contacted to determine which phase should be analyzed.

9.18. If more than one bench is to be set up, completely set up the 1st bench (set up through section 9.14 of this SOP) before starting set up of the 2nd bench. When setting up the 2nd bench, follow the same steps as above.

9.19. If more than one analyst is setting up the benches, follow Attachment C for efficient setup and extraction procedures.

9.19.1. Some clients send PAH samples preserved with acid. Extract those PAH samples as described: (Extract 2.5 hours at acidic pH and add 10mL 10N NaOH to bring the extraction to a basic pH. Continue extracting for 1.5 additional hours; extraction should not exceed 6 hours.).

9.20. **Extraction pH requirements:**

9.20.1. AK 102/103 – 8015, DRO/RRO: adjust to <2 with 4mL of 1:1 HCl

9.20.2. 8270 SVOC: Adjust pH with 1.5mL 1:1 H_2SO_4 to encourage acidic extraction. Extract 2.5 hours at acidic pH and add 10mL 10N NaOH to bring the extraction to a basic pH. Continue extracting for 1.5 additional hours; extraction should not exceed 6 hours.

9.20.2.1. TCLP leachates require ~ 15 mL of 10N NaOH due to the TCLP extraction fluid.

9.21. **For SW846 methods (i.e., 8270D-SIM, 8270D, 8082A, 8015C)**, each batch of twenty field samples or less requires a method blank (MB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is provided to perform an MS/MSD, then a laboratory control sample (LCSD) must be included.

9.21.1. Sulfolane requires a method blank (MB) and laboratory control sample (LCS) for every batch of twenty field samples or less. ***In addition:***

9.21.2. Sulfolane batches of 1-9 samples will include a field duplicate (DUP) sample. If insufficient sample is provided to perform a duplicate, then a laboratory control sample duplicate (LCSD) must be included.

9.21.3. Sulfolane batches of 10-20 samples will have a matrix spike (MS) and a matrix spike duplicate (MSD). If insufficient sample is provided to perform an MS/MSD, then a laboratory control sample duplicate (LCSD) must be included.

9.22. **For AK-series methods** (i.e., AK102/103), each batch of twenty samples or less requires a method blank (MB), a laboratory control sample (LCS) and a laboratory control sample duplicate (LCSD). These QC shall be pH adjusted with 4mL 1:1 HCl to extract in a similar pH as the preserved samples. The client may also request a matrix spike (MS) and matrix spike duplicate (MSD); in which case, they are prepared in addition to the LCS and LCSD. **Note:** ***ADEC requires AK102/103 batches to have both an LCS and an LCSD, even if the client provides sufficient sample for an MS/MSD.*** To prepare the MB, LCS or LCSD, use 1000mL de-ionized water (organic free).

9.23. Measure the initial sample volume (section 9.8.) by filling the sample bottle to the meniscus mark with tap water. Pour the water into a graduated cylinder and record this volume as the initial sample volume in the ELN.

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9.23.1. Be sure to keep samples in the order that they are set up on the bench when moving the containers to measure sample volume. Verify the container IDs with those in the ELN – samples should appear in the same order in both places. If something doesn't match up, do a double check to ensure correct information is being recorded (i.e. make sure a sample switch is not occurring).

9.24. After the extraction is complete, close the stopcock and allow the solvent to concentrate in jacketed concentrator to less than 10mL before turning off system.

9.25. To turn off extraction system:

1. Turn the heater power knob to **"off"**
2. Flip the switch on the end of cabinet to **"off"**
3. Turn the yellow flow control levers under the bench to **"drain"**
4. Turn the yellow lever in the pump cabinet to **"drain"**
5. Flip the switch on the end of cabinet to **"drain"** & loosen the top glycol hose attached to each concentrator to vent the system
6. Once the glycol has drained, turn the switch on the end of the cabinet to **"off"**
7. Rotate all green A & B valves to vertical
8. Turn the chiller **"off"**. Allow samples to equilibrate to room temperature before removing jacketed concentrators.

9.26. **Concentration:**

9.26.1. Check the water level in the TurboVap and adjust the temperature to 38ºC.

9.26.2. To collect the extraction solvent, unscrew both glycol hoses from the jacketed concentrator and remove the "bell" & "salt ball" together. After rinsing the salt chamber with methylene chloride, gently knock the salt ball against a gloved hand hard enough to disturb the Na2SO4 and allow the DCM to drain into the concentrator. Ensure the label on the concentrator tube has been accurately transcribed (i.e. the sample ID matches the ID on the "bell"). Remove the "salt ball" and pour the contents of the bell into a labeled concentrator tube. Rinse the bell three times with methylene chloride and pour the rinse directly into a labeled concentrator tube. After the extraction solvent has been transferred into the concentrator tube rinse the sides of the concentrator tube with methylene chloride. Repeat this process for all samples in batch.

9.26.3. When using large concentrator tubes, start the nitrogen flow at about 1.5 L/min or 5 psi and place the concentrator tubes containing the sample into the TurboVap. The TurboVap nitrogen flow pressure can be increased as the sample volume goes down but must not exceed 4.5 L/min or 22 psi. When using small concentrator tubes start the nitrogen flow at about 0.5 L/min and place the concentrator tubes containing the sample into the TurboVap. The TurboVap nitrogen flow pressure can be increased as the sample volume goes down but must not exceed 1.2 L/min. The surface of the solvent should be moving gently, not splashing.

9.26.4. Monitor the samples closely. When the sample level is down to ~2mL, thoroughly rinse the sides of the tube with methylene chloride. For samples that are analyzed in methylene chloride, continue concentrating until the sample level is in the tube nipple (~1mL). Rinse the slanted tube bottom with methylene chloride and concentrate to about 1mL.

9.26.5. Samples with a high concentration of oil or other heavy organic compounds will not concentrate to 1mL. When the sample volume is obviously not reducing, or the concentrate is viscous and black or starting to bubble, do not try to concentrate as low as it will go. If the volume is less than 5mL, bring it up to 5.0mL using the calibrated 5.0mL reference vial. Record the final dilution volume (i.e., 5.0mL) in the ELN and on the side of the vial. Extracts that are more than 5mL can be

measured with a class A graduated cylinder. DO NOT adjust the volume by (blowing down with nitrogen gun or adding solvent) once the final volume has been recorded. Transfer 1mL of the extract to an auto sampler vial. Record the total final volume on the side of the vial using a black marker and put a dot on the edge of the vial cap top. Contact the project manager and record the reason for the deviation in "comments" in the ELN.

9.26.6. Calibrate 1.0mL, 2.0mL, and 5.0mL reference vials daily by measuring methylene chloride using a gas tight syringe into each vial (i.e., a crimp top auto sampler vial for the 1.0mL and a 17x60mm 2-dram vial w/rubber lined cap for the 2.0mL and 5.0mL reference vial).

NOTE: *The following procedures must be used to prevent mixing sample ID during transfer from concentration tube to sample vial. This is the final critical step to insure the validity of the extract.*

9.26.7. To assure samples are not switched, confirm labeling on the concentrator tube and GC vial match. Rinse the slanted tube bottom with a few drops of methylene chloride and concentrate to ~0.8 mL. *No less than 0.5mL*. Transfer the sample from the tube to the labeled vial, (in a vial tray with the vials lined up to correspond with the tube position in the tube rack), using a 2mL glass pipette. Rinse the tube with a few drops of methylene chloride and add to the vial. The now empty concentrator tube is replaced in the tube rack in the same corresponding x-y position. If the sample volume is over 1.0mL by comparison with the reference vial use the nitrogen gun to reduce the volume to 1.0mL. If the sample volume is under 1.0mL, adjust to proper volume by adding methylene chloride using glass transfer pipettes. **Cap the vial and record the final volume**. (The final volume **must** be entered in ELN at the time the vial is capped.) When all samples have been transferred to vials, check the sample vial numbers with the empty tube numbers to assure that no errors have been made.

9.26.8. In the event that a sample vial needs to be relabeled (for example a poorly printed label or the sample placed in an incorrect vial) make sure that the vial's barcode is unreadable, and the new sample ID is clearly written. This will require immediate peer review and a note placed in the ELN.

NOTE: *Use of the nitrogen gun to reduce the sample volume in the vial creates an exothermic reaction that causes water condensation on & in the vial. Therefore it is to be used sparingly.*

9.26.9. Sulfolane extracts: Minimize use of the nitrogen gun as this analysis is extremely sensitive to moisture.

9.26.10. SVOC & PAH extracts: must never go below 0.5mL because lighter compounds will be lost.

9.26.11. Never under any circumstance allow a sample to concentrate to dryness.

9.26.12. **For 8082 PCB** samples that are analyzed in hexane, a solvent exchange to hexane must be done.

9.26.12.1. Monitor the sample in the TurboVap closely and when the sample level is down to ~1mL, rinse the sides of the tube with about 15mL of hexane and continue concentrating the sample. When the volume reaches 0.5mL, (1cm from the bottom of the tube) add 10mL of hexane. Repeat the 10mL hexane rinse again, reducing the sample volume to 0.5mL after each hexane rinse. Transfer the final concentrate (~0.8mL) to a sample vial and bring to the proper volume by adding hexane with a pasteur pipette. The following are the concentrate and rinse volumes for the hexane exchange process:

9.26.12.2.	Sample Volume	Hexane Rinse
	(First) 1.0mL	15mL
	0.5mL	10mL
	0.5mL	10mL
	~0.8mL	Done

9.26.12.3. For 8082 PCB analysis, the sample is brought to 1.0mL with hexane and is ready for analysis. Of note is the difference from Sonication 8082 PCB analysis (see SOP 761) which rinses with hexane a final time for a final volume of 5.0mL.

9.27. Deliver extracts to Fridge #33. For 24-hour Rush analysis, deliver extracts to the analyst.

9.27.1. For 8015, AK102/103, DRO/RRO, and PCB, deliver in clear glass auto sampler 1mL vials

9.27.2. For Sulfolane, 8270 SVOC, 8270 SIM, PEST SIM, use amber glass auto sampler 1 mL vials

9.28. Make sure that any & all sample oddities & deviations are documented and all blank entries in the ELN are filled, including the TurboVap ID and temperature, and the lot numbers of all solvents, standards and materials used. Post the data into LIMS. Double check entries (See attachment D). Print PDF version of the ELN and add to a new folder with the PRR pages from limsaide. Name folder in the following way: XXX [batch number] [analysis]. Add this folder to the XXX Batches for Review folder.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1. For each batch of up to twenty extracted: if there is not sufficient volume for an MS/MSD, an LCSD sample is extracted. **All batches for AK102/103 require both LCS and LCSD.** These samples are used to evaluate extraction efficiency and ensure that the extraction process is not contributing to sample results. Sample spikes and sample spike duplicates (MS/MSD set) may be requested by the client to evaluate site specific effects on the extraction/analysis process.

12.2. All samples, method blanks, LCS/LCSD and MS/MSD are fortified with the surrogate solutions.

12.3. Samples can be added to an active batch (samples still in the extraction process), with less than 20 samples, to the full 20 sample batch size. Examples would be RUSH samples or samples broken or spilled. The process end time must be the same for all samples in the batch, no less than 4 hours or more than 6 hours.

12.4. Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

Refer to the Peer Review SOP 101.

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative, a face shield, arm sleeves, and an apron must be worn.

15.0. POLLUTION PREVENTION:

15.1 SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

15.2 This custom made, automated system for continuous liquid-liquid extraction reduces solvent use.

16.0. METHOD PERFORMANCE:

N/A

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

N/A

20.0. REFERENCES:

SW846, Revision 3, December 1996
Method 3520C, Revision 3, December 1996
Method 3500C, Revision 3, February 2007

21.0. ATTACHMENTS:

Attachment A: Spiking Requirements Table
Attachment B: Flow Chart
Attachment C: Multi-Person Process for GC Prep
Attachment D: ELN Waters Checklist

ATTACHMENT A: Spiking Requirements Table

Method	Surrogate	Volume	Spike	Volume	QC ID
8270D Semi-vol.	**8270**(100/200µg/mL acid & BN Cpds)	1.0mL			MB
	8270(100/200µg/mL acid & BN Cpds)	1.0mL	8270 spike (100µg/mL)	1.0mL	LCS
	8270(100/200µg/mL acid & BN Cpds)	1.0mL	8270 spike (100µg/mL)	1.0mL	MS/MSD
	8270(100/200µg/mL acid & BN Cpds)	1.0mL			PS
8082 PCB	Tetra/Deca (0.4µg/mL)	1.0mL			MB
	Tetra/Deca (0.4µg/mL)	1.0mL	Aroclor 1016/1260 (1µg/mL)	1.0mL	LCS
	Tetra/Deca (0.4µg/mL)	1.0mL	Aroclor 1016/1260 (1µg/mL)	1.0mL	MS/MSD
	Tetra/Deca (0.4µg/mL)	1.0mL			PS
8270D-PAH SIMS (also for Low Volume)	PAH (0.5µg/mL)	1.0mL			MB
	PAH (0.5µg/mL)	1.0mL	PAH Spike (0.5µg/mL)	1.0mL	LCS
	PAH (0.5µg/mL)	1.0mL	PAH Spike (0.5µg/mL)	1.0mL	MS/MSD
	PAH (0.5µg/mL)	1.0mL			PS
	All samples and QC spiked with 100µl nonane				
Sulfolane	Sulfolane d_8 (80µg/mL) w/ 8270(100µg/mL acid & BN Cpds)	1.0mL			MB
	Sulfolane d_8 (80µg/mL) w/ 8270(100µg/mL acid & BN Cpds)	1.0mL	Sulfolane Spike (15µg/mL)	1.0mL	LCS
	Sulfolane d_8 (80µg/mL) w/ 8270(100µg/mL acid & BN Cpds)	1.0mL	Sulfolane Spike (15µg/mL)	1.0mL	MS/MSD
	Sulfolane d_8 (80µg/mL) w/ 8270(100µg/mL acid & BN Cpds)	1.0mL			PS
8270D-Pest SIMS	Pest (0.5µg/mL)	1.0mL			MB
	Pest (0.5µg/mL)	1.0mL	Pest spike (0.25µg/mL)	1.0mL	LCS/LCSD
	Pest (0.5µg/mL)	1.0mL	Pest spike (0.25µg/mL)	1.0mL	MS/MSD
	Pest(0.5µg/mL)	1.0mL			PS
AK102/103 DRO/RRO (also for Low Volume)	**5-Alpha & DTC (100µg/mL)**	1.0mL			MB
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL			PS
Silica Gel AK 102/103 DRO/RRO	**5-Alpha & DTC (200µg/mL)**	1.0mL			MB
	5-Alpha & DTC (200µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	2.0mL	LCS/LCSD
	5-Alpha & DTC (200µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	2.0mL	MS/MSD
	5-Alpha & DTC (200µg/mL)	1.0mL			PS

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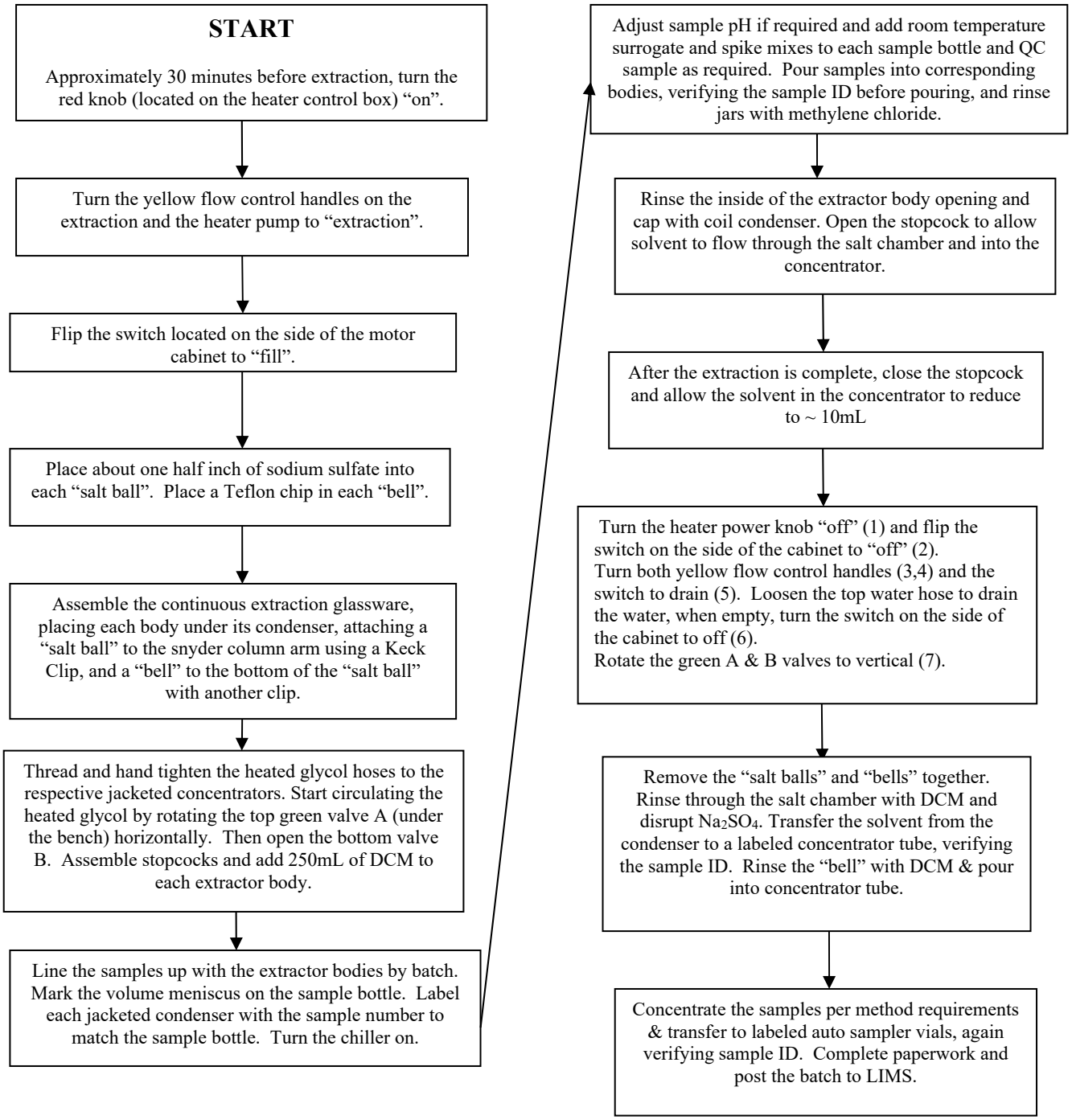
Method	Surrogate	Volume	Spike	Volume	QC ID
8015C DRO/RRO	**5-Alpha & DTC (100µg/mL)**	1.0mL			MB
	5-Alpha & DTC (100µg/mL)	1.0mL	DRO by 8015 (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	DRO by 8015 (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL			PS

Attachment B: Flow Chart



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Attachment C: Multi-Person Process for GC Prep

Before Liquid-Liquid extraction has started:

- Collect samples for the batch
- Build Batch in LIMS
- Retrieve Spikes and Surrogates
- Mark sample volumes
- Fill bodies
- Label concentrators
- Fill out ELN comments if necessary
- Spike Witness

After Liquid-Liquid extraction has started:

- Fill out ELN start date and Spike IDs
- Measure sample volumes
- Label TurboVap Tubes
- Print Vials
- Prepare appropriate reference vials
- Routinely check DCM level in concentrators
- Adjust stopcocks as necessary

Once Liquid-Liquid extraction time has elapsed:

- Turn stopcocks on each side of bench off
- Watch each side of bench for concentration <10mL
- Perform Shutdown Procedure
    - Refer to Attachment B
- Concentrate batch
- Complete ELN documentation
- Review ELN for Posting

Special Attention Needed

- Watch TurboVaps
    - Raise Pressure
    - Rinse tubes at ~2mL
    - Remove tubes at ~0.8mL
    - Record TurboVap Temperatures in ELN
- Communicate as tasks are completed
- Document ELN at each step
    - Do not fill out final volume in advance of concentration
        - Enter final volume as concentration is completed
    - Do not fill out Analyst initials until step has been completed
    - Add comments to samples and batches when analysis differs from normal

Attachment D: ELN Waters Checklist

	DRO/RRO	PAH	PCB	Silica Gels	8270	Pesticide	8015 (HI)	Sulfolane
Initial Volume	X	X	X	X	X	X	X	X
Final Volume	X	X	X	X	X	X	X	X
Analyst Initials	X	X	X	X	X	X	X	X
Start Date and Time	X	X	X	X	X	X	X	X
End Date and Time	X	X	X	X	X	X	X	X
Analysis	X	X	X	X	X	X	X	X
TurboVap Temperature	X	X	X	X	X	X	X	X
TurboVap ID	X	X	X	X	X	X	X	X
Surrogate Lot ID	X	X	X	X	X	X	X	X
Surrogate Amount Added	X	X	X	X	X	X	X	X
Surrogate Concentration	X	X	X	X	X	X	X	X
LCS(D)/MS(D) Lot ID	X	X	X	X	X	X	X	X
LCS(D)/MS(D) Amount Added	X	X	X	X	X	X	X	X
LCS(D)/MS(D) Concentration	X	X	X	X	X	X	X	X
Silica Gel Lot #				X				
Hexane Lot #			X					
Na2SO4 Lot #	X	X	X	X	X	X	X	X
Ch2Cl2 Lot #	X	X	X	X	X	X	X	X
HCl Lot #	X							
H2SO4 Lot #					X			
NaOH Lot #					X			
Additional Spike Amount							X	
Measure Sample Volume Initials	X	X	X	X	X	X	X	X
Spike/Surrogate	X	X	X	X	X	X	X	X
Witness Spike/Surrogate	X	X	X	X	X	X	X	X
Concentrate	X	X	X	X	X	X	X	X
Posted By	X	X	X	X	X	X	X	X
Nonane Lot #		X						

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure	
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.		
S.O.P. Title: Sonication Extraction of Semi-Volatile Compounds in Soil		**Revision Date**: June 2023
Method No: SW846 3550C		**SOP No**: 761r24
Page: 1 of 16		**Supersedes:** 761r23

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	6/7/23	Jamaica Beatty	6/7/23

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

This electronically signed PDF will be considered the controlled copy for staff. Any printouts or photocopies are invalid.

Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Addendum #1 added 7/11/23 TPR
Addendum #2 added 10/12/23 TPR

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Sonication Extraction of Semi-Volatile Compounds in Soil	**Revision Date**: July 2023
Method No: SW846 3550C	**SOP No**: 761r24 Addendum #1
Page: 1 of 1	**Supersedes**: 761r23

Signatures below reflect approval for the following changes to the current SOP. These changes will be incorporated into the SOP during the next review. This addendum will be incorporated into the electronic SOP (i.e., PDF file).

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	7/11/23	Samara Rentty	7/11/23

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

The above referenced SOP should be modified as follows:

Change section:

12.3.1. PAH/DRO Method Blank and LCS/ LCSD are spiked with 2mL of DI before adding surrogates or spike compounds.

To:

12.3.1 PAH/DRO Method Blank and LCS containing Teflon are spiked with 2mL of DI before adding surrogates or spike compounds. PAH/DRO Method Blank and LCS/LCSD containing baked sand are not spiked with DI.

Controlled copy distributed to Brie Hicknell with VHB on 2/5/2024.

SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Sonication Extraction of Semi-Volatile Compounds in Soil	**Revision Date**: October 2023
Method No: SVW846 3550C	**SOP No**: 761r24 Addendum #2
Page: 1 of 1	**Supersedes**: 761r23

Signatures below reflect approval for the following changes to the current SOP. These changes will be incorporated into the SOP during the next review. This addendum will be incorporated into the electronic SOP (i.e., PDF file).

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	10/12/23	Jamaica Penty	10/12/23

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

The above referenced SOP should be modified as follows:

Change:

7.1 Sonicator: ~~Misonix Corporation~~, Model Q700 (or equivalent); 300 watts minimum, ¾ inch horn

To:

7.1 Sonicator: QSONICA sonicators, Model Q700 (or equivalent); 300 watts minimum, ¾ inch horn

Change:

9.4.1. Sonicator is set to achieve:

- Extract the sample ultrasonically for 3 minutes with manufacturer's recommended power setting. To do so repeat the 1 ½ minute sonication 3 times. ~~Observe the amplitude is 70, watts shown between 51-70 W, and energy at end of each sequence repetition around 4,964 J.~~
- Mode: Pulse
- Duty Cycle: 50%

To:

9.4.1. Sonicator is set to achieve:

- Extract the sample ultrasonically for 3 minutes with manufacturer's recommended power setting. To do so repeat the 1 ½ minute-sonication 3 times.
- Mode: Pulse
- Duty Cycle: 50%

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Summary of Changes from Previous Revision:

- Incorporated addendum from previous revision
- Updated Attachment A to combine the DRO and PAH soil extractions
- Updated section 9.5 to reflect current practice.
- Updated section 9.6 to standardize methylene chloride volume across all extractions.
- Updated section 9.9 to standardize methylene chloride volume across all extractions.
- Updated section 9.11.3.1 to include PAH.

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1.0. OBJECTIVE:

This method can be used to isolate organic compounds from soil, solid, and wipe samples. This method is applicable to the isolation and concentration of semi-volatile organics in preparation for a variety of chromatographic procedures.

2.0. SCOPE AND APPLICATION:

This SOP is suitable for use with methods SW8 8270D, PAH by 8270D-SIMS, 8270 Pest SIMS, 1625B / 8270D Sulfolane Isotopic Dilution, 8015C, AK 102, AK 103, and 8082A. A wipe or measured mass of sample approximately 22.5 to 30 grams is serially extracted by sonication with methylene chloride. The extract is dried, concentrated, and, if necessary, exchanged into a solvent compatible with the cleanup or the analysis being employed. The extracts are then analyzed with gas chromatography or gas chromatography/mass spectroscopy.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. Total extraction solvent volume for DRO/RRO and wipes is 200mL. The reference method states that 300mL should be used for 30 grams of sample. Industry standards use smaller volumes to minimize chlorinated solvent waste.

3.2. The initial sample weight is approximately 22.5 grams for 8270D, 8270D-SIM, and 8082A. The reference method states that a 30-gram sample will be used. Since the concentration device has a maximum volume of 260mL, the lab is keeping the same solvent-to-sample ratio as stated in the reference method.

3.3. Horn tip size used is one size- ¾". Method 3550C states that a smaller tip needs to be used for samples with high concentration. Samples are extracted at low levels to meet Alaska state requirements for detection limits.

3.4. Sodium sulfate is added to achieve a grainy consistency. Method 3550C states that wet samples and nonporous samples should at a minimum have 60g added to the samples. Industry standards use smaller volumes to minimize chlorinated solvent waste.

3.5. Methylene chloride is used for all analysis extractions. Method 3550C has three options for different solvents and solvent mixtures for 8082A. Method based performance (e.g. PT studies and LCS recoveries) supports efficiency of practice.

3.6. Hexane solvent exchanges use one 15mL, two 10 mL, and one 4.5 mL exchanges. Method 3550C states one exchange at 50 ml. Industry standards use smaller volumes to minimize solvent waste. Method based performance (i.e. PT studies and LCS recoveries) supports efficiency of practice.

3.7. XcelVap and TurboVap baths are maintained at 38ºC for all analysis. Method 3550C states that baths used in the concentration step are to be set at 30ºC. Method based performance (e.g. PT studies and LCS recoveries) supports efficiency of practice.

3.8. QC compounds, (MS, LCS, surrogates), are added to the samples at the time of extraction after they have been processed in the weight room & dried with sodium sulfate. Method 3550C states that samples are to be spiked before the use of sodium sulfate. Method based performance (e.g. PT studies and LCS recoveries) supports efficiency of practice.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a "read only" file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have "read" access to these SOPs. Only QAQC has access to "write" on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Samples that contain high hydrocarbon content may interfere with surrogate/spike recovery.

5.2. Phthalate esters from laboratory equipment (e.g., gloves, soaps, plastic, and rubber) may contaminate samples. Phenols may react to form tannates.

6.0. SAMPLE HANDLING:

6.1 Sample Matrix – Soil, solid, and wipe samples.

6.2 Sample Size – 4 or 8oz amber glass jars with Teflon-lined lids. Wipes – 4 oz amber glass jars with Hexane.

6.3 Sample Preservation – Soil samples are stored at 0-6°C until extraction. Wipe samples should be stored at ambient temperature in the flam room until extraction. Hexane extracts are refrigerated at 0-6ºC. Methylene chloride extracts are stored at $\leq$ -10ºC.

6.4 Holding Times – Soil samples (with the exception of PCB analysis) must be extracted within 14 days of sample collection. Soil and wipe samples for PCB analysis have a hold time of 1 year. Samples must be analyzed within 40 days after extraction.

6.5 Criteria for Acceptance/Rejection of Samples – If samples have broken hold time or were not stored at 0-6ºC, notify the Project Manager (PM) before proceeding with extraction.

6.6. Multi-Incremental Sample Handling: Select clients/projects may require special handling as outlined by The Interstate Technology & Regulatory Council (ITRC), February 2012. **Sample aliquots designated for multi-incremental (MI) handling will be identified by Sample Receiving (based on Project Notice and client COC), clearly labeled for "MI" handling.** As outlined in ITRC, the client will submit a field-prepared aliquot for each organic extraction *(Duplicate containers will be submitted for each analysis.)* which has already been sieved and homogenized. A separate, full container for Total Solids determination should also be submitted for each sample. MI samples for both volatiles and semi-volatiles will have been received as two separate samples, each with their own jar for total solids determination. ***If no other containers are submitted, the aliquot required for Total Solids determination may be removed from the "MI" labeled container prior to extraction, provided the mass for extraction still meets the method requirement.*** **(Note: The "MI" labeled container must be used in its entirety and may not, under any circumstances, be used for any other analysis.)** Each client requesting laboratory support for MI projects is reminded that **the maximum sample size which can be extracted is 30 grams**. If sample aliquots exceed this limit, contact the Project Manager immediately.

7.0. APPARATUS:

7.1. Sonicator: Misonix Corporation, Model Q700 (or equivalent); 300 watts minimum, ¾ inch horn

7.2. Virtuoso autosampler vial bar code printer.

7.3. Pyrex glass concentrator tubes with a 1mL tip not graduated.

7.4. Concentrator Apparatus: TurboVap, XcelVap or equivalent.

7.5. Short-stem or stemless glass funnels

7.6. 500mL jars with Teflon®-lined lids

7.7. 1mL syringes: Hamilton gastight, in house certified calibrated, as stated in SOP 104

7.8. Virtuoso autosampler vials: amber and clear, with Teflon®-lined crimp tops

7.9. Disposable glass pipettes: 1, 5, and 10mL

7.10. Disposable Pasteur pipettes, 7 and nine inch.

7.11. FisherBrand screw cap vial, 2 dram, w/rubber lined cap. (5mL vial)

7.12. Glass Wool: (heated at 400°C for ≥3 hours)

7.13. Pipette bulbs and pumps

7.14. Vial cap crimper

7.15. Teflon wash bottles, 500mL, labeled: Methylene Chloride FILLED DAILY

8.0. REAGENTS:

8.1. Dichloromethane (CH2Cl2 / DCM): Fisher Optima (or equivalent) chromatographic grade.

8.2. Hexane: B&J -GC2 (or equivalent) high purity Pesticide grade

8.3. Sodium sulfate: Granular, Anhydrous (heated at 400°C for ≥3 hours)

8.4. Sand: Industrial quartz, 40 mesh (heated at 400°C for ≥3 hours)

8.5. Teflon boiling chips

8.6. n-Nonane: Acros 99% or equivalent. Used as PAH keeper.

9.0. EXTRACTIONS:

9.1. The procedure for weighing and preparing the sample for extraction is found in SOP 143. All weighed sub-samples must be extracted within regulatory hold times, 14 days for all analysis with the exception of PCB. The hold time for PCBs is 1 year.

9.2. Obtain the weighed sub-samples from the Sample Control, allow samples to reach room temperature and **verify that they are free flowing**. Some matrices, such as peat, muskeg, or non-soil solids, may not actively mix. If sample is not free flowing make sure adequate sodium sulfate was added to dry the sample and mix to free flowing consistency. If there is a problem, enter the deviation in "comments" in the ELN and contact the Project Manager. Weighed soil samples will be organized in batches which include QC (Refer to SOP 143). Use the bar code reader to enter weights into the ELN. Wipes are extracted as received (that is, in their entirety), so their weights are not determined. For wipe QC samples, use large sterile gauze pads.

9.3. Label concentrator tubes and sample jars with sample ID for all samples and QC in the extraction batch. Fill the stemless funnels with glass wool and baked sodium sulfate. Enter all reagent lot numbers in ELN at this time. All glassware is kiln baked or solvent rinsed according to the SGS "Glassware Cleaning and Preparation" SOP #117.

9.4. The Misonix Model S Q700 sonicators do not require tuning. They have a diagnostic program that is used at the beginning of each day.

9.4.1. Sonicator is set to achieve:

- Extract the sample ultrasonically for 3 minutes with manufacturer's recommended power setting. To do so repeat the 1 ½ minute sonication 3 times. Observe the amplitude is 70, watts shown between 51-70 W, and energy at end of each sequence repetition around 4,964 J.
- Mode: Pulse
- Duty Cycle: 50%

9.5. Add appropriate room temperature surrogate standards to the samples and all quality control (QC) samples with a gas tight syringe. A laboratory standard spike solution is added to the LCS/LCSD and MS/MSD.

9.5.1. For PAH SIM/AK102/103 sample extraction, add 100µl of nonane (as a keeper) using a 100µl gas tight syringe.

9.5.2. For PAH SIM/AK102/103 and 8270 SVOC sample extraction, add 2.0mL of DI water to **QC samples that only contain Teflon chips and sodium sulfate** before adding their surrogate and spike.

9.5.3. The PAH SIM/AK102/103 surrogate mix is not in solution at refrigerated temperatures. It must be warmed to room temperature & mixed back into solution.

9.6. Add the first aliquot of methylene chloride immediately after spiking. For all analyses, add 75mL of methylene chloride to each sample. Record identification of the standards used in ELN (See attachment C). **NOTE: Some samples may be slightly aqueous (ex. muddy samples) and require additional sodium sulfate to produce a dry, free flowing aliquot. This can result in the volume of sample/salt mixture exceeding the surface of the added solvent. In this case, add additional solvent until the solid material is at least 1cm beneath the solvent level and record the extra volume in the ELN.**

9.7. Place the horn in the sample container (approximately 1cm beneath the surface of the solvent, above the solid material) and start the sonicator program. **Active mixing of the sample to the bottom of the jar must occur when the ultrasonic pulse is activated.**

9.8. Confirm labeling on the concentrator tubes and the sample containers to ensure samples are not switched. Once the sonicator program has finished, decant the solvent through the sodium sulfate funnel into the concentrator tube.

9.9. After pouring extract into tube, add 75mL of methylene chloride to all samples. This step is repeated once to give a total of three sonication / pour off cycles. Once the solvent has completely drained after each extraction, rinse the funnels with a small amount of methylene chloride using the Teflon squeeze bottles. (The horn is rinsed between samples with methylene chloride and wiped with a KimWipe. A green scrubbie pad can be used if the horn cannot be cleaned easily.)

NOTE: *Any deviations of procedure or sample reaction during extraction and concentration of samples MUST be documented in the ELN in the "comments" section provided.*

9.10. **Wipes**:

9.10.1. Samples are received in a jar with 25mL hexane. Wipe and hexane are transferred to a 500mL jar for extraction.

9.10.2. Dispense 50mL methylene chloride to the 4-oz amber bottle that the wipe was preserved in and pour into the extraction jar. The final volume of the first extraction should be 75 mL of methylene chloride and hexane combined. Perform steps 9.6 through 9.9 for all samples, and QC.

9.10.3. Required QC: MB & LCS / LCSD (This matrix will not be available in sufficient quantities for MS/MSD.) All QC samples are clean, sterile gauze pads, cut in half with 75mL of methylene chloride. Introduce surrogate & spiking solutions into the extraction jar.

9.10.4. Proceed with the extraction procedure for the normal 3 sonication periods, and complete the extraction / concentration per normal process.

9.11. **CONCENTRATION***:*

9.11.1. Check the water level in the XcelVap and adjust the temperature to 38ºC.

9.11.2. Adjust the nitrogen flow to about 1.5 L/min, or 5 PSI, and place the concentrator tubes containing the sample into the XcelVap. The surface of the solvent should be moving gently, not splashing. The PSI can be programmed to ramp up as the solvent volume decreases, up to a maximum of 4.5 L/min, or 22 PSI, when the sample volume is below the half way point.

9.11.3. Monitor the samples closely; when the sample level is down to ~10mL, rinse the sides of the tube with methylene chloride. For analysis that are analyzed in methylene chloride, continue concentrating until the sample level is in the tube nipple. Rinse the slanted tube bottom with methylene chloride and concentrate to about 1mL, taking care to not concentrate below 0.5mL.

9.11.3.1. Samples for PAH SIM/AK102/103 analysis will be concentrated to 5.0mL.

9.11.4. Samples with a high concentration of oil or other heavy organic compounds, will not concentrate to 1mL. When the sample volume is obviously not reducing, **do not try to concentrate as low as it will go. If the volume is less than 5.0mL, bring the volume up to 5.0mL using a calibrated 5.0mL reference vial.** The volume of extracts that are more than 5mL can be determined **with a class A graduated cylinder. DO NOT** adjust the volume by (blowing down with a nitrogen gun or adding solvent). Record the final volume and reason for the increased extract volume into the ELN. Transfer a 1.0mL portion of the sample to a labeled vial and cap~~,~~ record the total final volume on the side of the vial using a black marker and put a dot on the edge of the vial cap top. Contact the project manager.

9.11.4.1. **PCB extracts that will not concentrate to less than 1mL are a common occurrence. Proceed as follows for these extracts to hexane exchange:**

9.11.4.1.1. Concentrate the DCM extract to 5mL, 10mL, 15mL or 20mL only. The sample matrix determines the volume. If the sample will not concentrate 20mL notify production manager.(NOTE the regulatory action limit is met at 20mL)

9.11.4.1.2. Only 5mL of sample concentrate is used for solvent exchange. For the samples that concentrate to 10, 15, or 20mL, remove 5.0mL for solvent exchange. Add an extra hexane exchange to maximize DCM removal, (see section 9.11.3.) Bring the sample back to 5.0mL volume for acid cleanup.

9.11.4.1.3. Follow the acid cleanup procedure (SOP 765).

9.11.4.1.4. Record the final volume in ELN. In comments record the portion used for solvent exchange and volume submitted for analysis. For example: if a sample concentrated to 20.0mL:

Enter the final volume in ELN – 20.0mL
Comment: 5 of 20 mL solvent exchanged
& acid cleanup. 1 of 5.0mL submitted for analysis.

9.11.5. Calibrate 1.0, 2.0, and 5.0mL reference vials daily by measuring methylene chloride using a gas tight syringe into each vial (i.e., a crimp cap autosampler vial for the 1.0mL and a 5 dram septum lined screw cap vial for the 2.0 and 5.0mL reference vial).

NOTE: *The following procedures must be used to prevent mixing sample ID during transfer from concentration tube to sample vial. This is the final critical step to insure the validity of the extract.*

9.11.6. To assure samples are not switched, confirm labeling on the concentrator tube and GC vial match. Rinse the slanted tube bottom with a few drops of methylene chloride and concentrate to ~3/4 of a mL. *No less than 0.5mL*. Transfer the sample from the tube to the labeled vial, (in a vial tray with the vials lined up to correspond with the tube position in the tube rack), using a 2mL glass pipette. Rinse the tube with a few drops of methylene chloride and add to the vial. The now empty concentrator tube is replaced in the tube rack in the same corresponding x-y position. If the sample volume is over 1.0mL by comparison with the reference vial use the nitrogen gun to reduce the volume to 1.0mL. If the sample volume is under 1.0mL, adjust to proper volume by adding methylene chloride using glass transfer pipettes. **Cap the vial and record the final volume**. (The final volume **must** be entered in ELN at the time the vial is capped.) When all samples have been transferred to vials, check the sample vial numbers with the empty tube numbers to assure that no errors have been made.

NOTE: *Use of the nitrogen gun to reduce the sample volume in the vial creates an exothermic reaction that causes water condensation on & in the vial. Therefore it is to be used sparingly.*

9.11.6.1. Sufolane extracts: Minimize use of the nitrogen gun as this analysis is extremely sensitive to moisture.

9.11.6.2. SVOC & PAH extracts: must never go below 0.5mL because lighter compounds will be lost.

9.11.6.3. Never in any circumstance allow a sample to concentrate to dryness.

9.11.7. In the event that a sample vial needs to be relabeled, for example a poorly printed label or the sample placed in an incorrect vial. Make sure that the vial's barcode is unreadable, and the new sample ID is clearly written. This will require immediate peer review and a note placed in the ELN.

9.12. **CLEANUP**

9.12.1. Silica Gel samples are concentrated to about 2mL and split for silica gel clean up. Transfer ½ of the well mixed homogenous concentrate into an autosampler vial for analysis. The remaining ½ of the concentrate is used for silica Gel cleanup. ***The two sample aliquots must be equal in concentration and volume.*** Refer to SOP 765 for silica gel procedure.

9.12.2. For PCB samples that are analyzed in hexane, a solvent exchange to hexane must be done.

9.12.3. Solvent Exchange: Monitor the sample in the XcelVap closely, when the sample level is down to ~1mL, rinse the sides of the tube with about 15mL of hexane. Concentrate to 0.5mL, (1cm from the bottom of the tube), and add 10mL of hexane. Again, concentrate to 0.5mL and rinse with 10mL hexane. Concentrate to 0.5mL, remove from XcelVap, and rinse with 4 mL hexane. Final volume is adjusted to exactly 5.0mL and transferred to a scintillation vial for acid clean-up (refer to SOP#765). If no acid clean up is required then a 1.0mL aliquot is measured and transferred to the auto sampler vial.

9.13. Deliver extracts to **Fridge #33** storage for 8015, AK 102/103 and PCB in clear glass auto sampler 1mL vials. For Rush analysis deliver to analyst.

9.14. Deliver extracts to **Fridge #33** sample storage for Sulfolane, 8270 SVOC, PAH SIMS, and PST SIMS in amber glass auto sampler 1mL vials. For Rush analysis deliver to analyst.

9.15. Fill in all blank entries in the ELN including the XcelVap temperature, and the lot numbers of all solvents, standards and materials used. Post the data into LIMS. Double check your entries, submit a copy of the ELN to the analyst with the sample extracts.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1. For each batch of up to twenty samples (excluding matrix spikes/spike duplicates and other extraction QC) a method blank, LCS and MS/MSD is extracted. If the client does not specify a sample for spiking, one sample in the batch is selected. If there is not sufficient volume for an MS/MSD, a LCS duplicate sample is extracted. **All batches for AK102/103 require both LCS and LCSD.** These samples are used to evaluate extraction efficiency and ensure the extraction process is not contributing to sample results. Sample spikes and sample spike duplicates (MS/MSD set) may be requested by the client to evaluate site specific effects on the extraction/analysis process.

12.2. Sulfolane required batch QC:

12.2.1. Batches of 9 or less samples, extract a MB, LCS, and a duplicate sample.

12.2.2. Batches of 10 -20 samples, extract a MB, LCS, and a MS/MSD.

12.3. PAH/DRO requirements:

12.3.1. PAH/DRO Method Blank and LCS/ LCSD are spiked with 2mL of DI before adding surrogates or spike compounds.

12.3.2. All PAH/DRO samples and QC are spiked with 100µl of Nonane after surrogate & spike fortification.

12.3.3. PAH/DRO soil samples are concentrated to a final volume of 5.0mL. 1.0mL of this concentrate is sent for analysis.

12.4. Surrogate solutions are added to all samples, method blanks, LCS/LCSD and MS/MSD for all analysis.

12.5. Samples can be added to an active batch (samples are still being concentrated), with less than 20 samples, to the full 20 sample batch size. Examples would be, RUSH samples or samples broken or spilled.

Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

Refer to the Peer Review SOP 101.

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling any solutions with concentrated acid or base preservative, a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

N/A

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANITATION (LOQ):

N/A

20.0. MAINTENANCE:

Non-routine maintenance will be recorded in maintenance logbooks. Documentation will include a synopsis of the deviation requiring maintenance, and the corrective action performed. In addition, the batch an instrument was returned to service in will be recorded, if applicable. Regular and accurate logbook entries will provide troubleshooting during future non-routine maintenance.

21.0. REFERENCES:

SW846, Current Edition
Method 3500C, Revision 3, February 2007
Method 3660B, Revision 3, December 1996
Method 3665A, Revision 1, December 1996
The Interstate Technology & Regulatory Council Incremental Sampling Methodology, February 2012

22.0. ATTACHMENTS:

Attachment A: Spiking Requirement Table
Attachment B: Process for GC Prep- Soils
Attachment C: ELN Soil Checklist
Attachment D: Concentrate Solvent & Final Volume

ATTACHMENT A: SPIKING REQUIREMENT TABLE

Method	Surrogate	Volume	Spike	Volume	QC ID
Sulfolane	Sulfolane d_8 & B/N Surrogate (25& 5µg/mL)	1.0mL			MB
	Sulfolane d_8 & B/N Surrogate (25& 5µg/mL)	1.0mL	Sulfolane Spike (1.5µg/mL)	1.0mL	LCS/LCSD
	Sulfolane d_8 & B/N Surrogate (25& 5µg/mL)	1.0mL	Sulfolane Spike (1.5µg/mL)	1.0mL	MS/MSD
	Sulfolane d_8 & B/N Surrogate (25& 5µg/mL)	1.0mL			PS
8270D Semi-vol.	8270 (100µg/mL BN & 200 µg/mL acid surrogates)	1.0mL			MB
	8270 (100µg/mL BN & 200 µg/mL acid surrogates)	1.0mL	8270 spike (100µg/mL)	1.0mL	LCS
	8270 (100µg/mL BN & 200 µg/mL acid surrogates)	1.0mL	8270 spike (100µg/mL)	1.0mL	MS/MSD
	8270 (100µg/mL BN & 200 µg/mL acid surrogates)	1.0mL			PS
8270D-PAH SIMS/ AK102/103	PAH/DRO (2.50 + 500µg/mL)	1.0mL			MB
	PAH/DRO (2.50 + 500µg/mL)	1.0mL	PAH Spike (2.50µg/mL)	1.0mL	LCS PAH
	PAH/DRO (2.50 + 500µg/mL)	1.0mL	LSB / LSR (25000µg/mL)	1.0mL	LCS DRO
	ADD 2mL DI to MB/LCS/LCSD				
	PAH/DRO (2.50 + 500µg/mL)	1.0mL	PAH Spike (2.50µg/mL)	1.0mL	MS/MSD PAH
	PAH/DRO (2.50 + 500µg/mL)	1.0mL	LSB / LSR (25000µg/mL)	1.0mL	MS/MSD DRO
	PAH/DRO (2.50 + 500µg/mL)	1.0mL			PS
		100µl	n Nonane (Keeper)	QC &	All Samples
8270D-Pest SIMS	PST (0.5µg/mL)	1.0mL			MB
	PST (0.5µg/mL)	1.0mL	Pest spike (0.250µg/mL)	1.0mL	LCS
	PST (0.5µg/mL)	1.0mL	Pest spike (0.250µg/mL)	1.0mL	MS/MSD
	PST (0.5µg/mL)	1.0mL			PS
8082 PCB	Tetra/Deca (10µg/mL)	0.5mL			MB
	Tetra/Deca (10µg/mL)	0.5mL	Aroclor 1016/1260 spike 10µg/mL)	0.5mL	LCS/LCSD
	Tetra/Deca (10µg/mL)	0.5mL	Aroclor 1016/1260 spike 10µg/mL)	0.5mL	MS/MSD
	Tetra/Deca (10µg/mL)	0.5mL			PS
8015C DRO/RRO	5-Alpha & DTC (100µg/mL)	1.0mL			MB
	5-Alpha & DTC (100µg/mL)	1.0mL	DRO by 8015 (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	DRO by 8015 (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL			PS
Silica Gel AK102/103 DRO/RRO	5-Alpha & DTC (200µg/mL)	1.0mL			MB
	5-Alpha & DTC (200µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	2.0mL	LCS/LCSD
	5-Alpha & DTC (200µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	2.0mL	MS/MSD
	5-Alpha & DTC (200µg/mL)	1.0ml			PS

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ATTACHMENT B: Process for GC Prep-Soils

Before a Sonication batch has been started:

- Retrieve Spike and Surrogates
- Set out batch for spiking
- Create batch in LIMS
- Build batch in ELN
- Shake samples and loosen lids
- Spike Witnessing

After a Sonication batch has been started:

- Label TurboVap/Xcel Vap Tubes
- Print Vials
- Prepare next Sonication batch

After a Sonication batch has been blown down

- Concentrate batch
- Complete ELN documentation
- Review ELN for Posting

Special Attention Needed

- Watch TurboVaps/Xcel Vaps
    - Raise Pressure
    - Rinse tubes at ~2mL
    - Remove tubes at ~0.8mL
    - Record TurboVap/Xcel Vap Temperatures in ELN

- **Communicate as tasks are completed**
- **Document ELN at each step**
    - Do not fill out final volume in advance of concentration
        - Enter final volume as concentration is completed
    - Do not fill out Analyst initials until step has been completed
    - **Do add comments to samples and batches when analysis differs from normal**

ATTACHMENT C: ELN Soil Checklist

	DRO/RRO	PAH	PCB	Silica Gels	8270	Pesticide	8015 (HI)	Sulfolane	PCB Oils	DRO & 8270 Oils
Stand in for BMS(D)		X	X		X	X		X		
Initial Weight	X	X	X	X	X	X	X	X	X	X
Final Volume	X	X	X	X	X	X	X	X	X	X
Analyst Initials	X	X	X	X	X	X	X	X	X	X
Start Date and Time	X	X	X	X	X	X	X	X	X	X
End Date and Time	X	X	X	X	X	X	X	X	X	X
Analysis	X	X	X	X	X	X	X	X	X	X
Turbo Vap Temperature	X	X	X	X	X	X	X	X		
Turbo Vap ID	X	X	X	X	X	X	X	X		
Surrogate Lot ID	X	X	X	X	X	X	X	X	X	
Surrogate Amount Added	X	X	X	X	X	X	X	X	X	
Surrogate Concentration	X	X	X	X	X	X	X	X	X	
LCS(D)/MS(D) Lot ID	X	X	X	X	X	X	X	X	X	
LCS(D)/MS(D) Amount Added	X	X	X	X	X	X	X	X	X	
LCS(D)/MS(D) Concentration	X	X	X	X	X	X	X	X	X	
Isooctane Lot #									X	
Silica Gel Lot #				X						
Hexane Lot #			X							
Na2SO4 Lot #	X	X	X	X	X	X	X	X		
Glass Wool Lot #	X	X	X	X	X	X	X	X		
Ch2Cl2 Lot #	X	X	X	X	X	X	X	X		X
Sand Lot #	X		X	X		X	X	X		
Teflon Lot #		X			X					
H2SO4 Lot #			X*						X	
Additional Spike Amount							X			
Spike/Surrogate	X	X	X	X	X	X	X	X	X	
Witness Spike/Surrogate	X	X	X	X	X	X	X	X	X	
Concentrate	X	X	X	X	X	X	X	X	X	X
Posted By	X	X	X	X	X	X	X	X	X	X
Nonane Lot #		X								
Mineral Oil									X	

*Only add if acid cleanup is needed

ATTACHMENT D: Concentrate Solvent & Final Volume

Matrix	SOIL		WATER	
Analysis	**Solvent**	**Final Volume**	**Solvent**	**Final Volume**
DRO	**DCM**	**5.0mL**	**DCM**	**1.0mL**
DRO SG	**DCM**	**2.0mL:** DRO 1.0mL SG 1.0mL		
PAH	**DCM**	**5.0mL**	**DCM**	**1.0mL**
PCB	Exchange to **Hexane**	**5.0mL**	Exchange to **Hexane**	**1.0mL**
SVOC	**DCM**	**1.0mL**		
PST	**DCM**	**1.0mL**	**DCM**	**1.0mL**
SULFOLANE	**DCM**	**1.0mL**	**DCM**	**1.0mL**

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S.O.P. Title: Purge and Trap Aqueous	**Revision Date**: September 2021
Method No: SW846 8000B & 5030B	**SOP No**: 764r13
Page: 1 of 8	**Supersedes:** 764r12

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Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/7/21	Samara Rentz	9/7/21

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- Updated section 12.4
- Added Attachment A: Volumes to Use for Various Dilutions
- Updated basic formatting, spelling, and grammar throughout SOP

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1.0. OBJECTIVE:

This SOP provides the guidance for purge and trap analysis for aqueous samples.

2.0. SCOPE AND APPLICATION:

This method is used to analyze water and water miscible samples, for volatile organic compounds by purge and trap prep gas chromatograph analysis methods by SW846. (Refer to method SOPs for analytical details.) An inert gas is bubbled through a water sample, reagent water, or methanol extract (for oil, sludge, and soils) contained in a specially designed purging chamber. Heated purging may be used to increase analyte transfer. The purging of the sample transfers purgeable compounds from the liquid to the vapor phase. The vapor is then swept to a porous polymer trap where they are collected. Once purging is complete, the trap is then rapidly heated and back flushed with the inert gas desorbing the analytes onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the compounds, which are then detected.

3.0. DEVIATIONS FROM REFERENCE METHOD:

This SOP contains no deviations from method 5030B.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Samples can be contaminated by diffusion of volatiles (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage.

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5.2. The laboratory where volatile analysis is performed should be completely free of solvents, which are part of the target analyte list.

5.3. The majority of sample contamination occurs at the instrument (purge gas, outgassing ahead of the trap etc.) The instrument must be demonstrated to be free from contamination under the conditions of analysis.

5.4. Contamination due to carryover can occur when high concentration samples and low concentration samples are analyzed sequentially. High concentration samples should be followed by a blank to check for carryover. The system may need to be baked out to eliminate contamination.

6.0. SAMPLE HANDLING:

6.1. Samples are tracked by the sample custodian. TCLP leachates are relinquished to the VOC Analyst by the EPTOX Analyst, along with the associated leachate blank(s) and a copy of the bench sheet for the batch that documents the leaching of the samples, blanks, and their batch assignments. A HSN QC number will be assigned to blanks and noted on the leach log. This number is required in order to track leach blanks from the preparation through the analytical process. LIMS Sample types specific to the different types of leach fluid blanks are available in the analytical queues to post leach blank data.

6.2. Holding Times: Volatile organic analysis of water and water miscible samples pre-preserved with HCl must be performed within 14 days after sample collection. Unpreserved water and water miscible samples, including unpreserved TCLP leachates and blanks must be analyzed within 7 days of the end of the leaching period (fourteen days if the leachate is preserved to pH<2 with 1:1 HCL:water).

6.3. Samples should be stored at 0-6^{o}C with minimal headspace in an area free of solvent fumes.

6.4. The size of any bubble caused by degassing upon cooling should not exceed 5-6 mm ^{2} if a bubble is present, check the vial's cap and septum to ensure a proper seal was made at time of sampling. If there is evidence of leaking the sample should be discarded.

7.0. APPARATUS:

7.1. See method specific SOP for instrumentation (e.g., 8021, 8260, 524, 602)

7.2. Microsyringes for spiking of sample

7.3. Class A volumetric flasks

8.0. REAGENTS:

8.1. Organic free water defined as water in which contamination is not observed at the LOQ value of the parameters of interest. (**Note:** For DOD, the water must be free of contaminants to < ½ LOQ.) Deionized (DI) water is dispensed from the volatiles RO system before using it for sample dilutions, standard preparation, trip blank preparation, and quality control sample preparation.

8.1.1. Acidified RO water, (used to prepare samples and calibrations), is prepared by adding ~1mL of 50% HCL per one liter of reagent water to obtain a pH of ≤ 2.

8.2. Purge and Trap grade Methanol

9.0. EXTRACTION:

N/A

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

11.1 Samples to be analyzed are obtained from the sample custodian.

11.2 Allow samples to come to room temperature prior to analysis.

11.3 Place samples into auto sampler tray. Record any observations of sample color, sediment or bubbles.

11.4 Internal standards or surrogates are added automatically.

11.5 Check sample pH on the remaining volume with narrow range paper after acquisition has finished. If the pH is greater than 2 it must be noted in sample comments in LIMS. Record pH check in the run log. The data is valid if the pH is greater than 2 and analyzed within 7 days.

11.6 Send remaining sample to post analysis

12.0. QUALITY CONTROL:

12.1. Water used for extraction is analyzed in each analytical batch to check for contamination. This check is the instrument blank, which can also serve as the tune (for GC/MS analysis) and a method blank for a water sample batch.

12.2. Each instrument with a purge and trap unit must have a valid calibration curve established, which is verified before use by the analysis of a second source standard. The curve is verified at the beginning of each analytical batch by the analysis of a mid-range calibration standard. Each analytical method contains acceptance criteria for the analysis of these two standards. An average of the response factors or calibration factors from the initial calibration curve is used for surrogate and target analyte calculations.

12.3. MB, LCS, MS and MSD must be associated with each preparation batch of 20 field samples or less. If sufficient field sample volume is not available to create an MS/MSD set, an LCSD sample is added to the batch. See method specific SOP for QC acceptance criteria.

12.4. Dilutions - If dilutions are necessary the amount of purged water must remain constant.

Example (10-fold dilution) – Fill a 50 mL volumetric flask half full with acidified RO water. Add 5 mL of sample and dilute to volume with acidified water. Invert the flask three times and transfer solution to a 40 mL vial. For additional dilutions, see Attachment A: Volumes to use for various dilutions.

12.5. Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

See method specific SOP for information (e.g., 8021, 8260, 524, 602).

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

N/A

20.0. REFERENCES:

SW846 5030B Dec. 1996 Rev. 2, Pg. 1 Sec 2.0
SW846 5030B Dec. 1996 Rev. 2, Pg. 5 Sec. 6.1

21.0. ATTACHMENTS:

Attachment A: Volumes to Use for Various Dilutions

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ATTACHMENT A: VOLUMES TO USE FOR VARIOUS DILUTIONS

	OIL VFC (250µL)	**OIL VMS** (500µL)	**TCLP** (500µL)	**SOIL** (1000µL)	**H2O** (50,000µl)
5X	50 µL	100 µL	100 µL	200 µL	10 mL
10X	25 µL	50 µL	50 µL	100 µL	5 mL
20X	12.5 µL	25 µL	25 µL	50 µL	2.5 mL
50X	5 µL	10 µL	10 µL	20 µL	1 mL
100X	2.5 µL	5 µL	5 µL	10 µL	500 µL
200X	1.25 µL	2.5 µL	2.5 µL	5 µL	250 µL
500X	0.5 µL	1 µL	1 µL	2 µL	100 µL
1000X				1 µL	50 µL

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S.O.P. Title: Purge and Trap Extraction, Non-aqueous	**Revision Date**: September 2021
Method No: SW846 5035A	**SOP No**: 767r13
Page: 1 of 11	**Supersedes**: 767r12

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Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	10/1/21	Jamaica Renty	10/1/21

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Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

Summary of Changes from Previous Revision:

- Addendum incorporated
- Reference to 17 mL vial removed from section 7.2
- Added 1000X Dilution to Attachment B.
- Removed all references to Encore samples. Subsequent sections were renumbered, where applicable.
- Minor grammatical fixes throughout

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1.0. OBJECTIVE:

This method is used to extract sediment/soil, solid waste and oil samples for volatile gas chromatograph methods by SW846 5035A.

2.0. SCOPE AND APPLICATION:

2.1 Soil and solid waste samples are typically preserved in the field with methanol. They may be sodium bisulfate preserved or collected and frozen for low level analysis. The preserved/extracted sample is then sent to the laboratory for analysis. At the time of writing this SOP, the State of Alaska Department of Environmental Conservation has not approved the use of sodium bisulfate preservation technique. SGS recommends the use of the low-level freezing technique (i.e., 5 grams of soil in 5 ml DI water with stir bar). This SOP will cover the procedure for extracting samples with methanol at the laboratory.

2.2 A portion of sample is weighed and extracted with BFB methanol. Due to the volatile nature of the components of interest this procedure must be done before the sample is used in any other analysis. Samples extracted by this technique at the laboratory may be considered invalid due to volatile loss before the sample arrives at the laboratory or during the transfer procedure. **Samples received at the laboratory for extraction will be flagged as biased low on the Sample Receipt Form**. Extraction efficiency for solid waste and oil samples may not be the same as for the LCS/LCSD that are extracted with them.

3.0. DEVIATIONS FROM REFERENCE METHOD:

Oil analysis quality control will consist of: An acceptable curve, Initial tune, Initial calibration blank, and a Continuing Calibration Verification.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

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4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

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4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

Samples can be contaminated by diffusion of volatiles (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. Trip blanks which accompany the samples during shipment should be analyzed as a check of such contamination.

6.0. SAMPLE HANDLING:

6.1. Jars for field preserved methanol samples are weighed before they are sent in the client sample kit. These jars must be weighed again when the sample is received at the lab, prior to any analysis, to determine sample mass per SGS Weighing SOP#143. If a non-tared jar is received, the sample jar may be emptied and weighed post analysis for an estimated jar weight; however, this technique is not recommended.

6.2. All samples for volatile analyses should be stored in a location free from volatile contaminants. Field extracted and in-house extracted soils are stored at 0-6° C in the reach-in fridge that is located inside the volatiles prep room. When it is determined that no further analysis is necessary, samples are stored at 0-6° C in Sample Control's GC reach-in for methanol extracted soils. Methanol extracted soils must be segregated from all other samples to prevent contamination. Low-level frozen vials are stored at -7 to -20° C in Sample Control's freezer to segregate them from other samples, etc.

6.3. Holding Times:

6.3.1. Samples preserved/extracted in the laboratory: Volatile organic analysis of soils, oils and solid/sludge samples must be performed within 14 days after sample collection. ADEC requires samples to be preserved within 10 seconds of collection. If not, field preserved the extraction/preservation should occur as soon as possible after sample receipt.

6.3.2. Samples preserved/extracted in the field (methanol and sodium bisulfate): Analysis for soil samples must be performed within 14 days (depending on analysis method). Check backlog for the holding time status. Best results are obtained by allowing the preserved sample to stand under the methanol for at least 24 hours.

7.0. APPARATUS:

7.1. Balance - Analytical, capable of accurately weighing 0.01 g.

7.2. Glass vials – 40mL and 4-oz with Teflon-lined caps.

7.3. Disposable pipettes: Pasteur, serological glass 10ml

7.4. Pipette bulbs

7.5. Refrigerator for extract storage located in Volatile Department.

7.6. Class A volumetric flasks: 25mL, 50mL, 100mL, 200mL and 500mL

7.7. Hamilton gas tight syringes: 5, 10, 50, 100, 250, 500µL, 1, 2.5, 5, and 10mL

8.0. REAGENTS:

8.1. Methanol – Purge and Trap grade, Fisher Chemical High Quality Solvent – Methanol.

8.2. BFB MeOH for field preserved samples: This solution is prepared in the laboratory at a concentration of 2.5 µg/mL BFB in MeOH.

8.2.1. Start by rinsing a glass funnel and 2,000 mL flask with methanol 3 times. Fill the flask with methanol and pour carefully into a 4L storage container, repeat this step once more. Using a 1.0 mL syringe, remove exactly 1 mL of methanol from the storage container.

8.2.2. Before BFB Standard Stock (Restek cat# 30082 1,4-bromofluorobenzene (BFB) @ 10,000µg/mL in methanol or equivalent) may be used, invert the vial 3 times slowly. Rinse the 1.0 mL syringe with a small portion (~0.05mL) of the BFB stock before adding 1.0 mL to the container (partly submerge the needle before injecting the BFB stock). Ensure the cap is tightly closed and homogenize vigorously. If possible, the container should be allowed to sit overnight to achieve homogeneity before the test vial is prepared.

8.2.3. A test vial must be analyzed before the BFB MeOH may be used. To prepare the test vial, 1.0mL of the BFB MeOH is diluted with acidified RO water from the volatiles lab to 50mL using a 50mL volumetric flask. The solution is then poured into a 40mL amber vial and given to the analyst for evaluation. Once analyzed, the analyst will approve that lot of BFB MeOH by completing the "Approved by" section of the label on the 4L container.

8.2.4. *Note: If surrogated methanol is purchased prepared from a vendor, an SGS purchasing lot number (per SGS SOP#112) is used.*

8.3. Working solutions and standards: See method specific SOP#710 for GRO/BTEX and SOP#783 for 8260. See Attachment A: Extraction Q.C.

9.0. EXTRACTION:

9.1 Refer to SOP#143 for weighing procedures. Prepare containers for extraction of quality control samples.

9.1.1. Remove the cap, place the jar (4-oz) on the balance, shut the balance door and tare the balance.

9.1.2. Weigh out 50g of sample for preservation/extraction. Add 25mL of 2.5µg/mL BFB methanol as quickly as possible, and cap vial quickly. The methanol extract is then stored at 0-6ºC for 24hrs before analysis.

9.2. All samples should be refrigerated for a minimum of 24 hours after methanol addition.

9.3. Samples should be allowed to come to room temperature prior to analysis.

9.4. OILS/ORGANIC LIQUID WASTE EXTRACTION

9.4.1. Weigh 1.0 g (± 0.1g) of the sample, (matrix 3 oil) into a screw capped septum sealed vial. Record the weight to two decimal places. Immediately add 5.0 ml BFB methanol.

9.4.2. If the sample dissolves in methanol it is ready for analysis. Excessive sample handling will result in loss of volatile compounds.

9.4.3. Samples that are not miscible in methanol must be stored at 0-6º C for 24 hours before analysis.

9.4.4. Record anything unusual about the sample or any apparent odors.

9.4.5. The standard dilution used is: 100µl/50mL, post as 5X. For additional dilutions, see Attachment B: Volumes to use for various dilutions.

9.5. **Multi-Incremental (MI) Soil Samples:** Special preparation is required before running multi-incremental (MI) soil samples. Using a 5mL syringe, remove 5mL of methanol from each of the provided sample containers (i.e. 1A, 1B, 1C, etc.) and combine them into a single VOA vial. If it is not possible to get 5mL of methanol out of each container, then a lesser volume may be used, but the methanol volume removed from each container must be equal. The combined methanol extracts will constitute the final sample for preparation and analysis as per SOP#783.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

See method specific SOP#710 for GRO/BTEX and SOP#783 for 8260.

12.0. QUALITY CONTROL:

12.1. For soil samples, a separate aliquot will be provided for percent solids determinations per SOP#115.

12.2. Each soil batch will have a MB, LCS, and MS/MSD. A MS/MSD is not required for GRO methods unless requested by the client. A LCSD may be used if insufficient sample is available for MS/MSD.

12.3. Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1. Moisture Adjustment for Methanol Preserved Soils:

% Moisture = 100 – % Solid EQUATION 1

Final Volume = ((Mass of sample * % Moisture) / 1000 + Volume of MeOH EQUATION 2

13.2. Dilution Factor: Soil

(wet wt, g) (% Solid) = Dry weight, g EQUATION 3

$$\text{High level D.F.} = (5\text{ mL MEOH} / \text{Dry weight, g}) * \frac{5\text{ mL water}}{\text{Amount injected, mL}} \quad \text{EQUATION 4}$$

* Note for low level soil methods 8260:

Low level D.F. = (5 g water / Dry weight of sample, g) EQUATION 5

13.3. Dilution Factor: oil

(Weight of oil, g) / Density of oil, g/mL = Volume of oil, mL (if required) EQUATION 6

Oil DF = 5 mL of MEOH / volume of oil, mL * 5 mL water / Amount injected, mL

or Oil DF = 5 mL MEOH / mass of oil, g * 5 mL water / Amount injected, mL EQUATION 7

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP#108 for further instruction.

14.3. All surplus reagent acids shall be neutralized daily using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves, and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

Method performance does not apply to this SOP.

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

N/A

20.0. REFERENCES:

SW846 8000B (Dec. 1996)
SW846 5035A (July 2002)

21.0. ATTACHMENTS:

Attachment A: Extraction QC
Attachment B: Volumes to Use for Various Dilutions

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ATTACHMENT A: EXTRACTION Q.C.

BTEX/GRO Soil QC setup	Standard added	MeOH (µL)	Final Volume
IB/MB		2mL	100mL
NAS	100µL NAS		100mL
CCV2/CCV	100µL CCV2/CCV		100mL
LCS/LCSD BTEX/GRO	50µL CCV2/CCV	2mL	100mL
MS/MSD soil BTEX/GRO	50µL CCV2/CCV	2mL sample	100mL

BTEX/GRO Water QC setup	Standard added	MeOH (µL)	Final Volume
IB/MB		2mL	100mL
NAS	100µL NAS		100mL
CCV2/CCV	100µL CCV2/CCV		100mL
MS/MSD water	100µL CCV2/CCV	All Sample	100mL

8260 Water samples QC setup	Internal Standard / Surrogate	Primary CCV/ Early Gas	MeOH (µL)	Final Volume
IB / MB	Added by system			100mL
CCV / LCSD	Added by system	60µL	680µL	100mL
Matrix Spike	Added by system	60µL		100mL
Matrix spike Dup	Added by system	60µL		100mL
ICV	Added by system	60µL 2nd source	680µL	100mL

8260 Soil samples QC setup	Internal Standard / Surrogate	Primary CCV/ Early Gas	MeOH (mL)	Final Volume
IB / MB	Added by system		2mL	100mL
CCV / LCS	Added by system	60µL each mix	1.8mL	100mL
Matrix Spike	Added by system	60µL each mix	2mL sample	100mL
Matrix Spike Dup	Added by system	60µL each mix	2mL sample	100mL
ICV	Added by system	60µL 2nd source	1.8mL	100mL

524.2 WATER QC:					
	Methanol	**CCV Standard**	**Surrogate**	**EG Standard**	**Sample**
CCV	680µl	60µl		60µl	

Soil EDB samples QC setup	Internal Standard / Surrogate	EDB Standard	MeOH (mL)	Final Volume
IB / MB	Added by system		2mL	100mL
CCV / LCS	Added by system	80µL	1.92mL	100mL
Matrix Spike	Added by system	80µL	2mL sample	100mL
Matrix Spike Dup	Added by system	80µL	2mL sample	100mL
ICV	Added by system	80µL 2nd source	1.92mL	100mL

Water EDB samples QC setup	Internal Standard / Surrogate	EDB Standard	MeOH (mL)	Final Volume
IB / MB	Added by system			100mL
CCV / LCSD	Added by system	80µL	320µL	100mL
Matrix Spike	Added by system	80µL		100mL
Matrix Spike Dup	Added by system	80µL		100mL
ICV	Added by system	80µL 2nd source	320µL	100mL

ATTACHMENT B: VOLUMES TO USE FOR VARIOUS DILUTIONS

	OIL VFC (250µl)	**OIL VMS** (500µL)	**TCLP** (500µL)	**SOIL** (1000µL)	**H20** (50,000µL)
5X	50µl	100µL	100µL	200µL	10mL
10X	25µl	50µL	50µL	100µL	5mL
20X	12.5µl	25µL	25µL	50µL	2.5mL
50X	5µl	10µL	10µL	20µL	1mL
100X	2.5µl	5µL	5µL	10µL	500µL
200X	1.25µl	2.5µL	2.5µL	5µL	250µL
500X	0.5µl	1µL	1µL	2µL	100µL
1000X				1µL	50µL

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
NOTE: This document contains CONFIDENTIAL business information and is not intended for distribution.	
S.O.P. Title: Purgeable Organic Compounds Analysis by GC/MS and GC/MS-SIM	**Revision Date**: September 2021
Method No: 8260D and 624	**SOP No**: 783r06
Page: 1 of 26	**Supersedes**: 783r05

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	10/1/21	Jamaun Penty	10/1/21

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This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

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Each staff member responsible for this SOP will print & sign this cover page upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- All references to 8260C were removed and replaced with 8260D to reflect current practices.
- 12.14.1 was added to include reference to surrogate associations.
- Removed references to 8260-modified.
- Updated Table 1: Tune criteria
- Small grammar and formatting corrections made throughout.

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1.0. OBJECTIVE:

This document outlines a technique and control parameters for identifying and determining the concentrations of volatile organic compounds in various sample matrices. The instrument used for analysis is a GCMS (gas chromatograph/mass spectrometer).

2.0. SCOPE AND APPLICATION:

2.1. Method 8260 is used to determine volatile organic compounds in a variety of waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludge, waste solvents, oily wastes, mousses, tars, fibrous waste, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

2.2. The following analytes can be determined by this method. It is possible to add analytes to this list on a project-by-project basis upon client request; however, special arrangements may be required.

Analyte	CAS Number	Analyte	CAS Number
Chloromethane	74-87-3	Vinyl chloride	75-01-4
Bromomethane	74-83-9	Chloroethane	75-00-3
1,1-Dichloroethane	75-34-3	Carbon Disulfide	75-15-0
Methylene chloride	75-09-2	*trans*-1,2-Dichloroethene	156-60-5
1,2 - Dichloroethane	107-06-2	*cis*-1,2-Dichloroethene	156-59-2
Chloroform	67-66-3	1,1,1-Trichloroethane	71-55-6
1,1-Dichloroethene	75-35-4	Benzene	71-43-2
2-Butanone	78-93-3	1,2-Dichloropropane	78-87-5
Carbon tetrachloride	56-23-5	Methyl-tert-butyl ether	1634-04-4
Trichloroethene	79-01-6	*cis*-1,3-Dichloropropene	10061-01-5
Bromodichloromethane	75-27-4	1,1,2-Trichloroethane	79-00-5
trans-1,3-dichloropropene	10061-02-6	Bromoform	75-25-2
Dibromochloromethane	124-48-1	MIBK	108-10-1
2-Hexanone	591-78-6	Tetrachloroethene	127-18-4
Toluene	108-88-3	Ethylbenzene	100-41-4
Chlorobenzene	108-90-7	*o*-Xylene	95-47-6
p&m-Xylene	108-38-3, 106-42-3	1,1,2,2-Tetrachloroethane	79-34-5
Styrene	100-42-5	1,3-Dichlorobenzene	541-73-1
1,2-Dichlorobenzene	95-50-1	1,4-Dichlorobenzene	106-46-7
Bromochloromethane	74-97-5	1,3-Dichloropropane	142-28-9
Dichlorodifluoromethane	75-71-8	Dibromomethane	74-95-3
2,2'-Dichloropropane	591-20-7	Trichlorofluoromethane	75-69-4
1,2,3-Trichlorobenzene	87-61-6	1,1-Dichloropropene	563-58-6
Isopropylbenzene	98-82-8	1,1,1,2-Tetrachloroethane	630-20-6
1,2,3-Trichloropropane	96-18-4	Bromobenzene	108-68-1
2-Chlorotoluene	95-49-8	*n*-Propylbenzene	103-65-1
1,3,5-Trimethylbenzene	108-67-8	4-Chlorotoluene	106-43-4
1,2,4-Trimethylbenzene	95-63-6	*tert*-Butylbenzene	98-06-6
1,2-Dibromo-3-chloropropane	96-12-8	*sec*-Butylbenzene	135-98-8
Hexachlorobutadiene	87-68-3	1,2,4-Trichlorobenzene	120-82-1
n-Butylbenzene	104-51-8	Naphthalene	91-20-3
1-Chlorohexane	544-10-5	Acetone	67-64-1
Vinyl acetate	158-05-04	Freon 113	76-13-1
1,2-Dibromoethane	106-93-4	4-Isopropyltoluene	99-87-6

Methyl Iodide	74-88-4	Acrylonitrile	107-13-1
trans 1,4-Dichloro-2-Butene	110-57-6	Cyclohexane	110-82-7
n-hexane	110-54-3		

2.3. Method 624 is used determine volatile organic compounds in a water matrix. The following analytes can be determined, and are routinely analyzed, by this method. It is possible to add analytes to this list upon client request.

Analyte	**CAS Number**	**Analyte**	**CAS Number**
Chloromethane	74-87-3	Vinyl chloride	75-01-4
Bromomethane	74-83-9	Chloroethane	75-00-3
1,1-Dichloroethane	75-34-3	*cis*-1,2-Dichloroethene	156-59-2
Methylene chloride	75-09-2	*trans*-1,2-Dichloroethene	156-60-5
1,2 - Dichloroethane	107-06-2	1,1,1-Trichloroethane	71-55-6
Chloroform	67-66-3	Benzene	71-43-2
1,1-Dichloroethene	75-35-4	1,2-Dichloropropane	78-87-5
Carbon tetrachloride	56-23-5	*cis*-1,3-Dichloropropene	10061-01-5
Trichloroethene	79-01-6	1,1,2-Trichloroethane	79-00-5
Bromodichloromethane	75-27-4	Bromoform	75-25-2
trans-1,3-dichloropropene	10061-02-6	Tetrachloroethene	127-18-4
Dibromochloromethane	124-48-1	Ethylbenzene	100-41-4
Toluene	108-88-3	*o*-Xylene	95-47-6
Chlorobenzene	108-90-7	1,1,2,2-Tetrachloroethane	79-34-5
p&m-Xylene	108-38-3, 106-42-3	1,3-Dichlorobenzene	541-73-1
Styrene	100-42-5	1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1	Trichlorofluoromethane	75-69-4
Total Xylenes			

2.4. An autosampler is used to move a 5-mL or 25-mL aliquot of a sample into a specially designed purging tube. An inert gas, such as nitrogen, helium is bubbled through the tube at 40°C (or 60ºC for 8260D-SIM). This transfers purgeable compounds as a vapor phase. The vapor passes through a sorbent trap. Once purging is complete, the trap is then heated and back flushed with inert gas desorbing onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the compounds, which are then detected with a mass spectrometer.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. 8260: Surrogates are spiked by the instrument, into all calibration levels, blanks, and all analysis at 30µg/L. For soils, BFB is spiked into all calibration level, blanks, and all analysis at 80µg/L.

3.2. 8260: The CCV, LCS, LCSD, MS, & MSD will be prepared using the primary standards.

3.3. A second source ICV is run to verify each calibration acceptance criteria per the DOD QSM. The recovery for all 8260 compounds should be ±20%.

3.4. 624: The trap material and temperatures. Note: There have been substantial improvements to instrumentation and trap packing material since Method 624 was revised.

3.5. 624: Internal standards and surrogates are as listed in this document in section 8.8.

3.6. 624 QC from published method sections (8.1.–8.7.) will be replaced by these SOP sections (8.5. - 8.11.) and section 9.0

3.7. BFB Key Ion Abundance Criteria for MS Tuning will be as listed in section 12.1 of this SOP and will replace section 10 of method 624.

4.0. RESPONSIBILITIES:

4.1. The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

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5.0. INTERFERENCES:

There are three primary sources of contamination:

5.1. Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for most contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of analysis by running laboratory reagent blanks each day. The use of non-Teflon plastic tubing, non-Teflon threads sealant, or flow controllers with rubber components in the purge and trap system should be avoided.

5.2. Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

5.3. Contamination by carry-over can occur whenever high level and low-level samples are analyzed sequentially. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Unusually concentrated samples should be followed by analysis of reagent water to check for cross contamination. Since the trap and other parts of the system are also subject to contamination, baking and purging of the entire system may be required.

5.4. Some liquid samples foam up with agitation. This causes contamination in the lines going from the sparge tube. Antifoam is used to inhibit foam production.

6.0. SAMPLE HANDLING:

6.1. Proper Handling: The samples must be iced or refrigerated at 0 – 6ºC from the time of collection until analysis. Frozen soil in 40ml purge vials and Encore samples must be kept at less than or equal to -10°C until analysis or, in the case of Encores, extraction with methanol.

6.2. Holding Time: All properly preserved water, soil, oils, and sludge samples must be analyzed within 14 days of collection. Water samples not preserved to a pH of less than two must be analyzed within 7 days of collection.

6.3. Storage Procedures: Sample bottles must remain hermetically sealed and stored in a location free from any solvents and isolated from all standards and standard solutions.

6.4. Preservation: Water samples are collected in vials containing 0.5mL of 50% HCl as a preservative. This adjusts the pH of the sample to less than 2. Sodium thiosulfate or ascorbic acid must be added prior to acidification if source is chlorinated. Soil samples should be preserved with methanol for medium-high concentration samples or, for low concentration samples, sodium bisulfate or DI water before freezing (refer to SGS SOP#767). In the case of the latter, a methanol preserved sample volume must also be collected in the event concentrations above the calibration range are encountered in the analysis of the sodium bisulfate or frozen aliquot.

6.5. When prepping samples for volatile analysis the only type of gloves that can be used is neoprene. Latex and nitrile gloves will result in carbon disulfide contamination.

7.0. APPARATUS:

7.1. Sample Vials (aqueous samples) – 40mL capacity equipped with a screw cap with a Teflon-faced silicone septum.

7.2. Sample jars (solid samples) - glass jars equipped with a Teflon-lined screw cap lid.

7.3. The Closed System Autosampler, Centurion has vial trays designed to hold 40mL vials. The Centurion holds 100 vials. The system automatically transfers a 5mL/25mL aliquot of sample to a purge vial for purging. The gaseous headspace between the water column and the inlet should be a total volume of less then 15mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3mm at the origin. The purge gas must be introduced no more than 5mm from the base of the water column.

7.4. Purge and Sample Concentrator Module - The hydrocarbon trap must be at least 25cm long and have an inside diameter of at least 0.105in. The desorber must be capable of rapidly heating the trap.

7.5. Gas Chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, and detector. (Agilent Technologies 7890B/7890A)

7.6. Mass Spectrometer - Capable of scanning from 35 - 300amu every 1 seconds or less, using 70 volts (nominal) electron energy in the electron impact mode and producing a mass spectrum that meets all the criteria in Table 1 when 5-50ng gets to the detector by purging 4-Bromofluorobenzene (BFB) as part of the instrument blank. If 3uL of 50ng/uL SURR solution is added to the samples, that equals 15ng of 4-BFB to the detector assuming a 10:1 injector split.

7.7. Software: MSD Chemstation and Mass Hunter are used for data analysis.

7.8. Class A volumetric flasks: 25mL, 50mL, 100mL, and 500mL and gas tight syringes: 5, 10, 50, 100, 250, 500µL, 1, 2.5, 5, and 10mL.

7.9. Narrow range pH test strips: pH 0.5-5.5

7.10. For instrument specific operating parameters see the instrument maintenance manual.

7.11. Analytical traps – 'K' type trap for 8260 and SIM EDB analysis, 'J' type trap for 1,4-dioxane.

8.0. REAGENTS:

Note: All in-house standards have an expiration date of one year from production; providing it does not surpass the vendor's expiration date*

8.1. Reagent water – organic free RO water in which the analytes of interest are not observed at ½ the LOQ values.

8.1.1. Acidic RO, (used to prepare samples and calibrations), is prepared by adding ~4mL of 1:1 HCL to 4 liters of reagent water to obtain a pH of ≤ 2.

8.2. Hydrochloric acid (1:1) - Add 50mL of concentrated HCl to 50mL of reagent water.

8.3. Methanol – Purge and Trap grade, Fisher Chemical High Quality Solvent – Methanol

8.4. 20% Sodium Bisulfate solution – Add 100g of sodium bisulfate (Fisher Scientific part # 5240-500) to 500mL of reagent water.

8.5. Calibration and quality control standards for 8260: Certified solutions are purchased from two venders, to be used as calibration and second source standards. The vendors will be chosen from the "ISO Guide 34" list when possible.

8.5.1. Primary 8260 Early Gas Solution: Using a gas tight syringe transfer 1.25 mL each Restek (2000µg/mL) vinyl acetate standard and Agilent (2000µg/mL) VOC gas standard into a 50mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields 50/50µg/mL solution.

8.5.2. Primary 8260 CCV Solution: Using a gas tight syringe transfer 1.25mL Restek (2000µg/mL) 502.2 Cal 2000 mega-mix, 0.75mL of Restek (5000µg/mL) custom SGS-8 primary standard, and 1.25mL of Restek (5,000µg/mL) VOA calibration mix #1 into a 50mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields a 50/75/150µg/mL solution.

8.5.3. Primary Source 8260-SIM: Using a gas tight syringe transfer 125µL Restek (200µg/mL) 504.1 calibration mix and 250µL Restek (2000µg/mL) 1,4-dioxane standard into a 100mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields 0.25/5µg/mL solution.

8.5.4. Second Source 8260, Early Gas Solution (ICV EG): Using a gas tight syringe transfer 1.25mL each Restek (2000µg/mL) 502.2 calibration mix #1 and SPEX (2000µg/mL) vinyl acetate standard

into a 50mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields 50/50µg/mL solution.

8.5.5. Second Source 8260 CCV Solution (ICV): Using a gas tight syringe transfer 1.25mL Ultra (2000µg/mL) VOC standard, 0.75m/L of Restek (5000µg/mL) custom SGS-8 secondary standard, and 1.25mL of Ultra (5,000µg/mL) ketones standard into a 50mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields a 50/75/150µg/mL solution.

8.5.6. Second Source 8260-SIM (ICV): Using a gas tight syringe transfer 125µL Ultra (200µg/mL) EPA method 504.1 mixture and 250µL Ultra (2000µg/mL) 1,4-dioxane solution into a 100mL volumetric flask filled up to the neck with methanol. Bring to volume with methanol and invert three times. This yields 0.25/5µg/mL solution.

8.6. Spike solution (SPK) – Use the appropriate Primary Standard to spike samples (MS and MSD) when required.

8.7. Internal Standard and Surrogate Solution for Soil and EDB/1,4-Dioxane.

8.7.1. Surrogate solution (SUR) for 4-Bromofluorobenzene: Place approximately 40mL of methanol in a 50mL class A volumetric flask. Inject 0.75mL Restek (10,000 µg/mL) 4-Bromofluorobenzene standard into the methanol. Bring to volume with methanol. Invert three times. This yields a 150µg/mL surrogate solution.

8.7.2. Internal Standard Solution (INT) and Surrogates 1,2-Dichloroethane-d4 and Toluene-d8: Place approximately 80mL of methanol in a 100mL class A volumetric flask. Inject 0.3mL Restek (10,000 µg/mL) custom VOA internal standard mix, 1.5mL Restek (2000µg/mL) 1,2-dichloroethane-d4 mixture, and 1.5mL Restek (2000µg/mL) toluene-d8 standard into the methanol. Bring to volume with methanol. Invert three times. This yields a 30/30/30µg/mL internal standard/surrogate solution. This mix is used 3.0µl per 25mL sample.

8.8. Combined Internal Standard and Surrogate mix for Water, Closed System Centurions.

8.8.1. Place approximately 80mL of methanol in a 100mL class A volumetric flask. Inject 0.3mL of Restek (10,000µg/mL) custom VOA internal standard mix and 1.2mL of Restek (2,500µg/mL) VOA surrogate spike mix into the methanol. Bring to volume with methanol. Invert three times. This yields a 30/30µg/mL solution for the 5mL purge.

8.9. Purchased standards should not be used past manufacturer's expiration date. Standards prepared from stock solutions are valid for one year from creation, manufacturer's expiration date, or when signs of degradation are observed.

8.9.1. Transfer each stock standard solution into its own Teflon-sealed screw cap bottle and store in the freezer protected from light with minimal head space. Label and date bottle in accordance with the Laboratory Standards Labeling SOP #112.

8.10. BFB MeOH for field preserved samples: This solution is prepared in the laboratory at a concentration of 2.5µg/mL BFB in MeOH. Fill a 2,000mL flask more than halfway with methanol. Using a 1.0mL syringe, add 1.0mL Restek Car #30082 4-Bromofluorobenzene Standard (initial concentration 10,000 µg/mL), and fill to the mark with additional methanol. The flask is then carefully inverted three times and placed into a 4 L storage container. The flask is filled a second time to mark with methanol and added into the 4 L container. Before the BFB MeOH may be used, a test vial must be prepared and analyzed. To prepare the

test, inject 1.0 mL of the BFB MeOH into a 50mL flask filled up to the neck with reagent water, then fill to the mark with additional reagent water. That solution is then poured into a 40mL amber vial and given to the analyst for evaluation. Once analyzed, the analyst will approve that lot of BFB MeOH by completing the "Approved by" section of the label on the 4L container.

8.11. Antifoam: liquid polymer without volatile compound interferences, used to reduce surface tension.

9.0. EXTRACTION:

Refer to SGS SOPs 764 and 767.

10.0. CALIBRATION:

10.1. Before recalibrating, each instrument shall be tuned using PFTBA. Compare abundance of ion 69 to ratios of ions 131 & 219, and peak widths of each ion to previous tune print outs. If out of control values are observed, perform MS autotune and then retune manually. When acceptable results are gained, the tune (along with parameters) will be printed and filed in instrument specific binders.

10.1.1. Calibration Setup – Calibration standards are prepared from the primary solutions in the previous section.

10.1.2. The compounds from the custom 8 analyte standards are calibrated at 1.5 times the "normal" concentration of 0.4 – 70µg/mL. Calibrated at 2 times the "normal" concentration is: p&m xylene. Calibrated at 3 times the "normal" concentration are: acetone, 2-butanone, 4-methyl-2-pentanone, and 2-hexanone.

Waters Table:
For 5mL purge

Prepared in acidic DI water

Conc. (µg/L)	CCV Std. and Early Gas Std. (µL in 100mL)	Methanol (µL in 100mL)
0.075	0.15	800
0.4	0.8	798
1.0	2.0	796
3	6.0	788
10	20	760
30	60	680
50	100	600
70	140	520
200	400	0

Transfer to two 40mL vial. The IS and Surrogate are added automatically (30µg/L) by the system.

Soils Table:
For 5mL purge

Prepared in DI water

Conc. (µg/L)	CCV Std. and Early Gas Std. (µL of each)	Methanol (mL in 100mL)
0.4	0.8	1.998

1.0	2.0	1.996
4.0	8.0	1.984
8.0	16	1.968
30	60	1.88
50	100	1.8
70	140	1.72
200	400	1.2

Transfer to two 40mL vial. The IS and surrogate are added automatically (30µg/L) by the system.

Low Level Soils Table:
For methanol 25mL purge

Prepared in DI water

Conc. (µg/L)	CCV Std. and Early Gas Std. (µL of each)	Methanol (mL in 100mL)
0.02	0.02 (in 500mL)	10 (in 500mL)
0.08	0.08 (in 500mL)	10 (in 500mL)
0.4	0.8	1.998
1.0	2.0	1.996
4.0	8.0	1.984
8.0	16	1.968
30	60	1.88
50	100	1.8
100	200	1.6

Transfer to two 40mL vial. The IS and surrogate are added automatically (30µg/L) by the system.

Low Level Soils Table
For 5mL in-vial purging

Conc. (µg/L)	CCV/Early Gas Std. (µL/ea)	Final vol. (mL)	MeOH (µL)
0.4µg/L	0.8	100	279
1.0µg/L	2	100	278
3.0µg/L	6	100	268
10µg/L	1	5	12
30µg/L	3	5	8
50µg/L	5	5	4
70µg/L	7	5	0

A 5mL aliquot is then added to a 40mL vial containing blank sand and fitted with a stir-bar. The IS and Surrogate are added automatically (30µg/L) by the system.

EDB/1,4-dioxane
Waters Table:

Conc. (µg/L)	EDB CCV Std. (µL)	Methanol (µL in 100mL)
.005	2	398
.01	4	396
.02	8	392

Controlled copy distributed to Brie Hicknell with VHB on 04/01/2022.

.05	20	380
.1	40	360
.2	80	320
.4	160	240
1.0	400	0

Transfer to two 40mL vial. The IS and surrogate are added automatically by the system.

EDB/1,4-dioxane
EDB Soils Table:

Conc. (µg/L)	EDB CCV Std. (µL)	Methanol (mL in 100mL)
.005	2	1.998
.01	4	1.996
.02	8	1.992
.05	20	1.980
.1	40	1.960
.2	80	1.920
.4	160	1.840
1.0	400	1.6

Transfer to two 40mL vial. The IS and surrogate are added automatically by the system.

10.2. Calibration Processing Steps:

10.2.1. Load the current calibration for the instrument of interest. (VRA, VQA, etc.)

10.2.2. Save the method to the date run. (VSAYYYY-MMDD.M)

10.2.3. Load the data file. Load the 30 point for 8260.

10.2.4. Quant the 30 point.

10.2.5. Init. Cal. – update levels – retention times

10.2.6. Init. Cal. – Clear all calibration responses.

10.2.7. Init. Cal. – update levels – responses

10.2.8. Init. Cal. – Global Update – set curve fit – average response

10.2.9. Save Method

10.2.10. Quant 30 point.

10.2.11. Con. Cal. – Save ISTD responses

10.2.12. Q Edit 30 point. All values should be 30.00, 45.00, or 90.00 respectively. Save Method.

10.2.13. If all values do not meet the exact value, repeat the process, after saving the method.

10.2.14. Save the method.

10.2.15. Quant each of the other points of the curve, Q Edit, Save, Update responses in Init. Cal., Save Method.

10.2.16. Review each analyte to meet QC criteria per section 10.5.

10.3. To evaluate the calibration, a response factor report is prepared. The report calculates the average response factor for each analyte and the relative standard deviation between response factors for the different concentrations.

10.3.1. Each of the most common analytes in the calibration verification standard should meet the minimum response factors as noted in Attachment B.

10.3.2. If the minimum response factors are not met the system should be evaluated and corrective action should be taken before samples are analyzed.

10.3.3. All target compounds of interest must be evaluated using a 15% variability criterion. Use percent difference when performing the average response factor model calibration.

10.4. System performance check compounds (SPCC) -8260D does not have SPCCs.

10.5. The criteria used for curve fit is as follows:

10.5.1. Average Response Factor (RRF): (Preferred), Analytes with an RSD less than or equal to 15% for their average response factor are quantitated using the average factor. A minimum of 5 points are required. If the RSD is greater than 15% then one of the following options will be used.

10.5.2. Linear (1^{st} order) least squares regression: This option requires a minimum r^2 value of 0.990 (or a r value of 0.995). A minimum of 5 points are required. As per method 8000C, forcing through zero is not permitted.

10.5.2.1. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ±30% of the standard's true concentration.

10.5.2.2. Analytes which do not meet the minimum quantitation calibration refitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/ or reporting those "out of control" target analytes as estimated when concentration is at of near the lowest calibration point may be appropriate.

10.5.3. Quadratic non-linear (2^{nd} order) regression: This option requires a minimum r^2 value of 0.990 (or an r value of 0.995). A minimum of 6 points are required. As per method 8000C, forcing through zero is not permitted.

10.5.4. The use of weighted least squares regression may be used. The deciding factor in the use of these techniques will be the residuals for the calibration points (which fit best matches the points). The residual is the difference between the known concentration of the calibration point and the

calculated value based on the calibration curve. The analyst should strive to reduce the residual error at each point in the calibration to less than 20%.

10.6. Relative retention times (RRT) for all calibration points must agree within 0.06 RRT units from the mid point of the calibration curve. RRT= RT Analyte (minutes)/RT Internal Standard (minutes).

10.7. After a calibration is complete, initial calibration verification (ICV) is run using a Second Source Solution (sections 8.5.3. and 8.5.4). Goals are achieved if all analytes are ± 20% of the calculated true value of the solution.

11.0. ANALYSIS:

11.1. Initial screening of samples is done by visually inspecting the appearance of the sample. The LIMS queue backlog is checked for the required analysis and special QA/QC requirements.

11.1.1. Liquid samples that demonstrate a tendency to foam with agitation are treated with antifoam to inhibit the formation of foam in the sparge tube. A blank sample, with antifoam added, is processed with any samples analyzed with the addition of antifoam.

11.2. For water samples – After allowing the sample to come to ambient temperature place the 40mL vial on the Centurion autosampler. The Centurion will remove a 5mL aliquot from the vial and before transferring will inject IS/Surr mix to yield a final concentration of 30µg/L. After the analysis, the pH of the sample is taken using narrow range pH indicator strips (pH 0.5 - 5.5) from the sample remaining in the vial and recorded on runlog.

11.3. All TCLP leachates and 100% liquid TCLP samples must come to ambient temperature before analysis. These samples are analyzed at a 50X dilution or per client request. This meets the regulatory TCLP limits. 100% liquid TCLP samples must be filtered prior to analysis using a syringe filter. Matrix "6" samples that are not TCLP analyses are diluted 50X before analysis. Matrix "6" samples that are not TCLP analyses may or may not be filtered prior to analyses per client request. Matrix "6" TCLP samples are filtered and analyzed at a 50X dilution.

11.4. For methanol preserved soil samples – Preparation of this matrix requires dilution prior to analysis. After allowing the sample to come to ambient temperature a 1.0mL aliquot of the methanol extract is diluted to 50mL. Transfer solution to a 40mL vial and place on the autosampler for analysis. The Centurion will remove a 5mL or 25mL aliquot from the vial and before transferring will inject IS and surrogate to yield a final concentration of 30µg/L.

11.5. Frozen (thawed soil/DI water) and sodium bisulfate preserved soil samples are set-up on the closed system instrument as received from the field. Refer to SOPs # 764, & 767 (methods 5030C and 5035A) for purge and trap extraction.

11.6. Soil samples not extracted in the field will be treated as field extracts. {Refer to SOP # 764, & # 767 (methods 5030C & 5035A) for extraction.} Weigh out approximately 50g of sample and add 25mL of BFB-methanol into a pre-weighed 4.0 oz. jar. Both the initial weight and final weight will be stored in LIMS and recorded in the solids control spreadsheet (refer to SOP#143 for weighing procedures). The extracted samples must be iced or refrigerated at 0 - 6°C for 24 hours before analysis can be performed.

11.7. Qualitative Analysis. The qualitative identification of compounds in a sample is based on retention time and comparison of mass spectrum with characteristic ions in a reference spectrum. The reference spectrum must be generated using the same conditions as the calibration. The characteristic ions from the reference mass spectrum are typically the three ions of greatest intensity, or any ions over 30% relative intensity if

less than three such ions occur in the reference spectrum. Positive identification of target compounds is confirmed when the following criteria are met:

11.7.1. The compound elutes within the retention time window set during calibration.

11.7.2. The relative intensities of the characteristic ions agree within 30% from the reference spectrum.

11.7.3. Identification is hampered when individual analytes are not resolved chromatographically and produce mass spectra containing ions contributed by more than one compound. The identification criteria may be met, but each compound spectrum will contain extraneous ions. Analyst judgment is imperative while deciding to assign or eliminate peaks that are target analytes.

11.7.4. For samples containing impurities not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. For guidance regarding Tentatively Identified Compounds (TICs), refer to SGS SOP 500.

11.7.5. Quantitation of calibrated compounds: Once a compound has been identified, quantitation is based on the integrated abundance of the primary characteristic ion. The internal standard used is the one closest in retention to that of the compound. The average RRF or calibration equation from the most recent initial calibration is used for the calculation.

11.7.6. The use of a selected ion monitoring (SIM) technique is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Using the Primary ion for quantitation and the secondary ions for confirmation, set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory's GC/MS software or manually calculated. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak.

12.0. QUALITY CONTROL:

12.1. At the beginning of each 12-hour period for non-SIM methods, the GC\MS tune must be checked to meet BFB specifications see Table 1. The instrument blank or CCV is used to evaluate the tune. The primary method for BFB tuning is to use the software's autotune feature which takes the apex, the scan prior, and the scan following averages them, and performs a background subtraction. The secondary method of tune (manual) evaluation is to pick the apex, the scan prior, or the scan following and have the software evaluate BFB for that scan.

Table 1
BFB Key Ion Abundance Criteria for MS Tuning

Mass	Ion Abundance Criteria
95	base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	50-200% of m/z 95
175	5 to 9% of m/z 174

176	95% to 105% of m/z 174
177	5 to 10% of m/z 176

Note that autotune is routinely used and the manual procedure is used in a minority of circumstances.

When manually performing the BFB tune, follow these steps to subtract the background:

A. Choose the area to be scanned, using the left mouse button.
B. Double click inside the chosen area.
C. Double click outside the peak area.
D. Choose the "subtract" option under the "TUNER" macro box.

12.2. Before processing any samples, the analyst analyzes an instrument blank. The instrument blank can serve as the tune blank and shows that interferences from the analytical system and reagents are under control. If any analytes are detected above the LOQ in the blank and are detected in the associated samples above the LOQ for non-DOD and ½ the LOQ for DOD, then those compounds must be reanalyzed. If the analytes detected in the blank are not detected in the samples above the LOQ, reanalysis is recommended, but not necessary.

12.3. One method blank is run per batch. This shows that interference from glassware and reagents is under control.

12.4. The ICV is prepared using the second source standards.

An acceptable curve, Initial tune, initial blank and continuing calibration verification.

DAILY QC SETUP

Water samples QC setup	Internal Standard / Surrogate	Primary CCV/ Early Gases	MeOH (µL)	Final Volume
IB / MB	Added by system			100mL
CCV / LCSD	Added by system	60µL each mix	160µL	100mL
Matrix Spike	Added by system	60µL each mix		100mL
Matrix spike Dup	Added by system	60µL each mix		100mL

Soil samples QC setup	Internal Standard / Surrogate	Primary CCV/Early Gas	MeOH (mL)	Final Volume
IB / MB	Added by system		2mL	100mL
CCV / LCS	Added by system	60µL each mix	1.8mL	100mL
Matrix Spike	Added by system	60µL each mix	2mL sample	100mL
Matrix Spike Dup	Added by system	60µL each mix	2mL sample	100mL

Soil EDB samples QC setup	Internal Standard / Surrogate	EDB Standard	MeOH (mL)	Final Volume
IB / MB	Added by system		2mL	100mL
CCV / LCS	Added by system	80µL	1.92mL	100mL
Matrix Spike	Added by system	80µL	2mL sample	100mL
Matrix Spike Dup	Added by system	80µL	2mL sample	100mL

Water EDB samples QC setup	Internal Standard / Surrogate	EDB Standard	MeOH (mL)	Final Volume
IB / MB	Added by system			100mL
CCV / LCS	Added by system	80µL	320µL	100mL
Matrix Spike	Added by system	80µL		100mL
Matrix Spike Dup	Added by system	80µL		100mL

12.5. OILS/ORGANIC LIQUID WASTE EXTRACTION:

12.5.1. Weigh 1.0 g (± 0.1g) of the sample, (matrix 3 oil) into a screw capped septum sealed vial. Record the weight to two decimal places. Immediately add 5.0 ml BFB methanol.

12.5.2. If the sample dissolves in methanol it is ready for analysis. Excessive sample handling will result in loss of volatile compounds.

12.5.3. Samples that are not miscible in methanol must be stored at 0-6º C for 24 hours before analysis.

12.5.4. Record anything unusual about the sample or any apparent odors.

12.5.5. For additional dilutions the standard used dilution is 100µg/L/50mL post as 5X.

12.6. The experience of the analyst performing GC\MS analysis is invaluable to the success of the methods. Each day that analyses are performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still usable, the injector is leaking, etc. If any changes are made to the system (e.g. column changed), recalibration of the system should take place. **Note:** For manual integration guidance, please refer to SOP#144.

12.7. The calibration verification is run at the beginning of each 12-hour period in the analytical lot after the tune check. The calibration verification standard (CCV) is used to check the validity of the initial calibration. The percent drift for a CCV and all other analytes (calculated as the percent difference between the true and recovered CCV concentrations) must be less than +/- 20%. If the CCV recovery is low, all data for that analyte will not be reported and will be reanalyzed the following day. In the event of a high bias, every attempt will be made to reanalyze the samples with recoveries for the affected analyte in an additional analytical run. If this is not possible due to hold time expiration or insufficient sample volume the results may be posted as estimated. (Refer to SOP#136 for Data Flagging.)

12.7.1. Internal Standard Retention Time- The retention times for the internal standards in the CCV must be evaluated. If the retention time for any internal standard changes by more than 10 seconds from that of the midpoint standard level of the most recent calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When the corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

12.7.2. **All methods for DOD samples:** In the event of a recovery failure for analytes of interest in continuing or closing CCV samples, the Analyst may immediately (within 1 hr of the failing CCV and before any other samples have acquired) rerun 2 successive CCVs. If both meet QC criteria for all analytes of interest, then the samples already analyzed may be reported and the run sequence may be continued. Samples following the two passing CCVs are also valid.

All CCVs must be reported in LIMS. Refer to SOP#101 for nomenclature (e.g., CCCV, CVC, CVCA, etc.)

12.8. The minimum response factors listed in Table 4 must be met. Internal Standard area counts must not change by more than a factor of two (-50 % to +100%) from the mid-point standard in the most recent initial calibration. Internal standard retention time of the CCV must not change by more than 10 seconds from the mid-point standard in the initial calibration. If the criteria are not met, investigate the problem. Reanalyze the standard or analyze a new calibration curve.

12.9. Laboratory Control Sample (LCS) - Each analyte should be within lab generated control limits. When analytes fall outside of control limits refer to Peer Review SOP#101 for sample comments. If problem persists corrective action should be taken to return the instrument to proper control.

12.10. Matrix spikes / Matrix Spike Duplicate (MS / MSD) - Spike two sample aliquots with the primary solution. The analytes should be within the LCS lab generated control limits for each analyte, and the relative percent difference (RPD) between the spikes must be less than or equal to 20%. MS recoveries are not used to control the acceptability of a batch. A footnote is to be generated discussing any outliers. If there is insufficient sample volume to perform an MS/MSD an LCSD must be analyzed as part of the extraction batch.

12.11. Initial Demonstration of Proficiency. – Once per Analyst. Four replicate standards are analyzed and evaluated for recovery and RPD per EPA 8000C section 8.0.

12.12. Refrigerator/storage blanks are used to monitor for potential cross contamination in temporary and long-term storage units. (Refer to SOP#104 for details.) These storage blanks will be logged into LIMS and treated on a RUSH basis. Those analyzed by 8260D will be evaluated to ½ LOQ. Anything in excess will be rerun to confirm.

12.13. Attachment A outlines QC criteria and corrective actions. Any corrective action required to address a QC outlier or technical problem which not listed in this SOP requires the prior approval of the QA Manager or Technical Director.

12.14. Surrogates – Surrogates are injected into every QC sample and client sample by the Centurion except for BFB in soil samples. BFB is a soil field surrogate added at the time of sampling. See Attachment A for acceptance criteria and corrective action for outliers.

12.14.1. Surrogate associations for all analytes can be found in FS-0118.

13.0. CALCULATIONS, REVIEW AND REPORTING:

13.1. **% Solid** = $\frac{\text{(g. of dry sample + pan) - (pan weight) x 100}}{\text{(g. of wet sample)}}$

Example $\frac{(3.034\ \text{gm}) - (1.567)\ \text{X}\ 100}{(5.034)} = 29.14\%$

13.2. **Moisture Adjustment for Methanol Preserved Soil:**

% Moisture = 100 - % Solid

Final Volume = (Mass of sample * % Moisture) / 100 + MeOH Volume

13.3. **Dilution factor for soils**:

$$[\text{wet weight (g.)}]\ \left[\frac{\%\ \text{solid}}{100}\right] = \text{True weight (g.)}$$

$$\frac{5\ \text{mL methanol}}{\text{True weight}} \times \frac{5\ \text{mL water}}{\text{mL of samples}} = \text{Dilution Factor}$$

Example(5 gm sample) $\left(\frac{92\%\ \text{solids}}{100}\right)$ = 4.6 gm true weight

$$\frac{(5\ \text{mL MEOH})}{(4.6\ \text{gm true weight})} \times \frac{(5\ \text{mL water})}{(0.100\ \text{mL sample})} = 58.69 \text{ dilution factor}$$

13.4. **Dilution factor for oil/sludges**:

$$\frac{5\ \text{mL methanol}}{\text{wt (g) of sample}} \times \frac{5\ \text{mL water}}{0.050\ \text{mL MeOH ext.}} = \text{Dilution factor}$$

Example for a 1 gm sample, injected at 50 µL

$$\frac{5\ \text{mL}}{1.0\ \text{gm}} \times \frac{5\ \text{mL}}{.050\ \text{mL}} = 500 \text{ dilution factor}$$

Note: All oil samples for TCLP must be density corrected.

13.5. **Dilution factor for oil/sludges w / Density correction**.

Example $\frac{2.1360\ \text{gm of sample}}{0.956\ \text{gm/mL}}$ = 2.2343 mL

Example for a 2.1360 gm sample, injected at 50 µL

$$\frac{5\ \text{mL}}{2.2343\ \text{mL}} \times \frac{5\ \text{mL}}{0.050\ \text{mL}} = 223.78 \text{ dilution factor}$$

13.6. **Estimated concentration for noncalibrated compounds**

$$\frac{(\text{Area of Sample from Total Ion Chromatogram}) \times (\text{Conc Internal Standard(ug/mL)}) \times (\text{Dilution Factor})}{(\text{Area of IS from Total Ion Chromatogram}) \times (\text{Response Factor=1.00})} = \text{Conc ug/L}$$

13.7. **Example calculation for compound concentration**

$$RF = \frac{As \times Cis}{Ais \times Cs}$$ RF is generated from calibration

$$Cs = \frac{As \times Cis}{RF \times Ais}$$ Cs is generated daily

Cis = Concentration of internal standard.
Ais = Area of Internal Standard
Cs = Concentration of Analyte
As = Area of Analyte

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP#108 for further instruction.

14.3. All surplus reagent acids shall be neutralized daily using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must always be worn. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base preservative a face shield and apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab regarding current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

There are no method performance measures to report at this time.

17.0. DETECTION LIMIT (DL) STUDY:

DL studies are performed when a significant change in instrument response is observed, when a new instrument is purchased for analysis. The DL is intended to demonstrate the capability of this method as it is implemented at SGS. An update to the DL does not necessitate an update to this document. Further guidance on performing a DL study can be found in SOP 116.

18.0. LIMIT OF DETECTION (LOD):

The LOD is defined per SOP 116; LOD verification shall be performed quarterly according to the schedule set by the QA Office.

19.0. LIMIT OF QUANTITATION (LOQ):

The LOQ is defined per SOP 116; LOQ verification shall be performed quarterly according to the schedule set by the QA Office.

20.0. MAINTENANCE:

Regular maintenance should be documented in the instruments' runlogs on the network. This includes general inspections, refilling standards and the water tank, adjusting the tune, cleaning the ion source, and checking the pump oil and vacuum pressure. All other maintenance, such as replacing parts, should be documented in the instruments' maintenance log book.

21.0. REFERENCES:

SW846 8000C (March 2003) and 8260C (August 2006)

SW846 5030C (July 2002) and 5035A (May 2003)

SW846 8260D Rev 4 (June 2018)

EPA624 (October 1984)

DOD QSM, current version

22.0. ATTACHMENTS:

Attachment A: Corrective Action Table
Attachment B: Recommended Minimum Relative Response Factors
Attachment C: Characteristic Ions for 8260

ATTACHMENT A: CORRECTIVE ACTION TABLE

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration for all analytes (ICAL)	Periodic	Average RF analytes ≤15% Linear/non-linear analytes $r^2 \geq 0.99$ or $r \geq 0.995$	Troubleshoot Repeat calibration.
Second Source Cal. Verification. (ICV)	Once per initial cal.	All analytes within 20 % true value.	Repeat analysis once. If criteria is still out, Troubleshoot, Repeat Calibration
Calibration Verification (CCV)	At the beginning of each 12 Hour period, and the end of the run for DOD samples only	SPCC average RF ≥ section 10.4. RPD for all analytes ≤ 20 **For DOD** samples: Closing CCV +/- 50%	**For non-DOD samples:** 1. Repeat analysis once. 2. If QC criteria not met, then recalibrate and reanalyze. **For DOD samples:** 1. Immediately (within 1 hr and before any other samples have acquired) run two successive CCVs. If both meet QC criteria, then samples already analyzed may be reported and the run may continue.
Method blank	One MB per extraction batch	< LOQ for analytes of interest. For DOD clients, <½ the LOQ.	If associated sample result > 10X the blank contamination, note in LIMS and apply "B" flag to all affected results. If associated sample result < 10X the blank contamination, re-analyze and or re-extract. Note in LIMS and apply "B" flag to all results.
LCS LCSD	One per extraction batch	LCS: Based on laboratory control limits, RPD ≤ 20% LCSD: Used only for sample duplicate RPD. Based on laboratory control limits, RPD ≤ 20%	Repeat analysis once. If target analytes are outside control, high, and not detected in sample, report the data with qualification. If target analytes are outside control, low, repeat analysis.
MS/MSD(Recovery) MS/MSD(RPD)	1 set per batch of 20samples or less	Recovery based on LCS criteria. RPD ±20%	Review / report data. MS recoveries not used for batch control.
Surrogates	Every Sample	Water and soil criteria dependent on in-house control limits.	If analyte is not detected, report. Rerun samples with positive results.
Tune Standard: Prior to Calibration And at the beginning of each 12-hour analytical period.		Method specific see SOP Section 11.1. and table 1	Rerun the tune If condition continues, Retune instrument and recalibrate.
Internal Standard	Every sample post extraction.	From 50% to 200% of midpoint calibration values. One client requires 50 – 130%	Troubleshoot Repeat analysis.
12-hour clock	Each analytical period	Passing Tune, IB, and CCV and the beginning of each analytical run.	Samples outside of the 12-hour clock cannot be reported.

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ATTACHMENT B: RECOMMENDEDED MINIUMUM RELATIVE RESPONSE FACTORS

Volatile Compounds	Minimum Response Factor (RF)	Typical Response Factor (RF)
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501

Volatile Compounds	Minimum Response Factor (RF)	Typical Response Factor (RF)
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

Reference: Table 4 8260C Revision 3 (August 2006)

ATTACHMENT C: Characteristic Ions for 8260

Compound	Primary	Secondary	Tertiary	Quaternary
Fluorobenzene IS	70	96		
dichlorodifluoromethane	85	87		
chloromethane	50	52		
vinyl chloride	62	64		
bromomethane	96	94		
chloroethane	64	66		
trichlorofluoromethane	101	103		
1,1,2-trichloro-1,2,2-trifluoroethane	101	151	153	
1,1-dichloroethene	96	61	63	
Acetone	43	58		
Acrylonitrile	53	52	51	
Methyliodide	142	127		
Carbon disulfide	76	78		
methylene chloride	84	86	49	
methyl-t-butyl ether	73	57	41	43
trans-1,2-dichloroethene	96	61	98	
Vinyl acetate	43	86		
n-hexane	56	86	42	
1,1-dichloroethane	63	65	83	
2-Butanone(MEK)	43	72		
2,2-dichloropropane	77	97		
cis-1,2-dichloroethene	96	61	98	
bromochloromethane	128	49	130	
cyclohexane	56	41	84	
chloroform	83	85		
Dibromofluoromethane <SURR>	111	113		
1,1,1-trichloroethane	97	99	61	
carbon tetrachloride	117	119		
1,1-dichloropropene	75	110	77	
1,2-Dichloroethane-D4 <surr>	65	67	51	
benzene	78	77		
1,2-dichloroethane	62	98		
trichloroethene	95	130	132	
1,2-dichloropropane	63	112		

dibromomethane	93	95	174	
2-chloroethylvinyl ether	63	65	106	
Compound	**Primary**	**Secondary**	**Tertiary**	**Quaternary**
bromodichloromethane	83	85	127	
cis-1,3-dichloropropene	75	110		
4-Methyl-2-pentanone	58	43	100	
1-chloro-3-fluorobenzene<IS>	95	130	132	
Toluene-d8 <surr>	98	100		
toluene	92	91		
trans-1,3-dichloropropene	75	110		
1,1,2-trichloroethane	83	97	85	
tetrachloroethene	166	168	129	
1,3-dichloropropane	76	78		
2-Hexanone	43	58		
dibromochloromethane	129	127		
1,2-dibromoethane	107	109	188	
1-chlorohexane	55	41	91	
chlorobenzene	112	77	114	
1,1,1,2-tetrachloroethane	133	131	119	
ethylbenzene	106	91	77	
P & M -Xylene	106	91		
o-xylene	106	91		
styrene	104	78		
bromoform	173	175		
Isopropylbenzene (Cumene)	105	120		
1,2-Dichlorobenzene-d4 <IS>	152	150		
4-Bromofluorobenzene <Surr>	95	174	176	
trans 1,4-dichloro-2-butene	53	89	39	
bromobenzene	156	77	158	
1,1,2,2-tetrachloroethane	83	131	85	
1,2,3-trichloropropane	110	75	77	
n-propylbenzene	91	120		
2-chlorotoluene	91	126		
4-chlorotoluene	91	126		
1,3,5-trimethylbenzene	105	120		
tert-butylbenzene	119	91		
1,2,4-trimethylbenzene	105	120		
sec-butylbenzene	105	134		
1,3-dichlorobenzene	146	111	148	

4-isopropyltoluene	119	134	91	
1,4-dichlorobenzene	146	111	148	
Compound	**Primary**	**Secondary**	**Tertiary**	**Quaternary**
1,2-dichlorobenzene	146	111	148	
n-butylbenzene	91	134		
1,2-dibromo-3-chloropropane	75	157		
1,2,4-trichlorobenzene	180	182		
hexachlorobutadiene	225	260		
naphthalene	128	102		
1,2,3-trichlorobenzene	180	182		

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SGS	SGS North America, Inc. - Alaska Division Standard Operating Procedure
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S.O.P. Title: Solid-Phase Extraction	**Revision Date:** September 2021
Method No: SW846 3535A	**SOP No**: 784r02
Page: 1 of 12	**Supersedes:** 784r01

Signatures below reflect periodic review of Standard Operating Procedures. If the procedure is found adequate with little or no editing necessary, this page is signed and dated. An Addendum may be issued for minor changes that need to be implemented immediately. If it is determined that major edits are required, a new revision will be released with a new signature page.

Technical Director	Date	Quality Assurance (QA) Manager, QA Staff or their Designee	Date
Stephen C. Ede	9/29/21	Lorraine Rentz	9/29/21

Original cover pages with wet signatures and the digitally signed electronic SOP versions are available in the Quality Assurance Office.

This document will be converted into a PDF file with the QA Manager's, QA Staff's or QA Designee's electronic signature and posted on the network: \\usfs700\ANK_GroupData\Public\DOCUMENT\SOP\~Approved_SOPs~

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Each staff member responsible for this SOP will print & sign this coverpage upon successful review, retaining it as a record in their training folder.

I have reviewed and understand the method reference(s) and this version of the SOP.
I agree to use only this currently approved version of the SOP.

Signature: _____ Printed Name: _____ Date: _____

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Summary of Changes from Previous Revision:

- Addendum from previous revision incorporated
- Section 2.0 edited to include the elution process
- Section 6.2 changed to include 250 mL sample volume
- Significant edits were made to section 9
- Minor grammar and formatting edits throughout

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1.0. OBJECTIVE:

This method is used to isolate organic compounds from aqueous samples using solid-phase extraction media. This method is applicable to the isolation and concentration of water soluble and slightly soluble organics in preparation for a variety of chromatographic procedures.

2.0. SCOPE AND APPLICATION:

This SOP is suitable for subsequent analysis with method PAH by 8270D-SIMS and AK102/103. A measured volume of sample, usually 250 mL or 1 liter, is passed through an analyte-specific cartridge and eluted. The extracts are then concentrated and analyzed with gas chromatography or gas chromatography/mass spectroscopy.

3.0. DEVIATIONS FROM REFERENCE METHOD:

3.1. The reference method states that a 10mL/min flow rate should be used. A 30-50mL/min flow rate is used instead.

3.2. DRO/RRO samples are collected in HCl-preserved bottles; therefore, associated extraction QC are adjusted to pH <2 with 4.0 mL of HCl.

4.0. RESPONSIBILITIES:

4.1 The QA Office maintains a master list of this SOP to ensure review on a timely basis. This system serves as an accounting of SOP distribution and ensures that distributed SOPs are current and complete. The QA Office also maintains a historical file of original cover pages with wet signatures and digitally signed electronic versions of this SOP; including the current revision and any versions archived within the past 5 years.

4.2 The electronic (Word Document) versions of this SOP, both current and any prior versions, are maintained on the computer network in a secure location as a “read only” file.

4.3 It is the responsibility of all personnel to follow this SOP as written, document and gain QA or Technical Director approval for deviations to the SOP and submit needed SOP revisions to the QA Office.

4.4 This SOP is scheduled for review on an annual basis. Any addendum will be incorporated into the SOP and a new revision of the SOP will be distributed by QA. The superseded version will be archived by the QA Office. If there are no addenda to incorporate or revisions to make, the SOP cover page is signed and dated by the Technical Director and QA to document the review, and then the updated cover page and SOP are distributed.

4.5 A PDF version of each SOP (generated in Adobe or scanned) is digitally signed by a member of the QA Office as a security measure. The digitally signed PDF, used online, is considered to be a controlled copy of the SOP and is stored on the network.

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All staff have “read” access to these SOPs. Only QAQC has access to “write” on SOPs. Staff is directed to use the controlled electronic versions of SOPs.

5.0. INTERFERENCES:

5.1. Organochlorine pesticides may dechlorinate, phthalate esters may exchange. The rates of these reactions increase with increasing pH and reaction times.

5.2. Samples containing a significant amount of solids may interfere with the extraction process due to particulates clogging the cartridge.

5.3. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis.

5.4. Bonded-phase silica (e.g. C_{18}) will hydrolyze on prolonged exposure to aqueous samples with pH levels of less than 2 or greater than 9. Hydrolysis will increase at the extremes of this pH range and with longer contact times. Hydrolysis may reduce extraction efficiency or cause baseline irregularities.

6.0. SAMPLE HANDLING:

6.1. Sample Collection – Samples should be unaltered prior to extraction. Aqueous samples are stored at 0-6ºC until extraction.

6.2. Sample Size – 250 mL or 1L amber glass jars with Teflon-lined lids. Small volume DRO/RRO and PAH or samples with a history of high concentrations of the analyte of interest, may be submitted in 250 ml amber glass jars with Teflon-lined lids.

6.3. Sample Preservation – Water samples are stored at 0-6°C until extraction. Prior to extraction water sample collection should include preservation as follows:

Method	Preservative
8270D-SIMS	Cool 0-6°C,
AK102 / AK103	Cool 0-6°C, acidic pH (less than 2) by 1:3 HCl

Methylene chloride extracts are cooled to $\leq$ -10ºC.

6.4. Holding Times – Unpreserved water samples for each analysis must be extracted within 7 days of sample collection. Water samples collected for AK 102 and/or AK103 that have been acid preserved must be extracted within 14 days of sample collection. Extracts must be analyzed within 40 days.

6.5. Criteria for Acceptance/Rejection of Samples – If samples have broken hold time, were not preserved properly, or were not stored at 0-6ºC, notify the Project Manager (PM) before proceeding with extraction.

7.0 APPARATUS:

7.1. Vacuum manifold

7.2. Vacuum pump

7.3. Heavy-walled vacuum tubing

7.4. Vacuum trap/waste container

7.5. Glass cartridge adaptor

7.6. Pyrex glass concentrator tubes with a 1mL (ungraduated) tip

7.7. Concentrator Apparatus: TurboVap by Biotage (compressed nitrogen gas source), or equivalent.

7.8. 40mL collection vial

7.9. Solid-phase extraction cartridge

7.10. 1mL syringes: Hamilton gastight, certificate of conformance stored in CDS, as stated in SOP 104

7.11. Autosampler vials: Virtuoso, amber and clear, with Teflon®-lined crimp tops

7.12. Graduated cylinders class A.

7.13. pH paper: 0 -14 range (with ≤0.5 pH unit increments)

7.14. Disposable glass pipettes: 2, 5, and 10-mL

7.15. Disposable Pasteur pipettes, 7 and 9 inch

7.16. Permanent, solvent-resistant marker (Sharpie Industrial)

7.17. Teflon wash bottles, 500mL, labeled 'methylene chloride', 'D.I water', or 'methanol' accordingly: FILLED DAILY

7.18. Fisher Brand screw top vial, 17X60mm, 2 dram, w/rubber lined cap. (5mL vial)

8.0. REAGENTS:

8.1. Dichloromethane (CH_2Cl_2 or DCM): Fisher Optima (or equivalent) chromatographic grade.

8.2. n-Nonane: Acros 99% or equivalent (PAH analysis keeper)

8.3. De-ionized water (organic free)

8.4. Granular anhydrous sodium sulfate (Na_2SO_4): JT Baker or Fisher (or equivalent), received and opened dates must be recorded on the side of container. Heat at 400°C for ≥3 hours in a shallow tray, record in the Prep working reagents book. Label jars with ID number, date, and initials.

8.5. Hydrochloric acid solution 1:1 HCl: Add 500mL HCl to 500mL organic-free water. Record lot number information in the Prep Working Reagents log book. (Only applicable to DRO/RRO extraction)

8.6. Methanol

9.0. EXTRACTION:

9.1. Assemble the 6 station SPE manifold system.

9.2. Place cartridges on the vacuum manifold.

9.3. With vacuum on, add 10mL of methylene chloride to the cartridge.

9.4. Open and close vacuum valve quickly, drawing a small amount of methylene chloride through the cartridge.

9.5. Let it soak for 1 minute.

9.6. Turn on vacuum and draw through to waste.

9.7. Repeat methylene chloride rinse, let soak for 1 minute, turn on vacuum and draw through waste.

9.8. Remove methylene chloride waste from manifold and dispose of in satellite container.

9.9. Add 10mL of methanol to the cartridge.

9.10. Open and close vacuum valve quickly, drawing a small amount of methanol through the cartridge.

9.11. Let it soak for 1 minute.

9.12. Draw the methanol to the level of the frit.

9.13. Add 10mL of filtered bench water to the cartridge.

9.14. Remove methanol waste from manifold and dispose of in satellite container. Do not replace waste collection vial in the manifold.

NOTE: Do not let the cartridge go completely dry after addition of methanol otherwise repeat starting at step 9.3.

9.15. Pull the batch to be extracted from fridge #7. Samples are sent in 1L or 250mL amber bottles. Scan the sample number into the ELN to build the batch. Ensure that all samples in a work order are together in the batch, unless the number of samples exceeds 20.

9.15.1. **For AK-series methods** (i.e., AK102/103), each batch of twenty samples or less requires a method blank (MB), a laboratory control sample (LCS) and a laboratory control sample duplicate (LCSD). These QC shall be pH adjusted with 4mL 1:1 HCl to extract in a similar pH as the preserved samples. The client may also request a matrix spike (MS) and matrix spike duplicate (MSD); in which case, they are prepared in addition to the LCS and LCSD. **Note: *ADEC requires AK102/103 batches to have both an LCS and an LCSD, even if the client provides sufficient sample for an MS/MSD.*** To prepare the method blank, LCS or LCS Duplicate, use 1000mL de-ionized water (organic free).

9.15.2. **For PAH** each batch of twenty field samples or less requires a method blank (MB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is provided to perform an MS/MSD, then a laboratory control sample (LCSD) must be included.

9.16. All containers will be marked with a solvent resistant permanent marker at the bottom of the meniscus. Then all volumes must be measured to the marked point.

Note: If there is more than ¼ inch of sediment in the bottom of the container, it is considered to be multi-phasic. If sample is multi-phasic, the Production Manager or Technical Director and the Project Manager must be contacted to determine which phase should be analyzed.

9.17. To prepare the method blank, LCS or LCS Duplicate, use an empty 250 mL or 1L amber jar and fill with filtered water from the blue hose. Add the room temperature spike standard to the QC (Quality Control) samples - LCS, LCSD, MS, and MSD - using the designated syringe. Add the room temperature surrogate standard to all of the samples containers, including all QC samples.

9.17.1. Spiking solutions must be introduced into the sample in the original sample container.

9.17.1.1. **SIM PAH:** After adding the spike and surrogate to the sample add 100µl of nonane using a 100 µl syringe.

9.17.2. Sample volumes are used in their entirety unless specified by Project Manager, Technical Director, or Production Manager. If volume used is different from sample volume received for any reason, a comment will be added to that sample in the electronic notebook. Care must be taken to ensure samples are documented at the correct volume.

9.18. Homogenize sample and add the sample to the cartridge. Turn on vacuum and draw sample through cartridge no faster than 20-30 minutes per liter.

9.18.1. In the event that a cartridge becomes clogged, a second cartridge may be used.

9.19. Allow the cartridge to dry under full vacuum for approximately 1 minute, excess drying will cause the lighter analytes such as naphthalene-d10 to be lost in the extraction.

9.20. Place a 40 ml collection vial in the vacuum manifold.

9.21. To remove any analyte from the glass, rinse the sample jar three times with DCM adding each rinse to the cartridge. Also rinse the Teflon lined lid with DCM and add to the cartridge. Top off with DCM until there's approximately 30 mL in cartridge.

9.22. Allow added DCM to soak cartridge for 1-2 minutes, then draw through and collect in vial. Let cartridge go dry but avoid excessive vacuum use as this can cause analytes to be lost.

9.23. Dry the sample extract by pouring it through a DCM pre-rinsed sodium sulfate and glass wool funnel, and collect in a TurboVap tube.

9.24. Rinse the vial three times, pouring each rinse through the funnel. Once all rinses have been added, rinse the funnel and the sides of the concentrator tube.

9.25. Repeat steps 9.14-9.20 for all samples in a batch.

9.26. Check the water level in the TurboVap and check that the temperature is approximately 38°C.

9.27. If the sample fills less than half of the concentrator tube, the nitrogen flow can be started at the maximum pressure. Otherwise, start the nitrogen flow at about 1.5 L/min or 5psi and place the concentrator tubes containing the sample into the TurboVap. The maximum flow must not exceed 3.7L/min or 22psi. The surface of the solvent should be moving gently, not splashing.

9.28. Monitor the samples closely. When the sample level is down to ~2-5mL, thoroughly rinse the sides of the tube with methylene chloride. For samples that are analyzed in methylene chloride, continue concentrating until the sample level is in the tube nipple (~0.8mL). Concentrate to exactly 1mL.

9.28.1. Be sure to keep samples in the order that they are in in the ELN when moving the containers to measure sample volume. Verify the container IDs with those in the ELN – samples should appear in the same order in both places. If something doesn't match up, do a double check to ensure correct information is being recorded (i.e. make sure a sample switch is not occurring).

9.28.2. Samples with a high concentration of oil or other heavy organic compounds will not concentrate to 1mL. When the sample volume is obviously not reducing, or the concentrate is viscous and black,

do not try to concentrate as low as it will go. If the volume is less than 5mL, bring it up to 5.0mL using the calibrated 5.0mL reference vial. Record the final dilution volume (i.e., 5.0mL) in the ELN and on the side of the vial. Extracts that are more than 5mL can be measured with a class A graduated cylinder. DO NOT adjust the volume by (blowing down with nitrogen gun or adding solvent) once the final volume has been recorded. Transfer 1mL of the extract to an auto sampler vial. Record the total final volume on the side of the vial using a black marker and put a dot on the edge of the vial cap top. Contact the project manager and record the reason for the deviation in "comments" in the ELN.

9.28.3. Calibrate 1.0mL, 2.0mL, and 5.0mL reference vials daily by measuring methylene chloride using a gas tight syringe into each vial (i.e., a crimp top auto sampler vial for the 1.0mL and a 21x70 septum lidded vial for the 2.0mL and 5.0mL reference vial).

NOTE: *The following procedures must be used to prevent mixing sample ID during transfer from concentration tube to sample vial. This is the final critical step to insure the validity of the extract.*

9.28.4. To assure samples are not switched, confirm labeling on the concentrator tube and GC vial match. Rinse the slanted tube bottom with a few drops of methylene chloride and concentrate to ~0.8 mL. *No less than 0.5mL*. Transfer the sample from the tube to the labeled vial, (in a vial tray with the vials lined up to correspond with the tube position in the tube rack), using a 1mL glass pipette. Rinse the tube with a few drops of methylene chloride and add to the vial. The now empty concentrator tube is replaced in the tube rack in the same corresponding x-y position. If the sample volume is over 1.0mL by comparison with the reference vial use the nitrogen gun to reduce the volume to 1.0mL. If the sample volume is under 1.0mL, adjust to proper volume by adding methylene chloride using glass transfer pipettes. **Cap the vial and record the final volume**. (The final volume **must** be entered in ELN at the time the vial is capped if it is not the usual volume that the sample is concentrated to.) When all samples have been transferred to vials, check the sample vial numbers with the empty tube numbers to assure that no errors have been made.

9.28.5. In the event that a sample vial needs to be relabeled (for example a poorly printed label or the sample placed in an incorrect vial) make sure that the vial's barcode is unreadable, and the new sample ID is clearly written. This will require immediate peer review and a note placed in the ELN.

NOTE: *Use of the nitrogen gun to reduce the sample volume in the vial creates an endothermic reaction that causes water condensation on & in the vial. Therefore, it is to be used sparingly.*

9.28.6. PAH extracts: must never go below 0.5mL because lighter compounds will be lost.

9.28.7. Never under any circumstance allow a sample to concentrate to dryness.

9.29. Deliver extracts to Fridge #33. For Rush analysis, deliver extracts to the analyst.

9.29.1. For AK102/103, DRO/RRO, deliver in clear glass auto sampler 1mL vials

9.29.2. For PAH, use amber glass auto sampler 1 mL vials

9.30. Measure the initial sample volume (section 9.12.) by filling the sample bottle to the meniscus mark with tap water. Pour the water into a graduated cylinder and record this volume as the initial sample volume in the ELN.

9.31. Make sure that any & all sample oddities & deviations are documented and all blank entries in the ELN are filled, including the TurboVap ID and temperature, and the lot numbers of all solvents, standards and materials used. Post the data into LIMS. Double check entries. Put together the batch folder with the peer review and ELN pages, sign with initials, and place in Batches for Review folder.

10.0. CALIBRATION:

N/A

11.0. ANALYSIS:

N/A

12.0. QUALITY CONTROL:

12.1. For each batch of up to twenty extracted: if there is not sufficient volume for an MS/MSD, an LCS duplicate sample is extracted. **All batches for AK102/103 require both LCS and LCSD.** These samples are used to evaluate extraction efficiency and ensure that the extraction process is not contributing to sample results. Sample spikes and sample spike duplicates (MS/MSD set) may be requested by the client to evaluate site specific effects on the extraction/analysis process.

12.2. All samples, method blanks, LCS/LCSD and MS/MSD are fortified with the surrogate solutions.

12.3. Samples can be added to an active batch (samples still in the extraction process), with less than 20 samples, to the full 20 sample batch size. Examples would be, RUSH samples or samples broken or spilled.

12.4. Any corrective action(s) needed to address a QC outlier or other technical challenges that are not listed in this SOP require the prior approval of the QA Office or Technical Department.

13.0. CALCULATIONS, REVIEW AND REPORTING:

Refer to the Peer Review SOP 101.

14.0. HEALTH AND SAFETY:

14.1. Samples shall be returned to the approved storage location until such time as it is determined that no further analysis will be required.

14.2. Once it has been established that no more analysis of a sample will be required, acid and alkaline preserved samples may be neutralized using the Elementary Neutralization Hood located in GC Prep, unless it is considered to be hazardous (i.e. mercury waste, cyanide waste). Refer to the current version of SOP 108 for further instruction.

14.3. All surplus reagent acids shall be neutralized on a daily basis using the Elementary Neutralization Hood located in GC Prep.

14.4. Small volumes of surplus volatile reagents may be allowed to evaporate in a hood at the end of the analytical session.

14.5. Proper Personal Protective Equipment (PPE) must be worn at all times. Proper PPE when handling samples include a lab coat, gloves and safety glasses. In addition, when handling concentrated acid or base

preservative, a face shield, arm sleeves, and an apron must be worn.

15.0. POLLUTION PREVENTION:

SGS is committed to evaluate all areas of the lab with regard to current and potential pollution prevention. Pollution prevention is described as any technique that reduces or eliminates waste at the point of generation. Further discussion on pollution prevention programs in the laboratory can be found in *SGS Pollution Prevention Plan* (Form F053).

16.0. METHOD PERFORMANCE:

N/A

17.0. DETECTION LIMIT (DL) STUDY:

N/A

18.0. LIMIT OF DETECTION (LOD):

N/A

19.0. LIMIT OF QUANTITATION (LOQ):

N/A

20.0. REFERENCES:

SW846, Revision 3, December 1996
Method 3535A, Revision 1, February 2007
Method 3500C, Revision 3, February 2007

21.0. ATTACHMENTS:

ATTACHMENT A: Spiking Requirements Table

ATTACHMENT A: Spiking Requirements Table

Method	Surrogate	Volume	Spike	Volume	QC ID
8270D-PAH SIMS (also for Low Volume)	PAH (0.5µg/mL)	1.0mL			MB
	PAH (0.5µg/mL)	1.0mL	PAH Spike (0.5µg/mL)	1.0mL	LCS
	PAH (0.5µg/mL)	1.0mL	PAH Spike (0.5µg/mL)	1.0mL	MS/MSD
	PAH (0.5µg/mL)	1.0mL			PS
	All samples and QC spiked with 100µl nonane				

Method	Surrogate	Volume	Spike	Volume	QC ID
AK102/103 DRO/RRO (also for Low Volume)	**5-Alpha & DTC (100µg/mL)**	1.0mL			MB
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	LCS/LCSD
	5-Alpha & DTC (100µg/mL)	1.0mL	LSB / LSR (5000µg/mL)	1.0mL	MS/MSD
	5-Alpha & DTC (100µg/mL)	1.0mL			PS

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved, total, and total recoverable metals content in environmental samples by Inductively Coupled Plasma/Mass Spectroscopy (ICPMS) using EPA SW-846 Method 6020, 6020A, 6020B and EPA Method 200.8.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes that can be determined by this SOP and the associated LOQ is provided in Table 1, Appendix A.

2.0 SUMMARY OF METHOD

The ICP-MS uses inductively coupled plasma as the source of metal cations for the quadrupole mass spectrometer detector. This results in an instrument capable of detecting a wide variety of metals. This method measures ions produced by a radio frequency inductively coupled plasma. Analytes originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are introduced in the plasma gas, by means of an interface, into a mass spectrometer. The ions produced are sorted according to their mass-to-charge ratios and are quantified with a channel electron multiplier.

The detection limits for ICP-MS are comparable to those of graphite furnace atomic absorption spectroscopy (GFAAS). Although ICP-MS data is acquired sequentially, ICP-MS has the capability of determining all the metals of interest in the time it would take GFAAS methods to determine one analyte. Internal standards are used to compensate for changes in instrumental sensitivity and mass transport phenomena.

3.0 INTERFERENCES

Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. They are:

- **Isobaric elemental interferences** - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process must be included with the report and data. It should be noted that such corrections would only be as accurate as the accuracy of the isotope ratio used in the

elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

- **Abundance sensitivity** - A property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences must be recognized and the spectrometer resolution adjusted to minimize them.

- **Isobaric polyatomic ion interferences** - Caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data must be established at the time of the analytical sequence as the polyatomic ion interferences are highly dependent on the sample matrix and chosen instrument conditions. In particular, the common 82Kr interference that affects the determination of both arsenic and selenium can be greatly reduced with the use of high purity krypton free argon.

- **Physical interferences** - Are associated with the physical processes that govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Ideally internal standards ideally should have similar analytical behavior to the elements being determined.

- **Memory interferences** - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical sequence and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limits must be noted. Memory interferences may also be assessed within an analytical sequence by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop

consecutively, the analyst must be alerted to the possibility of a memory effect and should examine the analyte concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample should be reanalyzed after a long rinse period.

- **Instrument Interferences-** The primary interferences in ICPMS are molecular ions such as oxides, doubly charged ions, and isobaric interferences. For the few target isotopes that isobaric interferences affect (Se), a correction is entered into the parameter set to compensate for each one. The interference check SRM solution, IC6020 is used to assess the effects of a variety of molecular interferences. This solution should pass for all components to be reported. In addition, the data for samples with requested target analytes above the MDL should be examined for evidence of interfering molecular ions or doubly charged ions. A list of the peaks that need to be monitored follows here.

Target Analyte	Common Interference	Monitor	Correction Based On
$^{111}Cd^{+}$	$^{95}Mo^{16}O^{+}$	$^{95}Mo^{+}$	$^{95}Mo^{+}$
114Cd	95Mo16o+, 50Sn	118Sn	NA
$^{65}Cu^{+}$	$^{25}Mg^{40}Ar^{+}$	$^{25}Mg^{+}$	NA
$^{65}Cu^{+}$	$^{32}S^{33}S^{+}$	$^{32}S^{+}$	NA
$^{60}Ni^{+}$	$^{44}Ca^{16}O^{+}$	$^{44}Ca^{+}$	**NA**
$^{75}As^{+}$	$^{40}Ar^{35}Cl^{+}$	$^{77}Se^{+}$	Method Blank
$^{40}Ar^{37}Cl^{+}$	$^{77}Se^{+}$	$^{82}Se^{+}$	Method Blank
$^{77,78,82}Se^{+}$	$^{82}Kr^{+}$, $^{1}H^{81}Br^{+}$	$^{83}Kr^{+}$, $^{79}Br^{+}$	$^{83}Kr^{+}$
$^{98}Mo^{+}$	$^{98}Ru^{+}$	$^{101}Ru^{+}$	$^{101}Ru^{+}$
$^{115}In^{+}$	$^{115}Sn^{+}$	$^{118}Sn^{+}$	$^{118}Sn^{+}$
$^{66}Zn^{+}$	$^{50}Ti^{+16}O^{+}$	$^{50}Ti^{+}$	$^{50}Ti^{+}$

NA - Not Applicable

NOTE: 40 Ar 37 Cl + is not the target analyte monitored; rather any ion or combination thereof possessing a mass of 77amu is monitored.

- **Representative Correction Equations**

  As75 -3.127*[Arcl77-(.873*Se82)]

  Ba137 -0.000903*La139 - 0.002825*Ce140

  Cd111 -1.073*[Mo0108-(.712*Pb106)]

  Cd114 -0.027250*Sn118

  Fe54 -0.028226*Cr52

In115 -0.014032*Sn118

Mo98 -0.110588*Ru101

Pb208 +1*Pb206+1*Pb207

Sb123 -0.127189*Te125

Se82, 77, 78 -0.978*Kr83

Sn119 -0.013447*Te125

Sn120 -0.013447*Te125

V51 -0.0108*Cl37

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- Tuning Solution – A solution that is used to determine acceptable instrument performance prior to calibration and sample analyses.
- Linear Dynamic Range (LDR) - The range over which the instrument response to analyte concentration is linear.
- Serial Dilution (SD) – When the concentration of an element of interest is at least x25 the MDL, the sample must be diluted by a minimum of x5 dilution. One serial dilution must be prepared and analyzed per batch of samples, except for drinking water samples by EPA Method 200.8.
- Post Spike (PS) – A second aliquot of a field sample that is spiked with known concentrations of target analytes and analyzed to assess recovery of the spike. A post spike must be analyzed when the MS and/or MSD fail due to a suspected matrix effect. One sample is spiked after digestion and analyzed per batch. Post spikes are not performed for drinking water samples EPA Method 200.8.
- Interference Check Sample (ICS) – A series of two solutions (ICSA & ICSAB) to verify that inter-element interferences are correctly compensated. The ICSA and ICSAB provide an adequate on-going test of inter-element correction (IEC) factors.
    - ICSA – A solution containing only the interfering analytes at high concentrations.
    - ICSAB – A solution containing interferents plus other method analytes at the level of concern, which corresponds to the project specific action limits.

- Lower Limit of Quantitation (LLOQ) - A term associated with analysis per the requirements of Method 6010D. The lowest point of quantitation, which in most cases is the lowest concentration in the calibration curve.

5.0 HEALTH AND SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 .SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MTJL-0064 Sample Shipping, for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The laboratory does not perform sample collection or field measurements for this test method. Samples should be collected in accordance with a sampling plan and sampling procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Aqueous (Total Recoverable)	250mL	GW: 45mL DW: 25mL WW: 25mL	Thermal: Chemical: Acidified with (1:1) nitric acid: water to pH <2	Collection to Prep and Analysis: 6 Months

Aqueous (Dissolved)	250mL	GW: 45mL DW: 25mL WW: 25mL	Thermal: Chemical: Acidify the filtrate with (1:1) nitric acid: water immediately following filtration to pH <2	Collection to Prep and Analysis: 6 Months
Solid	4oz	0.5g	Thermal:NA Chemical:NA	Collection to Prep and Analysis: 6 Months

[1] *Amount of sample required for each discrete test.*

NOTE: For the determination of dissolved elements, the sample must be filtered through a 0.45-um pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. Use a portion of the sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with nitric acid immediately following filtration to pH<2. The sample is now ready for digestion. The hold time begins from the time of collection and ends with analysis within 180 days of collection.

For the determination of total recoverable elements in aqueous samples, samples are not filtered, but acidified with (1+1) nitric acid to pH < 2 (approximately, 3mL of (1+1) acid per liter of sample is sufficient for most ambient and drinking water samples). Preservation may be done at the time of collection; however, to avoid the hazards of strong acids in the field, transport restrictions, and possible contamination, it is recommended that the sample be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample should be mixed, held for 24 hours, and then verified to be pH < 2 just prior to withdrawing an aliquot for processing or "direct analysis". If for some reason, such as high alkalinity, the sample pH is verified to be > 2, a T2 qualifier is applied in Prep Data. If the sample is reactive with Nitric acid, the volume of sample is reduced to 5mL for digestion. The holding times for total recoverable elements begins from the time of collection and ends with analysis within 180 days of collection.

Thermal preservation is checked and recorded on receipt in accordance with laboratory SOP ENV-SOP-MTJL-0060, Sample Receiving. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 0-6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 0-6°C until sample analysis.

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

7.1 Equipment

- The inductively coupled plasma mass spectrometer used is an Agilent 7700x ICP-MS, or Agilent 7900 ICP-MS.
- Each ICP-MS is coupled to a Cetac ASX-500 series Autosampler or Agilent SPS 4 series Autosampler. A modified pumped rinse station was added to maintain fresh rinse solution at a constant level that ensures a clean Autosampler probe.

7.2 Supplies

- The pump tubing used is as follows:

Agilent Tubing:

Sample white-white1.02mm ID

Internal standard orange-blue 0.25mm ID

Drain yellow-blue 1.52mm ID

- Argon gas supply: High purity grade (99.99%).
- Adjustable pipettes (Eppendorf or equivalent).
- Class A volumetric flasks
- Ultra-high purity Helium gas supply for Agilent collision cell
- Samples and Standards is aspirated from polypropylene centrifuge tubes arranged in the Autosampler.

8.0 REAGENTS & STANDARDS

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected unless otherwise noted.

8.1 Reagents

- Hydrochloric acid, concentrated (sp. gr. 1.19) – HCl
- Nitric acid, concentrated (sp. gr. 1.41) - HNO_3
- Laboratory Reagent water

8.2 Standards

Calibration	Stock Std Cat # or equivalent
1ppb Standard #1	VHG-PACE-CAL-A-500 VHG-PACE-CAL-B-500
2ppb Standard #2	
10ppb Standard #3	
50ppb Standard #4	
100ppb Standard #5	
200ppb Standard #6	
1ppm Standard #7	

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Calibration	Stock Std Cat # or equivalent
ICV	PlasmaCal: S180223039
CCV	VHG-PACE-CAL-A-500 VHG-PACE-CAL-B-500
ICVLL	Inorganic Ventures: ESC-12
CCVLL	Inorganic Ventures: ESC-12
ICSA	Inorganic Ventures: 6020ICS-0A
ICSAB	Inorganic Ventures: 6020ICS-0A Inorganic Ventures: ESC-1 Inorganic Ventures: ESC-2
Agilent Tune	Agilent Tech: #5188-6564
EPA Tune	EE: HP2047-A-250
Calibration	Stock Std Cat # or equivalent
Internal Standard	VGH Labs: VHG-PTEH-500
	VHG Labs: VHG-PBIN-100
	VHG Labs: VHG-PSCN-100
	VHG Labs: VHG-PRHH-100
	VHG Labs: VHG-PGENF-100
2.5% Blank and Rinse	Fisher:UN2031/ VWR: UN1789

8.3 Formulations

Calibration	Working Standard Preparation
1ppb Standard #1	0.5mL of the 100ppb Cal Std #5 poured up to 50mL with 2.5% Rinse
2ppb Standard #2	1mL of the 100ppb Cal Std #5 poured up to 50mL with 2.5% Rinse

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Calibration	Working Standard Preparation
10ppb Standard #3	50uL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
50ppb Standard #4	0.25mL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
100ppb Standard #5	0.5mL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
200ppb Standard #6	1mL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
1ppm Standard #7	5mL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
ICV	0.5ml of ICV Std poured up to 50mL with 2.5% Rinse
CCV	0.5mL of Cal Std A and Cal Std B poured up to 50mL with 2.5% Rinse
ICVLL	0.5mL of LL Std A and LL Std B poured up to 50mL with 2.5% Rinse
CCVLL	0.5mL of LL Std A and LL Std B poured up to 50mL with 2.5% Rinse
ICSA	5mL of ICSA Std poured up to 50mL with 2.5% Rinse
ICSAB	5mL of ICSA Std, 0.5mL of ICSAB Std A and 0.5mL of ICSAB Std B poured up to 50mL with 2.5% Rinse
Agilent Tune	0.05mL of Agilent Tune Std poured up to 50mL with 2.5% Rinse
EPA Tune	5mL of EPA Tune Std poured up to 50mL with 2.5% Rinse

Calibration	Stock Std Cat # or equivalent	Working Standard Preparation
Internal Standard	VGH Labs: VHG-PTEH-500	2.5mL of Tellurium poured up to 1000mL with 2.5% Rinse
	VHG Labs: VHG-PBIN-100	0.5 mL of Bismuth poured up to 1000mL with 2.5% Rinse
	VHG Labs: VHG-PSCN-100	0.5 mL of Scandium poured up to 1000mL with 2.5% Rinse

Calibration	Working Standard Preparation	
	VHG Labs: VHG-PRHH-100	500uL of Rhodium poured up to 1000mL with 2.5% Rinse
	VHG Labs: VHG-PGENF-100	500uL of Germanium poured up to 1000mL with 2.5% Rinse

Calibration	Stock Std Cat # or equivalent	Working Standard Preparation
2.5% Blank and Rinse	Fisher:UN2031/ VWR: UN1789	Fill rinse container with 6L of DI water. Add 120mL of HNO3 and 30mL of HCL

- Mixed Calibration Standard Solutions are used to make the following calibration solutions. All standards are prepared in Class A volumetric flasks using adjustable pipettes. The final acid concentration is matrix matched to digested field sample concentrations. See table above for the preparation of this standard.
- Initial Calibration Verification (ICV) – The ICV is an analytical standard solution from a second source different from the calibration and CCV standards. The ICV is prepared at a mid-range concentration within the linear working range of the instrument. The ICV must have the same acid matrix as the Calibration Standards. See table above for the preparation of this standard.
- Continuing Calibration Verification (CCV) – The CCV is the mid-range calibration standard prepared from the same source as the initial calibration curve. The CCV is used to verify the regression of the initial calibration of the instrument and must be repeated following every ten samples and at the conclusion of the sequence. See table above for the preparation of this standard.
- Low Level Initial/Continuing Calibration Verification (ICVLL/CCVLL) – The ICVLL/CCVLL is prepared at a low concentration within the linear working range of the instrument and defines the lowest level of quantitation/reporting. The ICVLL/CCVLL must have the same acid matrix as the calibration standards. See table above for the preparation of this standard.
- For the preparation of Laboratory Control Samples (LCSs) and Matrix Spikes (MSs) see the applicable sample preparation SOPs.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

All support equipment used must be calibrated or verified prior to use according to SOP-ENV-SOP-MTJL-0374 Support Equipment current revision or replacement.

9.1.2 Instrument Set Up

9.1.2.1 Routine Instrument Operating Conditions

All instruments maintained per manufacturer's recommendations

9.2 Calibration

9.2.1 Calibration Frequency

To perform quantitative measurements, an initial calibration must be established before the analysis of samples. An initial calibration is an evaluation of the relationship between response of the instrument (or process) and the concentration of the target analytes.
All samples, where applicable, must be associated with an acceptable initial calibration. In general, if an initial calibration is not acceptable, corrective actions must be performed and all associated samples re-analyzed. If the sample re-analysis is not possible, data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers.

9.2.2 Calibration Levels

STD 1 – 0.2ppb Cd

STD 2 – 0.5ppb

STD 3 – 1ppb

STD 4 – 2ppb

STD 5 – 10ppb

STD 6 – 50ppb

STD 7 – 100ppb

STD 8 – 200ppb

STD 9 – 500ppb Cu

9.2.3 Calibration Sequence

CALBLK, STD1, STD2, STD3, STD4, STD5, STD6, STD7, STD8, STD9, ICV, ICB, CCV, CCB, RLV, LLICV, ICSA, ICSAB

9.2.4 Calibration Evaluation

9.2.4.1 Curve Fit

Run a calibration curve on a daily basis that employs a minimum of a calibration blank and six standards where each element is present in at least three of the standard levels. The concentration of the lowest standard must be at or below the reporting limit.

- EPA 200.8 & 6020/6020B - The correlation coefficient for the each analyte in the calibration curve lines must be 0.995 or better for EPA Methods 200.8 and 6020.

- NOTE: For Method 6010D - If a multipoint calibration is used the low standard must be at or below the LLOQ.

- SW-846 6020A – The correlation coefficient for each analyte in the calibration curve must be ≥ 0.998 for EPA 6020A

- NOTE: For Marathon sample analysis, simple linear curve fitting as identified in the instrument regression choices will be utilized when possible.

9.2.4.2 Relative Error

For calibrations evaluated using correlation coefficient, the lab evaluates relative error by measurement of the Relative Standard Error (RSE). This calculation shall be performed for 2 calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

9.2.4.3 Initial Calibration Verification

Verify the initial instrument standardization by analyzing appropriate check standards following calibration.

- **EPA Methods 6020 & 200.8** - The mid-level ICV standard recovery results must be ±10% of the true value for EPA methods 6020 and 200.8.

- **EPA Method 6020A** - The mid-level ICV standard recovery results must be ±10% of the true value for EPA methods 6020A. The low-level ICV standard recovery results should be within 70-130%

.

- **EPA Method 6020B** Low-level Readback or Verification - For a multi-point calibration, the low-level standard should quantitate to within 80-120% of the true value. For a single point calibration, a standard from the same source as the calibration standard and at the LLOQ is analyzed and should recover within 80-120% of the true value.

9.2.4.4 Continuing Calibration Verification

Verify the on-going instrument standardization by analyzing appropriate check standards during the sequence. Instrument calibration acceptability is demonstrated after every 10 samples and at the end of the analytical run using the CCV that must meet the following criteria:

- **For SW-846 Method 6020/6020B and EPA Method 200.8** – Continuing Calibration Verification (CCV) recovery must be within +10% of the target concentration. Instrument calibration verification is achieved by analyzing both a CCV standard and a CCB (inst. blank).

- **For SW-846 Method 6020A** - Instrument calibration verification for method 6020A is achieved by analyzing both a mid-level CCV standard, a low-level CCV standard, and a CCB (instrument blank). The mid-level Continuing Calibration Verification (CCV) recovery must be within ±10% of the target analyte concentration. The low-level Continuing Calibration Verification (LLCCV) recovery should be within 70-130%.

9.3 Sample Preparation

9.3.1 Homogenization & Subsampling

See ENV-SOP-MTJL-0030, Homogenization and Subsampling for further information.

9.3.2 Sample Preparation

- Digestion. All wastewater and drinking water samples and spikes that require a digestion follow ENV-SOP-MTJL-0208, *Digestion of Metals and Trace Elements in Drinking Water and Wastes for ICP-AES and ICPMS by Microwave and Hot Block Digestion*. All RCRA samples must be digested using one of the following SOPs: ENV-SOP-MTJL-0217 for 3050B/3051 and ENV-SOP-MTJL-0219 for 3005A, 3010A and 3015.

- Internal Standards. The internal standard solution (100ppb each Sc, Te, Ge, Rh, and Bi) and sample solution are mixed in a mixing block prior to nebulization. The ratio of sample to internal standard is 4:1.

9.4 Analysis

9.4.1 Sample Analysis

Instructions vary slightly depending on the specific instrument being used for analysis. Consult the instrument operating guides for each instrument for the exact directions for each instrument.

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide
- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Allow at least 1 hour for break-in period.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.
- Nebulizer waste and rinse waste flow into one waste container.

- Ar/O pressure - The argon supply pressure should be set at about 100psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Liquid Ar level - Liquid argon is maintained in a bulk supply tank. This tank is refilled on a regular basis before the argon supply is exhausted and bleeding of the argon lines is not necessary.
- Torch alignment – Torch alignment is done automatically during performance check.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure:
    - Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing all analytes needed.
    - The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly damaged tip.
- Chiller temperature, pressure, and water level - The temperature should be regulated at 15 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Nebulizer - Check the nebulizer to be sure it is clean, the capillary is not clogged and it produces a smooth mist.
- Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:
    - Torch Ignition - Click on the *Plasma* icon to open the *Instrument* window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
    - Warm-up: Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
    - Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution the Start-up configuration can be started. The instrument will then go through Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and Performance report.
- **Running Samples**: After Warm-Up is complete, perform the following steps to collect a dataset.
    - Tune - Open Tune tab. Switch ISIS valve to tune. Monitor signal to ensure sample is reaching the detector. When the instrument achieves proper counts, stop the monitor, and select the “semi auto tune: tab (The instrument performs five replicates of tuning solution.). The measured mass must be as follows:

Agilent:

Agilent monitored masses (No Gas)	Requirement

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7	CPS RSD<5%
89	CPS RSD<5%
205	CPS RSD<5%
156/140	CPS Ratio<1.5%
70/140	CPS Ratio<3.0%
Agilent monitored masses (He)	**Requirement**
59	CPS RSD<5%
89	CPS RSD<5%
51/59	CPS Ratio<3%
75	CPS ~5 or less
78	CPS ~5 or less

- Create New Dataset – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#).
- Enter Sample ID's: Open the routine analysis workspace previously saved. Enter the sample IDs in the sample template previously created. Enter any necessary dilution factors in the column provided along with both beginning and ending volumes. Place all calibration standards, calibration check solutions, samples, and QC in the Autosampler according to the template.
- For initial and daily operation, calibrate the instrument according to the instrument manufacturer's recommended procedures using mixed calibration standard solutions and the calibration blank. The concentration of the lowest standard must be at or below the reporting limit. A peristaltic pump must be used to introduce all solutions, samples, and internal standard to the nebulizer (Agilent 7900 uses ISIS-3 pump). To allow adequate time for equilibrium to be reached in the plasma, aspirate all solutions for at least 30 seconds after the solution reaches the plasma before accumulating data. Use the average value from three replicate integration periods of the signal to be correlated to the analyte concentration. Flush the system with the rinse blank for a minimum of 60 seconds between each standard. The calibration line is generated by first order linear regression from a calibration blank and six calibration standards where each element is present in at least three concentration levels. Calibration acceptance criteria are described in (Appendix B). For ICV/CCV/CCB/ICB/ICS requirements and frequency, see Appendix B.
- Begin Analyzing Samples: For Agilent instruments, in the mass hunter software select “sample list” and then choose the “unknown samples” tab. Once here, select “Start editing mode” and begin to add samples + instrument QC in the order desired for analysis. Assign appropriate vial

location numbers as well as dilution factors to each sample in the table. Once completed, select "End editing mode" and analysis will begin.

- The instrument performs sample analysis by executing 20 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.
- Monitor Initial QC. Check the calibration blank Summary Report for signs of contamination and the rest of the calibration and initial QC Summary Reports for signs of problems.
  - If analysis will not be complete until after hours, set to shutoff automatically when analysis is complete (Enable automatic shutoff in *Instrument* window. This can be done while the instrument is analyzing samples).
- Instrument Shutdown - When batch is complete, MassHunter will auto export all data to the dataset and shut off plasma if selected. Otherwise, select turn off plasma to stop a run immediately. Turn off the computer monitor if it will not be used further that day.

9.4.2 Example Analytical Sequence:

Cal Blank, STD 1, STD 2, STD 3, STD 4, STD 5, STD 6, STD 7, STD 8, ICV, ICB, CCV, CCB, RLV (client specific), LLICV, ICSA, ICSAB, Samples, CCV, CCB, CCVLL (client specific).

STATE NOTE: For work performed in support of the NC Department of Environment and Natural Resources (15A NCAC 02H .0805 (a) (7) (I)). For target analytes quantitated by ICP or ICPMS, a series of at least three standards must be analyzed along with each group of samples. The concentrations of these standards must bracket the concentration of the analytes in the field samples analyzed. Samples with target analyte concentrations above the highest level of calibration must be diluted to quantitate analytes within the calibration range. The use of the dynamic linear range studies to validate analyte/instrument calibration linearity must not be used for NC sample analysis.

10.0 Data Analysis & Calculations

10.1 Qualitative Identification

The ICP-MS analyzer software MassHunter Data Analysis is used to quantify metals

10.2 Tentatively Identified Compounds (TICs)

Not applicable to this SOP.

10.3 Manual Integration

Not applicable to this SOP.

11.0 Quality Control & Method Performance

11.1 Quality Control

Prepare the following QC samples with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action(s).

QC Check	Acronym	Frequency
Method Blank	MB	1 per batch of 20 or fewer samples.
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples.
LCS Duplicate	LCSD	As Required.
Matrix Spike	MS	1 per batch of 20 or fewer samples.
Matrix Spike Duplicate	MSD	1 per batch of 20 or fewer samples.
Sample Duplicate	SD	1 per batch of 20 or fewer samples
Post Digestion Spike	PDS	1 per batch of 20 or fewer samples.
Reporting Limit Verification	RLV	As required
Internal Standards	ISTD	Introduce to every field samples, standards, and QC samples.

11.2 Instrument QC

Perform the following checks to verify instrument performance. Refer to Appendix B for acceptance criteria and required corrective action.

Instrument Check	Acronym	Frequency
Tune (MS Only)		Prior to ICAL
Initial Calibration Verification	ICV	After ICAL analysis
Initial Calibration Blank	ICB	Immediately after the ICV
Continuing Calibration Verification	CCV	Every 10 samples and end of analysis sequence
Continuing Calibration Blank	CCB	Immediately after the CCV
Low-Level Calibration Check	LLICV	Daily
Linear Dynamic Range	LDR	Initial set-up and checked every 6 months

11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MTJL-0038, Data Review for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 MODIFICATIONS

The procedures in this SOP have been modified from the reference test method as follows:

Modifications:
Not all analytes listed as analyzed in this procedure are approved within the published EPA methods; however sufficient information is present within the laboratory to demonstrate acceptable performance of these analytes using this procedure. Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed. If a modification to the procedure is required to analyze a sample beyond those specified in the procedure as state notes, it must be properly documented. Any modification to the method is listed in the test report's case narrative, and the method is reported as modified.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Managemen*t and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: Routine Analyte List and LOQ

- Appendix B: QC Summary & Corrective Action Table
- Appendix C: DoD Requirements

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manua*l, current version.
- Agilent ICP/MS User's Guides.
- *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020, Revision 0, September 1994.
- *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020A, Revision 1, February 2007.
- *Inductively Coupled Plasma – Mass Spectrometry*, SW-846 Method 6020B, Revision 2, July 2014.
- Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, EPA Method 200.8, Supplement I, Revision 5.4, May 1994.
- Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, EPA Method 200.8, Revision 5.5, October 1999, EPA-821-R-99-017.
- *Chapter 3: Inorganic Analytes*, SW-846, Revision 4, February 2007.
- Identification of Test Procedures, 40 CFR Part 136.3.
- Inorganic Chemical Sampling and Analytical Requirements, 40 CFR Part 141.23.
- Department of Defense (DOD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Jeremy Gupton	Manager - Metals	7/18/2022

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section(s)	Description of Change
3.0, 4.0, 8.3	Updated in response to TDEC audit, definitions for ICSA and ICSAB added, equation for Cd114 corrected, the Internal Standard Stock standards (TI,

	Bi, Sc, Rh, Ge); (b) The ICV and CCV Stock standards; (c) The 2.5% Blank and Rinse solution is actually made by adding 120 ml of Nitric Acid and 30 ml of Hydrochloric Acid to 6 liters of DI water; (d) 0.05 ml of the Agilent Instrument Tuning solution 5188-6564 (ESC # 45910) is added to 50 ml of DI water with the 2.5% rinse solution, not 0.5 ml as listed.
All	Updated to corporate format

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ESC Lab Services SOP#340390 V0	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	1/18/2000
ESC Lab Services SOP#340390 V1	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	8/22/2000
ESC Lab Services SOP#340390 V2	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	9/10/2001
ESC Lab Services SOP#340390 V3	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	12/6/2001
ESC Lab Services SOP#340390 V4	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	4/11/2003
ESC Lab Services SOP#340390 V5	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	2/9/2004
ESC Lab Services SOP#340390 V6	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	4/16/2004
ESC Lab Services SOP#340390 V7	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	8/3/2004
ESC Lab Services SOP#340390 V8	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	10/15/2005
ESC Lab Services SOP#340390 V9	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	11/15/2006

ESC Lab Services SOP#340390 V10	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	10/29/2008
ESC Lab Services SOP#340390 V11	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	2/11/2009
ESC Lab Services SOP#340390 V12	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	7/13/2011
ESC Lab Services SOP#340390 V13	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	5/10/2012
ESC Lab Services SOP#340390 V14	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	11/11/2014
ESC Lab Services SOP#340390 V15	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	2/4/2016
ESC Lab Services SOP#340390 V16	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	10/27/2016
ESC Lab Services SOP#340390 V17	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	4/10/2017
ESC Lab Services SOP#340390 V18	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	11/30/2017
ESC Lab Services SOP#340390 V19	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	7/27/2018
ENV-SOP-MTJL-0221 V00	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	1/26/2019
ENV-SOP-MTJL-0221 V01	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	3/10/2019

ENV-SOP-MTJL-0221 V02	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	6/19/2019
ENV-SOP-MTJL-0221 V03	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	6/20/2019
ENV-SOP-MTJL-0221 V05	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	7/9/2019
ENV-SOP-MTJL-0221 V06	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	4/8/2020
ENV-SOP-MTJL-0221 V07, 08, 09	Metals by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) (EPA Methods 6020, 6020A, 6020B & 200.8)	2/23/2022

Appendix A: Target Analyte List and LOQ

Table 1: Standard Analyte List and LOQ

[1] Values as of effective date of this SOP. LOQ are subject to change, contact quality personnel for most current information.

Analyte	CAS #	200.8 LOQ[1] Aqueous (mg/L)	6020 LOQ[1] Aqueous (mg/L)	6020 LOQ[1] Solid (mg/Kg)
Aluminum	7429-90-5	0.1	**0.1**	**10**
Antimony	7440-36-0	0.005	**0.004**	**0.6**
Arsenic	7440-38-2	0.001	**0.002**	**0.2**
Barium	7440-39-3	0.005	**0.002**	**0.5**
Beryllium	7440-41-7	0.001	**0.002**	**0.5**
Boron	7440-42-8	0.04	**0.03**	**10**
Cadmium	7440-43-9	0.001	**0.001**	**0.2**
Calcium	7440-70-2	1	**1**	**100**
Chromium	7440-47-3	0.02	**0.002**	**1**
Cobalt	7440-48-4	0.002	**0.002**	**0.2**
Copper	7440-50-8	0.001	**0.005**	**1**
Iron	7439-89-6	0.1	**0.1**	**10**
Lead	7439-92-1	0.002	**0.002**	**0.4**
Lithium	7439-93-2	0.002	**0.002**	**0.3**
Magnesium	7439-95-4	1	**1**	**100**
Manganese	7439-96-5	0.005	**0.005**	**0.5**
Molybdenum	7439-98-7	0.005	**0.005**	**0.5**
Nickel	7440-02-0	0.002	**0.002**	**0.5**
Potassium	9/7/7440	1	**2**	**100**
Selenium	7782-49-2	0.002	**0.002**	**0.5**
Silver	7440-22-4	0.001	**0.002**	**0.1**
Sodium	7440-23-5	2	**2**	**100**
Strontium	7440-24-6	0.01	**0.01**	**1**
Thallium	7440-28-0	0.001	**0.002**	**0.4**

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Thorium	7440-29-1	0.01	**0.01**	**5**
Tin	7440-31-5	0.004	**0.002**	**2**
Titanium	7440-32-6	0.01	**0.02**	**1**
Uranium	7440-61-1	0.001	**0.001**	**3**
Vanadium	7440-62-2	0.005	**0.005**	**0.5**
Zinc	7440-66-6	0.02	**0.025**	**5**

Appendix B: QC Summary and Corrective Action Table

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99. For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint calibration standard.	Identify and correct source of problem, repeat	None. Do not proceed with analysis No samples shall be analyzed until ICAL has passed
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All analytes must be within ± 10% of the true value.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
CCV	Daily, before sample analysis, after every 10 field samples, and at end of analytical sequence.	All reported analytes within ± %10 of the true value	Immediately analyze two additional consecutive CCV's. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCV's cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated 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 valid CCV's. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards	Every field sample, standard and QC sample	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from matrix effect.	Flagging is not appropriate. Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

			Reanalyze the sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem and rerun all associated failed field samples.	
Method Blank	One per preparatory batch	The absolute values of all analytes must be <1/2 LOQ or <1/10th the regulatory limit, whichever is greater. **STATE NOTE:** For blanks associated with samples originating from West Virginia and analyzed by Method 200.8, blank results should be below the LLOQ for the target analytes being tested; otherwise, corrective action should be taken. For blanks associated with samples originating from West Virginia and analyzed by Method 6020B, Blanks are generally considered to be acceptable if target analyte concentrations are less than the LLOQ or are less than project-specific requirements. **STATE NOTE**: For Wisconsin samples, the method blank must not contain analytes more negative than the MDL value. If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. **STATE NOTE**: For West Virginia samples analyzed by Method 200.8, When blank values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained. For West Virginia samples analyzed by Method 6020B, if the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be reprepared and re-analyzed, along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should	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. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

			report the sample results along with the method blank results, and provide a discussion of the potential impact of the contamination on the sample results. However, if an analyte of interest is found in a sample in the batch near its concentration confirmed in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification. **STATE NOTE**: For Wisconsin samples, If target analytes are more negative than the MDL, the instrument must be recalibrated or a new LOD study performed.	
LCS	One per preparatory batch	The acceptance range for water samples is ±15% of the target concentration for 200.8 and +20% for 6020. If an LCS/LCSD pair is analyzed, the RPD of each analyte must be ≤20%. **For EPA Method 200.8** – The acceptance range for water samples is ±15% of the target concentration. The RPD for each analyte in the pair must be ≤20%. **For SW-846 Method 6020/6020A/6020B** – The acceptance range for water samples is ±20% of the target concentration. The RPD for each analyte in the pair must be ≤20%.	Correct the problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient 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. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Results may not be reported without a valid LCS.
MS/MSD or Sample/Matrix Duplicate (MD)	An MS/MSD pair is analyzed with each batch of samples	Acceptance ranges for accuracy of the MS and MSD are ±30% of the target analyte concentration. The RPD of the pair must be ≤20%. **For EPA Method 200.8** – The acceptance range for water samples is ±30% of the target	The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits or project limits, if specified. If the specific analyte(s) are	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative. For matrix evaluation only. If MS results are outside the limits, the data shall be

		concentration. The RPD for each analyte in the pair must be ≤20%. **For SW-846 Method 6020/6020A/6020B** – The acceptance range for water samples is ±25% of the target concentration. The RPD for each analyte in the pair must be ≤20%.	not available, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.	evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error). For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
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.
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 (LLCCV) LLCCV should be less than or equal to the LOQ If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.
ICB/CCB	Immediately after the ICV and immediately after every CCV	The absolute values of all analytes must be <1/2 LOQ or 1/10th the amount measured in any sample	ICB: Correct problem and repeat ICV/ICB analysis. If that fails, rerun ICAL. All samples following the last acceptable Calibration Blank must be reanalyzed. CCB's may not be reanalyzed without reanalysis of the associated samples and CCV(s)	Flagging is not appropriate. Results may not be reported without valid Calibration Blanks. Non-detects associated with positive blank infractions may be reported. Sample results >10x the LOQ associated with negative blanks may be reported. For CCB, failures due to carryover may not require an ICAL.
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 >50x LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike	One post digestion spike must be analyzed for each batch of samples analyzed. The recovery for the target analyte should be ±25% of true value.	**For SW-846 Method 6020A** - The recovery for the target analyte should be ±20% of true value. **For SW-846 Method 6020 & 6020**B - The recovery for the target analyte should be ±25% of true value. **For EPA Method 200.8 –** Post digestion spikes are not required	No specified 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.
Linear Dynamic Range	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 reestablish/verify the LDR	Flagging is not appropriate. Data cannot be reported above the calibration range without an established/passing high-level check standard.
Spectral Interference Checks	After ICAl and prior to analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes. ICS-AB: Within ±20% of true value Method 6020B - Results for the unspiked elements in the SIC solution should be less than two (2) times the LLOQ. Note that it may not be possible to obtain SIC spiking solutions that are completely free of the unspiked elements. If the presence and concentration of an unspiked element can be confirmed via vendor documentation and/or determination of multiple isotopes of the element in the correct ratios, the concentration actually present may be subtracted from the determined value prior to comparing to the LLOQ limits.	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. Note: The unspiked elements in the ICSA are not evaluated by the data capture software. The instrument analyst evaluates if the unspiked elements are <2 times the LLOQ for the ICSA
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if required by the project	N/A	N/A	Document use of MSA in the Case Narrative

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Serial Dilution	One serial dilution must be analyzed for each batch of samples analyzed, excedpt for drinking water samples by EPA 200.8.	**For SW-846 Method 6020 & 6020A** - The serial dilution should recover with <10% difference from the parent sample **For SW-846 Method 6020** - The recovery for the target analyte should be ±25% of true value. **For EPA Method 200.8 –** Serial Dilutions are not required by the published method.	If the post spike result is acceptable, no action is taken. If both the post spike and the serial dilution are out, the results are flagged with O1.	If both the post spike and the serial dilution are out, the results are flagged with O1.
Reporting Limit Verification	When required, the reporting limit verification must be analyzed. **METHOD NOTE**: For EPA Method 6020A analyses, an RLV must be performed periodically.	The reporting limit verification when analyzed must recover within +50% of the target concentration for the standard. **METHOD NOTE:** For EPA Method 6020A analyses, Recoveries must be within +30% of the expected true value for each analyte being reported using that method. **STATE NOTE:** For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within +40% of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.		

Appendix C: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes changing tubing, changing cones, changing nebulizer tips, changing oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

MassHunter 4.1, Version C.01.01, Agilent Technologies, Inc., 2014.

3.0 Troubleshooting

Table 1. ICP-MS Troubleshooting Guide

Problem	Cause	Treatment
Low Sensitivity	Peristaltic Pump Issues	Adjust pumping rate
	Nebulizer Clogged	Clean or replace nebulizer
	Leaks	Check tube connector and sample lines for leaks
	Cone Changes	Ensure that the diameters and shapes of the holes in cones have not changed.
	Torch	Ensure the torch is positioned correctly
	Mass Axis	Ensure the mass axis is adjusted correctly
	Lens	Make sure the signal changes when the lens voltage is varied.
	Gas Purity	If the sensitivity is acceptable in the no gas mode and is too low in He or H2 mode, the problem may be caused by the purity level of the cell gas
Plasma Does Not Ignite	Spray Chamber	Ensure that samples do not collect in the spray chamber
	Air	Ensure that air is not mixed from connection points.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.4 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks

Performance Check	Frequency	Acceptance Criteria
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e. 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: 0˚C to 6˚C Freezers: ≤-10˚C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.19 of this addendum

4.5 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.6 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.7 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.8 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.9 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst case basis (preparation method with all applicable cleanup steps).

4.10 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least two (2) times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- **The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.**
- **If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.**
- **The laboratory shall maintain documentation for all MDL determinations and LOD verifications.**
- **The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.**

4.11 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.12 For DoD, 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.

4.13 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is

acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.14 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.15 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.16 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- **If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.**
- **Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.**
- **If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.**
- **Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.**
- **Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.**

4.17 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.18 Surrogate spike results shall be compared with DoD LCS limits (see Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.19 The method blank shall be considered contaminated if:

- **The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater.**

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- **The concentration of any common laboratory contaminant in the blank exceeds the LOQ;**
- **If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.**

4.20 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.21 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

Table 3. LCS Control Limits – Method 6020 Solid Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	919	101	7.7	78	124
7440-36-0	Antimony	1911	98.2	8.7	72	124
7440-38-2	Arsenic	3686	99.8	6	82	118
7440-39-3	Barium	2598	100.6	5	86	116
7440-41-7	Beryllium	2457	100.3	6.6	80	120
7440-42-8	Boron	581	101.1	9	74	128
7440-43-9	Cadmium	2893	99.6	5.4	84	116
7440-70-2	Calcium	835	102.2	5.4	86	118
7440-47-3	Chromium	2420	100.8	6	83	119
7440-48-4	Cobalt	2005	99.7	5.1	84	115
7440-50-8	Copper	2548	101.3	5.8	84	119
7439-89-6	Iron	1131	102.7	7.1	81	124
7439-92-1	Lead	3228	101	5.7	84	118
7439-93-2	Lithium	162	97.8	7.5	75	120
7439-95-4	Magnesium	868	101.6	7.1	80	123
7439-96-5	Manganese	1830	100.3	5.1	85	116
7439-97-6	Mercury	226	99.9	8.8	74	126
7439-98-7	Molybdenum	1188	98.1	5.1	83	114
7440-02-0	Nickel	2617	101.4	5.8	84	119
7440-09-7	Potassium	803	102.3	5.7	85	119
7782-49-2	Selenium	3104	99.2	6.6	80	119
7440-22-4	Silver	2488	100.1	5.9	83	118
7440-23-5	Sodium	818	102.2	7.7	79	125
7440-24-6	Strontium	676	101.7	8.9	75	129
7440-28-0	Thallium	2589	100.1	5.9	83	118
7440-29-1	Thorium	341	98.4	5.7	81	116

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Table 3. LCS Control Limits – Method 6020 Solid Matrix						
CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7440-31-5	Tin	886	101.3	6.6	82	121
7440-32-6	Titanium	512	100.2	5.7	83	117
7440-61-1	Uranium	833	101.1	6.1	83	120
7440-62-2	Vanadium	1677	99.1	5.7	82	116
7440-66-6	Zinc	2352	100.1	6.2	82	119

Table 4. LCS Control Limits – Method 6020 Water Matrix						
CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
7429-90-5	Aluminum	3145	100.6	5.4	84	117
7440-36-0	Antimony	5172	100.9	5.3	85	117
7440-38-2	Arsenic	6404	100.1	5.3	84	116
7440-39-3	Barium	4452	99.9	4.8	86	114
7440-41-7	Beryllium	4297	102	6.3	83	121
7440-42-8	Boron	1460	101.5	9.6	73	130
7440-43-9	Cadmium	5699	100.8	4.7	87	115
7440-70-2	Calcium	2085	102.3	5.2	87	118
7440-47-3	Chromium	5569	100.6	5.1	85	116
7440-48-4	Cobalt	3885	100.7	4.7	86	115
7440-50-8	Copper	5092	101.4	5.4	85	118
7439-89-6	Iron	3135	102.4	5.2	87	118
7439-92-1	Lead	6868	101.7	4.5	88	115
7439-93-2	Lithium	461	102.3	8	78	126
7439-95-4	Magnesium	2399	100.4	5.9	83	118
7439-96-5	Manganese	4330	101.1	4.7	87	115
7439-97-6	Mercury	328	97.2	9	70	124
7439-98-7	Molybdenum	2908	99.3	5.4	83	115
7440-02-0	Nickel	5095	100.8	5.3	85	117
7440-09-7	Potassium	2154	101.2	4.7	87	115
7782-49-2	Selenium	5797	100.1	6.7	80	120
7440-22-4	Silver	4956	100.8	5.1	85	116
7440-23-5	Sodium	2313	100.7	5.3	85	117
7440-24-6	Strontium	1170	99.9	5.9	82	118
7440-28-0	Thallium	5352	99.3	5.6	82	116
7440-29-1	Thorium	313	103.7	5.7	87	121
7440-31-5	Tin	1509	100.6	4.8	86	115
7440-32-6	Titanium	1538	98.6	5.3	83	115
7440-33-7	Tungsten	130	103.5	6.2	85	122
7440-61-1	Uranium	1860	103.3	5.4	87	120
7440-62-2	Vanadium	3375	100.5	5	86	115
7440-66-6	Zinc	4253	101	6	83	119

Pace ANALYTICAL SERVICES	**ENV-SOP-SHRT-0162 v00_In Vitro Bioaccessibility Assay for Metals in Soil by EPA 1340**
	Effective Date: 05/01/2023

Management Approval:
Crystal Herman Approved on 5/1/2023 11:56:51 AM
Stephen Kasa Approved on 5/1/2023 10:53:16 AM
Wade Nieuwsma Approved on 4/27/2023 1:35:42 PM
Amy Eady Approved on 5/1/2023 12:10:06 PM

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of Lead by In Vitro Bioaccessibility Assay (IVBA) for lead-contaminated soil under field conditions.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analyte for the extraction solution from this procedure for which there are defined limits is lead. The EPA has some limits associated with arsenic as well. While it has not been directly evaluated by EPA, it is assumed that this extraction procedure and the subsequent analyses may include other heavy metals from the RCRA list with reasonable success.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated and established as dissolved parameters.

2.0 SUMMARY OF METHOD

After drying and sieving, 1 g of soil sample is rotated with 100 mL of buffered extraction fluid at 37±2°C for one hour. The supernatant is separated from the sample by filtration and analyzed for lead by an appropriate analytical method (e.g., Method 6010 or Method 6020).

3.0 INTERFERENCES

3.1 Reagents, plasticware, glassware, and other sample handling equipment may yield artifacts and/or interferences during sample analysis. These materials should be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Also refer to Methods 6010 and 6020 and or other determinative methods to be used for information regarding potential interferences.

3.2 At present, it appears that the predictive relationship between IVBA and RBA (Relative Bioavailability) is widely applicable, having been found to hold true for a wide range of different soil types and lead phases from a variety of different sites. However, the majority of the samples tested have been collected from mining and milling sites, and it is plausible that some forms of lead that do not occur at these types of sites might not follow the observed correlation. Thus, whenever a sample containing an unusual and/or untested lead phase is evaluated by the IVBA protocol, this sample should be identified as a potential source of uncertainty. In the future, as additional samples with a variety of new and different lead forms are tested by both in vivo and in vitro methods, the limits on applicability of the method will be more clearly defined. In addition, excess phosphate in the sample medium may result in interference (i.e., the assay is not suited to phosphate-amended soils).

4.0 DEFINITIONS

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Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

4.1 Bioavailability (BA)–The fraction of an ingested dose (i.e., in vivo) that crosses the gastrointestinal epithelium and becomes available for distribution to internal target tissues and organs.

4.2 Absolute bioavailability–Bioavailability expressed as a fraction (or percentage) of a dose.

4.3 Relative bioavailability (RBA) –The ratio of the bioavailability of a metal in one exposure context (i.e., physical chemical matrix or physical chemical form of the metal) to that in another exposure context. For this method, RBA is defined as the ratio of bioavailability of lead in soil to lead in water.

4.4 Bioaccessibility–An in vitro measure of the physiological solubility of the metal that may be available for absorption into the body.

4.5 Batch–A group of analytical and control/QC samples that are extracted simultaneously and is limited to 20 environmental samples in addition to the batch QC samples.

4.6 Phosphate-amended soil–phosphate-rich materials (e.g., fertilizers) applied to lead-contaminated soils

4.7 In vitro–outside the living body and in an artificial environment

4.8 In vivo–in the living body of an animal

4.9 In vitro bioaccessibility (IVBA)–the physiological solubility of the metal that may be available for absorption into the body.

5.0 HEALTH AND SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible

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with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Soil		5g dried and processed to <150µm	Thermal: none Chemical: none	Collection to Prep: Prep To Analysis:

[1] *Amount of sample required for each discrete test.*

After receipt, samples are stored at ambient temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ambient temperature until sample analysis. Once the samples are prepared, no preservatives or special storage conditions are required.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for five months and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

7.1 Equipment

Item	Vendor*	Model ID*	Catalog ID*	Description
Water bath				If the water bath option is used, the specific extraction device is an electric motor-(i.e., the same motor as is used in the toxicity characteristic leaching procedure [TCLP] Method 1311) driven flywheel, which drives a rotating

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Item	Vendor*	Model ID*	Catalog ID*	Description
				block situated inside a temperature-controlled water bath. The extraction device must be capable of holding a capped 125-mL wide-mouth high-density polyethylene (HDPE) bottle. The water bath should be filled such that the extraction bottles are completely immersed. Temperature in the water bath should be maintained at 37±2°C using an immersion circulator heater, and the water bath temperature should be monitored and recorded. The electric motor must be capable of 30±2 rotations per minute (rpm).
Incubated air chamber				If the air incubator option is used, the specific extraction device will rotate the extraction bottles within an incubated air chamber. It must be capable of rotating at 30±2 rpm and be designed to hold multiple capped 125-mL wide-mouth HDPE. The incubator must be capable of maintaining 37±2°C. The temperature inside of the incubator

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Item	Vendor*	Model ID*	Catalog ID*	Description
				should be monitored and recorded.
HDPE bottles				125 mL in size, equipped with airtight screw-cap seals should be used. Care should be taken to ensure that the bottles do not leak and to minimize contamination during the extraction procedure
Automated temperature compensation (ATC) pH electrode and meter				Used for measuring the pH of the extraction fluid both prior to and after extraction; readable to 0.01 s.u.

*or equivalent

7.2 Supplies

Item	Vendor*	Model ID*	Catalog ID*	Description
Analytical Balance	Various	Various	Various	Capable of weighing to 0.01g
Graduated cylinder	Fisher Scientific	Fisherbrand	100mL = 08-557D	Fisher Scientific
Volumetric flasks				2 L, Class A
Sample containers (Extraction vessels)	Fisher Scientific	Thermo	05-721-142	125 mL HDPE wide-mouth; free of target analytes
Sample containers				PP, used to receive extracted samples
Syringes				Disposable
Filter				Disposable cellulose acetate syringe filters, 0.45 µm
Thermometer				NIST Traceable Capable of measuring 37±2°C
Pipettes				Calibrated per lab Support Equipment Procedure
Timer				NIST Traceable

*or equivalent

8.0 REAGENTS & STANDARDS

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8.1 Reagents

Reagent	Description/Concentration	Vendor*	Catalog #*
Reagent water	DI water, free of analytes of interest at the MDL	N/A	N/A
Glycine			
Hydrochloric acid (HCl)	Trace metal grade, concentrated		
Extraction fluid	Add 60.06 g of glycine (free base) to a 2L volumetric flask containing 1.9 L of deionized water. Solution can be transferred to a wide-mouth HDPE bottle for ease of handling. Heat the extraction fluid to 37±2°C. Standardize the pH meter using an ATC pH electrode at 37±2°C or pH buffers maintained at 37±2°C in the water bath. Add trace metal-grade concentrated HCl (12.1 normal [N]) until the solution pH reaches 1.50±0.05. Transfer the pH-adjusted contents to the volumetric flask and bring the solution to a final volume of 2 L (0.4 M glycine). NOTE: If the extraction fluid is prepared in advance of the extraction, the extraction fluid must be heated to 37±2°C and the pH shall be adjusted to 1.50±0.05 using trace metal-grade concentrated HCl prior to conducting the extraction	N/A	N/A

*or equivalent

8.2 Standards

Standard	Description/Concentration	Vendor*	Catalog #*
NIST SRM 2711a	The NIST SRM 2711a should yield a mean IVBA result of 85.7% (acceptable IVBA range 75.2-96.2%). For the lead concentration (Pb soil) in the SRM, the median lead concentration presented in the Addendum to the NIST certificate for leachable concentrations determined using Method 3050 (1,300 mg/kg) should be used.	LGS Standards	NIST-2711A
pH buffer—Certified reference material	4.0		

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Standard	Description/Concentration	Vendor*	Catalog #*
pH buffer—Certified reference material	7.0		
pH buffer—Certified reference material	10.0		

* or equivalent

9.0 PROCEDURE

9.1 Equipment Preparation

Prior to measurement of extraction fluid pH, the pH meter should be calibrated using a minimum of two points usually pH4 and pH 7. Calibration is done at the temperature of 37±2°C with criteria of pH (1.50±0.05) of the extraction solution and samples after completion. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units.

9.1.1 Calibrate pH meter daily

9.1.2 Cleanliness of all materials used to prepare and/or store the extraction fluid and buffer is essential. All glassware and equipment used to prepare standards and reagents shall be properly cleaned, acid washed, and triple-rinsed with deionized water prior to use.

9.2 Sample Preparation

9.2.1 Homogenization / Sizing

All test soils should be prepared by drying (<40°C) and sieving the sample as received to <150 µm. Milling should NOT be employed to achieve the desired particle size. The <150 µm size fraction is used because this particle size is representative of that which adheres to children's hands. Stainless steel sieves are recommended. Samples should be thoroughly mixed prior to use to ensure homogenization.

9.3 Extraction Procedure

9.3.1 The extraction fluid for this procedure is 0.4 M glycine (free base, reagent-grade glycine in deionized water), adjusted to a pH of 1.50±0.05 at 37±2°C using trace-metal grade concentrated HCl. The extraction fluid should be pre-heated to 37±2°C.

9.3.2 Pre-heat the extractor water bath or incubator to 37±2°C. Record the temperature at the beginning and end of each extraction batch.

9.3.3 Soil samples should be thoroughly mixed immediately prior to subsampling for extraction to ensure homogenization (i.e., rotate sample bottles using X, Y, Z motion).

9.3.4 The extraction procedure begins by placing 1.00±0.05 g of sieved test material (<150 µm) into a 125-mL wide-mouth HDPE bottle. Record the weight of the soil to the nearest 0.01 g. (Care should be taken to ensure that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle.)

9.3.5 Measure 100±0.5 mL of the 37±2°C buffered extraction fluid (0.4 M glycine, pH 1.50±0.05), using a class A graduated cylinder or automated dispenser and transfer the extraction fluid to the 125-mL wide-mouth HDPE bottle. The bottle should be tightly sealed and then

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shaken or inverted to ensure that there is no leakage and that no soil is caked on the bottom of the bottle.

9.3.6 Fill the extractor (water bath extractor or rotating extractor inside of a pre-heated incubator) with the batch of 125-mL bottles containing test materials and QC samples. Record start time of rotation.

9.3.7 Samples are extracted by rotating the samples at 30±2 rpm for one hour.

9.3.8 After one hour, the bottles should be removed from the rotator, dried, and placed upright on the bench top to allow the soil to settle to the bottom.

9.3.9 An aliquot of sufficient quantity for analysis of the supernatant fluid is then removed directly from the extraction bottle and is filtered through 0.45 µm cellulose acetate filter into pre-cleaned PP sample containers. Use of disposable syringes with Luer syringe filters and a 40 mL volume is a suggested procedure to accomplish this.

9.3.10 Record the time that the extract is filtered (i.e., extraction is stopped). ***If the total time elapsed for the extraction and filtration process exceeds 90 minutes, the test must be repeated.*** (This may limit the total number of samples that can be processed in a batch.)

9.3.11 Measure and record the temperature and pH of fluid remaining in the extraction bottle. If the fluid pH is not within ±0.5 pH units of the starting pH, the test must be discarded, and the sample re-extracted.

NOTE: In some cases (mainly slag soils), the test material can increase the pH of the extraction buffer and this could influence the results of the bioaccessibility measurement. To guard against this, the pH of the fluid should be measured at the end of the extraction step (just after a sample was withdrawn for filtration and analysis). If the pH is not within 0.5 pH units of the starting pH (1.50±0.05), the sample should be re-extracted. If the second test also results in an increase in pH of >0.5 units, it is reasonable to conclude that the test material is buffering the solution. In these cases, the test should be repeated using manual pH adjustment during the extraction process, stopping the extraction at 5, 10, 15, and 30 minutes and manually adjusting the pH down to pH 1.50±0.05 at each interval by drop-wise addition of trace metal-grade HCl.

NOTE: In some cases, the amount of lead present in the sample will begin to saturate the extraction fluid, and the extraction response will cease to be linear. If the concentration of lead in the extract exceeds approximately 500 mg/L (depending on the sample matrix and mineralogy), this upper limit may have been reached. It is not recommended to analyze IVBA for soils exceeding a total lead concentration of 50,000 mg/kg in order to avoid saturation of the extraction fluid and because risk management decisions are not likely to be improved by analyzing IVBA for soil with concentrations of lead above this level. Reduce sample volume and re-extract report both values.

9.3.12 A worksheet has been developed to track the required components. Refer to ENV-FRM-SHRT-0181 for this information. Submit this document as it is necessary for the calculation of results.

9.4 Analysis

9.4.1 Sample extracts are analyzed for lead content by ICP or ICP-MS, following ENV-SOP-SHRT-0015, -0019 or other appropriate procedure.

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9.4.2 <150µm processed soil samples are also submitted for 3050/6010 for As/Pb in order to perform calculations.

10.0 DATA ANALYSIS & CALCULATIONS

10.1 Quantitative Identification

10.1.1 If the IVBA factor is to be determined, a split of each solid material (<150 µm) that has been subjected to this extraction procedure should be analyzed for total lead concentration using analytical procedures taken from SW-846 or a non-destructive method such as Instrumental Neutron Activation Analysis. If SW-846 methods are used, the solid material should be acid digested according to an appropriate preparation method (e.g., Method 3050 or Method 3051). The digestate should be analyzed for lead concentration by an appropriate analytical method.

10.2 Calculations

10.2.1 **In vitro bioaccessibility (IVBA)**

The IVBA is calculated and expressed on a percentage basis using the following equation: Where:

$$\boldsymbol{in\ vitro\ bioaccessibility} = \frac{\boldsymbol{Pb_{ext}} \times \boldsymbol{V_{ext}} \times \mathbf{100}}{\boldsymbol{Pb_{soil}} \times \boldsymbol{Soil_{mass}}}$$

Pb_{ext}= in vitro extractable lead in the in vitro extract (mg/L)

V_{ext}= extraction solution volume (L)

Pb_{soil}= lead concentration in the soil sample being assayed (mg/kg)

$Soil_{mass}$= mass of soil sample being assayed (kg).

Reference controlled form ENV-FRM-SHRT-0182 for performing calculations.

11.0 QUALITY CONTROL & METHOD PERFORMANCE

11.1 Quality Control

Prepare the following QC samples with each batch of samples. Refer to Appendix A for acceptance criteria and required corrective action(s).

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QC Check	Acronym	Frequency
Method Blank (extraction fluid run through the extraction procedure)	MB	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
LCS for Pb (spiked blank)	LCS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
NIST SRM 2711a for Pb		1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Matrix Spike for Pb	MS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Sample Duplicate	DUP	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Reagent Blank (extraction fluid not run through the extraction procedure)		1 per batch of samples

11.2 Instrument QC

Refer to analytical methods for specific instrument QC.

11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

11.3.2 Initial Demonstration of Capability

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

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All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-SHRT-0138 Data Handling Procedure for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix A for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

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14.0 MODIFICATIONS

The procedures in this SOP have not been modified from the reference test method(s) cited.

15.0RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Managemen*t and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: QC Summary & Corrective Action Table

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manua*l, current version.

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- US EPA SW-846 Update VI, Method 1340, Rev. 1, February 2017

18.0 REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
N/A	

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
N/A		

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Appendix A: QC Summary and Corrective Action Table

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Method Blank (MB)	1 per batch of 20 or fewer samples.	Less than report limit	Re-analyze; make new extraction fluid and reanalyze effected samples	Flag
LCS for Pb	1 per batch of 20 or fewer samples.	85-115%	Re-analyze or re-extract and reanalyze	Flag
NIST SRM 2711a for Pb	1 per batch of 20 or fewer samples.	75.2-96.2%	Re-analyze	None—do not proceed if this LCS does not meet acceptance criteria
Matrix Spike (MS) for Pb	1 per batch of 20 or fewer samples.	75-125%	Re-analyze; make new extraction fluid and reanalyze effected samples	Flag
Sample Duplicate	1 per batch of 20 or fewer samples.	RPD < 20%	Re-analyze; make new extraction fluid and reanalyze effected samples	Flag
Reagent Blank	1 per batch of samples	Less than report limit	Prepare new batch of extraction fluid and reprocess effected samples	None

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Standard Operating Procedure For Chain-of-Custody Records

INTRODUCTION

The chain-of-custody record allows for the tracking of possession and handling of individual samples from the time of field collection through laboratory analysis. All samples released from field operations shall be accompanied by either a laboratory-issued or VHB Chain-of-Custody Form (Attachment VHB-007-1). This is done to ensure the legal integrity of the sample materials collected. Every effort shall be made to keep as few people as possible in the chain of sample possession.

PROCEDURE

1. A completed laboratory-issued or VHB Chain-of-Custody Form shall accompany each set of samples released from the study site. The Chain-of-Custody Form for all samples shall include the following information:

   a. Client/Company Name
   b. Project Name/Number
   c. Sample Number/Identification
   d. Date and time of sample collection
   e. Analysis requested
   f. Inclusive Dates of Possession
   g. Signatures of persons involved in chain of possession

2a. After the Chain-of-Custody Form has been completely filled out, the sampler signs the initial "Relinquished by" box along with date and time. The completed COC should then be photographed or electronically scanned. If an overnight commercial courier service is used (e.g. FedEx) to ship the samples to the laboratory and the samples will be sealed in their own shipping container (e.g. cooler), and the sampler should seal the original Chain-of-Custody in the shipment container. Upon receipt of the samples, the laboratory representative will sign the “Received for Laboratory” box along with the data and time. If a local courier service is used, the sampler will obtain the signature of the next person (i.e. transporter) in the chain-of-custody (in the initial "Received by" box along with date and time. The transporter delivers the samples to the analytical lab, then signs the second “Relinquished by” box along with the date and time, and a laboratory representative signs the "Received for Laboratory" box along with the date and time.

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The final completed COC will be scanned by the laboratory and a copy will be provided to VHB in the sample log in information as well as the final lab report.

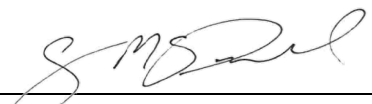
2b. Laboratory-issued Chain-of-Custody Forms will be treated in the same manner as above.

3. The Chain-of-Custody form shall be completed in legible hand writing with indelible ink, with all the appropriate information completed. Once completed, the form is either:

   a. placed in a water tight plastic bag and included with the samples in the sample shipping container, or;

   b. fixed in an envelope taped securely on top of the sample shipping container. This method allows for signatures to be included with each transfer of custody.

4. The sample shipping container shall be sealed with chain-of-custody tape, containing the date and sampler's signature. A minimum of one custody seal is required for each sample shipping container. Presence of an intact chain-of-custody tape will be documented by the laboratory upon receipt to ensure integrity of the sample shipment.

Revision Author:  Date: 3/14/2022
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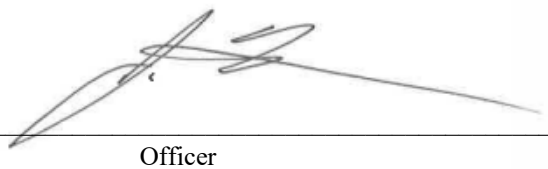
Reviewed:  Date: 3/14/2022
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Environmental Analysis Request/Chain of Custody

Acct. # _____ Group # _____ Sample # _____

Client:	
Project Name/#:	Site ID #:
Project Manager:	P.O. #:
Sampler:	PWSID #:
Phone #:	Quote #:
State where sample(s) were collected:	

For Lab Use Only

SF #: _____

SCR #: _____

Sample Identification	Collection Date	Collection Time	Grab	Composite	Matrix: Soil	Sediment	Water: Potable	Ground	Surface	NPDES	Other:	Total # of Containers	Analyses Requested / Preservation Codes	Remarks

Preservation Codes

H = HCl T = Thiosulfate

N = HNO_3 B = NaOH

S = H_2SO_4 P = H_3PO_4

O = Other

Turnaround Time Requested (TAT) (please check): Standard ☐ Rush ☐

(Rush TAT is subject to laboratory approval and surcharges.)

Date results are needed:

Rush results requested by (please check): E-Mail ☐ Phone ☐

E-mail Address:

Phone:

Data Package Options (please check if required)

Type I (Validation/non-CLP) ☐ MA MCP ☐

Type III (Reduced non-CLP) ☐ CT RCP ☐

Type IV (CLP SOW) ☐ TX TRRP-13 ☐

Type VI (Raw Data Only) ☐

EDD Required? Yes ☐ No ☐ If yes, format: _____

Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time
Relinquished by:	Date	Time	Received by:	Date	Time

Relinquished by Commercial Carrier:

UPS _____ FedEx _____ Other _____

Temperature upon receipt _____ °C

Standard Operating Procedure For Deviation from Protocols or Standard Operating Procedures and for Notation, Correction and Documentation of an Unforeseen Circumstance

INTRODUCTION

The objective of this procedure is to ensure the study quality and integrity in the event of:
(a) deviations from approved protocols or standard operating procedures, and
(b) unforeseen circumstances which may affect the quality of the study.

The procedure set forth herein is appropriate and desirable in all studies. However, it is a requirement of regulatory law for studies conducted under 40 CFR Part 160, the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA), Good Laboratory Practice Standards and 40 CFR Part 792, Toxic Substance Control Act (TSCA), Good Laboratory Practice Standards.

Specifically, FIFRA requires at 40 CFR § 160.81 (Standard Operating Procedures) that
"(a) A testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study. All deviations in a study from standard operating procedures shall be authorized by the Study Director and shall be documented in the raw data. Significant changes in established standard operating procedures shall be properly authorized in writing by management."

At 40 CFR 160.33 (Study Director)...
"The Study Director shall assure that:
(a) the protocol, including any change, is approved as provided by §160.120 and is followed. *[Emphasis added]* . . .
(c) unforseen *[sic]* circumstances that may affect the quality and integrity of the study are noted when they occur, and corrective action is taken and documented. *[Emphasis added]* . . .
(e) all applicable good laboratory practice regulations are followed."

Further, at 40 CFR § 160.35 (Quality assurance unit)…
"(b) The quality assurance unit shall: . . .
(5) Determine that no deviations from approved protocols or standard operating procedures were made without proper authorization and documentation." *[Emphasis added]*

PROCEDURE

A. Record of Deviation from Protocol or Standard Operating Procedure

1. Record the facts of the deviation legibly in ink using a Field Form, field book, bench log, or other recognized and reliable record-keeping device at the time of the deviation. Initial and date each page of entry at that time.

   The facts of the deviation should include but not necessarily be limited to:

   Date of occurrence:
   Time:
   VHB File No.:

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Study designation/Investigation name:
Test Substance:
CAS No.:
Study sponsor:
Study phase or segment:
Weather:
Temperature:
Personnel on site:
Visitors:
Contractors:
Equipment:
Standard Operating Procedure (title and number):
Protocol (title, date, section):
Type of deviation (inadvertent or planned):
Nature of deviation:
Observations:
Problems:

2. List anticipated consequences of deviation.

3. Determine corrective action alternatives and rank with most desirable first.

4. Contact Study Director/Project Manager as soon as conveniently possible for authorization to proceed with corrective action.

5. Document the corrective action, e.g. change in procedure, materials, equipment, personnel, time.

6. Retain all notes and records of the deviation for archiving as raw data.

7. Prepare full documentation of the facts of deviation, authorization and corrective action and file with the Project records..

B. Record of Unforeseen Circumstances Which May Affect the Quality of the Study

1. Record the facts of the unforeseen circumstances legibly using a Field Form field book, bench log, or other recognized and reliable record keeping device at the time of occurrence. Initial and date each page of entry at that time. The facts of the unforeseen circumstances should include, but not necessarily be limited to, those items listed under A. 1. above. A wide variety of unforeseen circumstances may affect the quality of a study. For example, a lightning strike induces a charge on a cable and distorts information in the data logger or destroys a sensing unit at the weather station; a hurricane flood submerges monitoring wells; a bee sting or threat of snake bite causes personnel to drop a box of water samples; or an accident delays delivery of refrigerated samples.

2. List the consequences of the unforeseen circumstance.

3. Determine if corrective action is possible.

4. List corrective action alternatives with most desirable first.

5. Notify Study Director/Project Manager as soon as conveniently possible of unforeseen

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circumstances.

6. Obtain authorization from Study Director/Project Manager to proceed with corrective action.

7. Document the corrective action, e.g. change in procedure, materials, equipment, personnel, time.

8. Retain all original notes and records of the unforeseen circumstances for archiving as raw data.

9. Prepare full documentation of the facts of the unforeseen circumstances, notification, authorization, and corrective action..

Author:_____ Date: 3/14/22
Scientist, Engineer or Technician

Reviewed:_____ Date:_____
Technical Reviewer

Approved:_____ Date: 3/14/22
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Standard Operating Procedure for Soil Sampling for Organic and Inorganic Compounds

INTRODUCTION

The soil sampling procedure detailed below outlines the collection and preservation procedures of soil, sediment, and sludge samples for organic and inorganic analysis. Organic compounds can be broken into two groups: volatile and semi-volatile. Volatile compounds are either gasses or those compounds which readily vaporize at standard temperature and pressure. Semi-volatile organic compounds are defined as those compounds that do not vaporize readily at standard temperature and pressure. Inorganic compounds addressed in this procedure include metals and cyanide. This procedure deals with semi-volatile and inorganic analyses only; if volatile organics sampling is to be performed, refer to VHB SOP 021.

PROCEDURES

A. Precautions

The following should be considered when collecting samples:

- Sample collection techniques should comply with the applicable site-specific workplan and methods. Field team members should review and be familiar with these prior to initiation of field activities.
- All field personnel should be familiar with the Site-Specific Safety and Health Plan and be aware of any site-specific challenges or requirements.
- Documentation of field activities should comply with VHB SOP 034 – Procedure for Use of Field Log Books

B. Sample Collection

1. Sample collection shall be performed in a manner that results in minimal disturbance of the medium. The methods of collection shall be chosen based on considerations of soil, sediment, or sludge integrity and representative sample collection. To the extent practical, sample collection devices should be constructed of non-reactive materials (e.g., stainless steel, Teflon, and glass). Examples of common collection devices include Eckman dredge, hand auger, split spoon, shovel or trowel.

2. Laboratory analytical samples should be obtained from material that is as undisturbed by the sampling equipment/collection process as possible.

3. Using the sample collection device, obtain the required volume of soil for the specific analytical technique. This information will be provided by the laboratory along with the necessary glassware. A properly decontaminated stainless-steel spoon or spatula, a dedicated disposable spatula, or a clean disposable nitrile glove may be used to transfer sediment or soil from the sampling device to the analytical jar. For composite sampling, the media may be transferred to a tray or bowl, then

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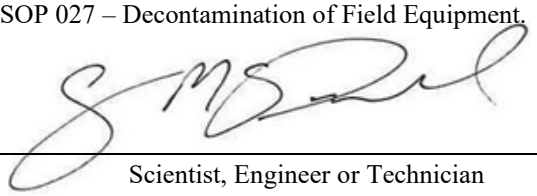
transferred to the sample containers after compositing.

4. Carefully cap the sample bottle tightly. Ensure the container is labeled with the proper sample information and recorded on the chain of custody. Sample containers should be placed in a cooler with ice as soon as possible if sample preservation requirements include refrigeration. For most analytes, samples should be stored and transported at a temperature of 4° +/- 2° Celsius.

5. Samples should be packed and transported to the analytical laboratory in accordance with U.S. Department of Transportation Rules and Regulations.

C. Decontamination

All non-dedicated sampling equipment should be decontaminated between uses in accordance with VHB SOP 027 – Decontamination of Field Equipment.

Author:  Date: 3/14/22
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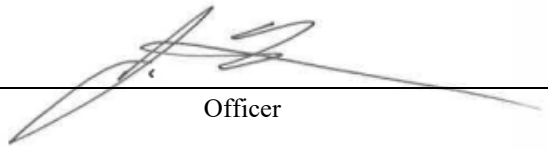
Approved:  Date: 3/14/22
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Standard Operating Procedure For
Decontamination of Field Equipment

INTRODUCTION

Decontamination procedures are performed as a quality assurance measure and a safety precaution. The overall integrity of the sampling program is maintained, in part, through a conscientious equipment decontamination program. The procedure prevents cross-contamination between sample stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination should be used in combination with other best practices to minimize the potential for cross-contamination between environmental samples. Other best practices include sampling low concentration media before relatively high concentration media (to the extent such information is known in advance) and selecting sampling equipment and sampling materials appropriate for the constituents of concern.

Decontamination is mainly achieved by rinsing with liquids which may include the following, as appropriate: detergent solutions, tap water, deionized water, and/or solvents such as methanol and Citra-solv. Equipment will be allowed to air dry after being cleaned or may be wiped dry with chemical-free cloths or paper towels.

Decontamination shall, in most cases, be accomplished at each sampling site, between uses at separate sample collection points. Waste produced by the decontamination procedures may require collection, handling, and disposal as investigation-derived waste (IDW). Always consult the Site Specific Workplan for specific instructions regarding disposal of IDW.

A. General Considerations

1. Planning: When planning fieldwork, if possible, create an investigation or sampling sequence that starts with the lowest relative contaminant concentration location and proceeds through progressively more concentrated locations to the highest concentration location. Possible exceptions include sediment and surface water sample collection, which should always proceed in an upstream direction. In the absence of pre-existing analytical data to indicate the degree of contamination, the judgment of the person planning the fieldwork will inform the sequence of investigation or sampling, in accordance with this guidance.

2. Clothing: Persons performing decontamination procedures shall at a minimum wear the same level and type of personal protective equipment (PPE) during decontamination as required for the associated fieldwork. Personnel performing decontamination should at a minimum use eye protection and chemical-resistant gloves.

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3. Location: Decontamination procedures are generally performed at the location of fieldwork to minimize the spread of contaminants. When numerous investigation or sample locations are very close together, as in the case with well clusters, decontamination stations may be warranted. Clean plastic sheets should be placed on the ground at the decontamination site to separate sampling equipment from ambient debris and soil, and to contain decontamination fluids.

4. Collection of decontamination fluids: Project-specific plans, protocols, or specifications may require collection of fluids used to decontaminate field equipment. Field workers should be aware of project-specific requirements before beginning fieldwork and should arrive prepared to collect and store the fluids used if this requirement is applicable.

PROCEDURES

A. Specific Procedures for Hand-held Field Equipment

The following procedure is to be used to decontaminate hand held field equipment such as: hand augers, split-spoon samplers, purge pumps, shovels, trowels, or other equipment that comes into contact with soil or water.

1. List of Required Equipment and Supplies: Field managers are advised to supply the following materials in advance of fieldwork. The absence of required decontamination equipment and supplies is not an acceptable deviation from this procedure and may result in unusable or limited use of sample results.

    - Deionized water (ample quantities)
    - 5-gallon buckets
    - Detergent, such as Liquinox, Alconox, or Luminox. Certain project-specific applications may require the use of other detergents or solvents such as isopropanol or methanol.
    - Several large clean plastic sheets, drop cloths, or a plastic wading pool
    - Clean hand-held brushes
    - Chemical resistant gloves appropriate for site contaminants
    - Sample bottles for field blank collection
    - Hose or pressure spray tank for decontaminating larger devices
    - Citrus-based solvent, such as Citra-solv (for gross NAPL or organic solids contamination)
    - Paper towels
    - If collection of waste water is required:
        - Shovel or lumber (to create berms for plastic sheeting)
        - IDW containers (e.g., 55-Gallon DOT drums)
        - Funnel

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- Pumps and hose

2. Remove Solids and Gross Contamination: Remove mud, sludge, vegetation or other solids from the equipment before decontamination. Removal of solids is most easily done using a brush and ample quantities of water. Distilled or deionized water is not required for this step. A bucket can be used to hold small devices during this step, while larger items, such as hand augers, can be cleaned over plastic with a hose or pressurized spray tank, if available.

   Restrict the use of solvents to those instances in which gross contamination, such as NAPL or organic-based solids, is present. On those occasions, solvents may be used to remove gross contamination from the equipment, followed by a triple rinse with tap water to remove the solvent residue.

3. Remove Trace Contamination: Remove trace amounts of contamination with a solution composed of a laboratory grade detergent, and water. Small field devices can be immersed in a bucket of the solution and scrubbed with a brush, while larger field devices, such as hand augers, must be cleaned with a brush and hose or pressurized spray tank containing the solution. Replace detergent solution daily, or more often if it becomes visibly soiled. Replace worn or stained scrub brushes.

4. Remove Detergent Solution: Remove residual detergent solution after scrubbing by rinsing the device with tap water. Small field devices can be immersed in a bucket of tap water, provided suds are not present from the previous use. Larger devices must be rinsed with a hose or pressurized spray tank. The volume of water used to rinse the equipment should be larger than the volume of detergent solution used in the previous step. Replace rinse water frequently.

5. Final Rinse: Rinse all field equipment with deionized or distilled water to remove any residual soap or tap water. Repeat the rinse with fresh deionized or distilled water at least twice.

6. Field Drying: The equipment can then be allowed to air dry or dried with clean paper towels or a hot air blower.

7. Collection of Field Blanks/Equipment Rinsate Blanks: Field blanks/equipment rinsate blanks may need to be collected to confirm the effectiveness of field decontamination procedures. Consult site-specific plans or specifications to determine the frequency of blank collection and the analytical parameters. Blanks are created by collecting samples of deionized water which has been poured into, onto or through a piece of equipment after it has been decontaminated and dried.

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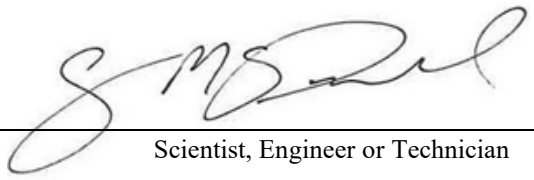
B. Specific Procedure for Drilling and Excavation Equipment

All drilling or excavation equipment that comes in contact with soil must be cleaned before use and between boreholes. This includes drill rods, bits and augers, dredges, backhoe buckets or any other large piece of equipment. Sampling devices, such as a split spoons and Shelby tubes, must also be decontaminated between uses.

DOCUMENTATION

Field workers shall record performance of decontamination procedures in the field logbook (see SOP-VHB-034) or on project-specific field data forms. The following information is to be recorded at a minimum: location of decontamination: sampling media; time of decontamination; list of decontaminated equipment; personnel performing decontamination; nature and identity of field blanks collected; deviations from the standard procedures; and any other relevant information. Proper documentation of decontamination procedures and blanks are critical quality analysis/quality control components of field sampling programs.

Author:  Date: 3/14/22
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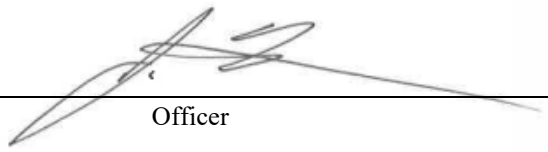
Approved:  Date: 3/14/22
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Standard Operating Procedure For Use of Field Log Books

INTRODUCTION

The use of a field log book is a component of an accurate and permanent record of activities, measurements and observations made in the field. The overall record of data and observations recorded in the field should be sufficiently clear and detailed to enable others to reconstruct field events.

PROCEDURE

All records, including original field and laboratory notes and reports, shall be recorded and maintained in the project file. Where appropriate, field data forms developed for specific field activities shall be used to reduce the potential for errors of omission. Field log books are used to record pertinent information not otherwise recorded on field data forms. Notes recorded in a field log book serve as a daily log of general field conditions, activities, site visitors, and personnel associated with the field work being performed.

A bound notebook shall be assigned to each member of the field team. Alternatively, a project-specific field notebook may be assigned and maintained by the project team. Use of water-resistant paper and indelible ink or marker shall be used when permitted by project-specific field protocols. Some field protocols, such as those associated with per- and polyfluoroalkyl substances (PFAS) sample collection and analysis, may include specific requirements for the types of paper, notebooks, and writing utensils permitted.

Each page in a field log book shall be labeled with the date, project name, project location, names(s) and initials of field personnel, and a page numbering sequence. The first page of each daily entry shall list the observed weather conditions; and name or initials of the day's personnel.

Data and observations generated during the conduct of a study, except those that are generated electronically, or recorded on separate project-specific field data forms, shall be recorded directly, promptly, and legibly in the field log book. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. Notes and records shall be accurate, composed for orderly use, complete, clean and legible.

Any changes to log book entries shall not obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or initialed at the time of the change. Erasures shall not be made.

All field notes shall be electronically scanned and added to the corresponding project file.

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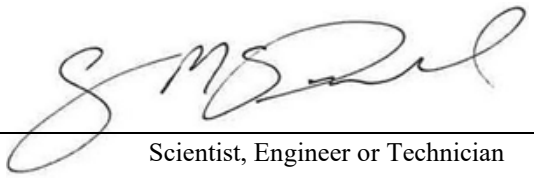
Author:  Date: 3/14/22
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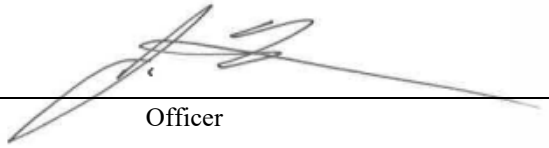
Approved:  Date: 3/14/22
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Standard Operating Procedure for Surface Water Sampling

INTRODUCTION

The goal of surface water sampling is to accurately assess the quality of the surface water at the sampling point. To accomplish this goal, measures must be taken to ensure that the sample is representative at that locality. Measures must also be taken to ensure the samples are not altered or contaminated during the sampling and handling process.

Sample collection station locations should be selected based upon knowledge of site conditions and with the objectives of the sampling program in mind. Site conditions to be examined include, but are not limited to, geologic conditions, such as natural drainage systems, identified environmentally sensitive areas, such as wetlands or wildlife habitats, stream morphology, and the proximity of the site to human activities.

The location of the sampling point in relation to the contaminant source is an important consideration. For instance, if the objective is to determine the overall water quality of a stream, composite samples can be taken in the center of the channel. Whereas, if the objective is to determine the quality of groundwater discharging to a stream, a sample may be taken along the stream bed close to the source area.

PROCEDURE

A. General

One of the two most common forms of surface water is standing water which includes lakes or ponds. Because circulation is usually slow in the enclosed basins of standing waters, contaminant plumes or stratification may result. It may be important to obtain samples that are representative of the water body both aerially and vertically, depending upon the objectives of the sampling program.

The other most common form of surface water is running water which includes streams, rivers and drainage swales. The selection of sampling locations for running waters is based on the objectives of the sampling program which may be one of the following:

1. To bracket a stream segment traversing a particular geomorphic zone or land use area.
2. To bracket a known or potential point source of pollution.
3. To investigate stream segments sensitive to water quality changes.
4. To quantify the rates of nutrient deposition at inlets or outlets of lakes or reservoirs.
5. To investigate water quality at the confluence of a tributary and a main stream or river.

B. Detailed

Surface water samples are generally obtained from the shoreline using a hand held sample container, or a sample container attached to the end of an extension device. Boats or overhead structures such as

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bridges may be required for sampling locations which are not accessible from shore. If the body of water is shallow and slow moving, it may be possible to wade to the sampling location.

When obtaining water samples at depth, more sophisticated sampling methods and boats are required. Weighted bottles and discrete samplers are the most common types of samplers used to obtain water samples at depth. These samplers remain closed until the required depth is reached. At that point, the collection chamber is opened and filled. Once the sampler is filled, the collection chamber is closed and the sampler is brought to the surface. Generally, when conducting water sampling at the surface and at discrete depth intervals, it is important to have a vertical datum established so that the depth of the sample can be accurately determined.

Following is a generalized sampling procedure which should be used when obtaining representative surface water samples. If possible, the order of sample collection should proceed from downstream to upstream.

1. Rinse the sample bottle at least three times with the water to be sampled, unless the sample bottle contains a preservative. Use only sample containers that are approved in the project workplan according to the specified analytical methods.

2. Clean and decontaminate the sampling equipment using the procedures set forth in SOP-VHB-027 if non-dedicated sampling equipment is being used. Lower a pre-cleaned sample collection vessel (eg. pond dipper, stainless steel beaker, weighted bottle, discrete sampler), or the actual sample bottle into the water at an approximate angle of 45° so as to minimize sample aeration. When sampling in flowing bodies of water, samples should always be collected facing upstream to avoid collection of any sediment or detritus disturbed during site access activities.

3. Upon complete immersion, remove the sampler and decant the sample into the pre-rinsed container described in Step 1. In the case of a sample bottle containing preservative, care must be taken to avoid overfilling the sample bottle and losing the preservative.

4. Cap, seal, and label the sample container according to the project-specified work plan and analytical methods.

5. Immediately place and store the sample in a cooler or other insulated storage device packed with refrigerant (bulk ice or ice-packs) to lower and maintain a sample temperature of less than 4° C.

6. If such sampling is required by the project workplan, perform field measurements of pH, conductivity, dissolved oxygen, and redox using the appropriate calibrated equipment (eg. pH meter, conductivity meter, dissolved oxygen meter). Wherever possible, the meter probes shall be placed directly into the sample source. Where access to the source is limited, a representative sample shall be placed in a decontaminated, clean container (SOP-VHB-027) and measurements taken immediately upon parameter stabilization.

The above described procedures are general in nature and may be altered based on the requirements of the project specific workplan and analytical program.

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C. Sample Size and Preservation

Specific data should be given for preservation, sample size, holding time, and analytical methodology in each project specific workplan. Attachment VHB-035-1, Table 1, Recommendation for Sampling and Preservation of Samples According to Measurement provides general guidance in the absence of overriding project specific guidance.

D. Documentation of Sample

A copy of VHB's standard Surface Water Sample Collection Record is attached. This form should be completed at each sample location to document sample date, time, and other required parameters based on the project specific workplan. Documentation of the sampling event will also consist of a completed Chain-of-Custody as described in SOP-VHB-007.

E. Transportation/Packing

Upon collection, samples are packed in an insulated cooler with refrigerant and delivered to the laboratory for analysis as soon as is reasonable, but within the maximum holding time for the sample. A completed Chain-of-Custody Form (SOP-VHB-007) will accompany the sample set.

Author:_____ Date: 3/14/22
Scientist, Engineer or Technician

Reviewed:_____ Date:_____
Technical Reviewer

Approved:_____ Date: 3/14/22
Officer

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Attachment SOP-VHB-035-1

Table 1. Recommendation for Sampling and Preservation of Samples According to Measurement

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TABLE 1. RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT [a]

Measurement	Vol. Req. (ml)	Container[b]	Preservative	Holding Time[c]
Physical Properties				
Color	50	P,G	Cool, 4°C	24 Hrs.
Conductance	100	P,G	Cool, 4°C	24 Hrs.[d]
Hardness	100	P,G	Cool, 4°C HNO_3 to pH<2	6 Mos.[e]
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	25	P,G	Det. on site	6 Hrs.
Residue				
Filterable	100	P,G	Cool, 4°C	7 Days
Non-Filterable	100	P,G	Cool, 4°C	7 Days
Total	100	P,G	Cool, 4°C	7 Days
Volatile	100	P,G	Cool, 4°C	7 Days
Settleable Matter	1000	P,G	None Req.	24 Hrs.
Temperature	1000	P,G	Det. on site	No Holding
Turbidity	100	P,G	Cool, 4°C	7 Days
Metals				
Dissolved	200	P,G	Filter on site HNO_3 to pH<2	6 Mos.[e]
Suspended	200		Filter on site	6 Mos.
Total	100	P,G	HNO_3 to pH<2	6 Mos.[e]

(continued)

TABLE 1. (continued)

Measurement	Vol. Req. (ml)	Container[b]	Preservative	Holding Time[c]
Mercury Dissolved	100	P,G	Filter on site HNO_3 to pH<2	38 Days (Glass) 13 Days (Hard Plastic)
Total	100	P,G	HNO_3 to pH<2	38 Days (Glass) 13 Days (Hard Plastic)
Inorganics, Non-Metallics				
Acidity	100	P,G	None Req.	24 Hrs.
Alkalinity	100	P,G	Cool, 4°C	24 Hrs.
Bromide	100	P,G	Cool, 4°C	24 Hrs.
Chloride	50	P,G	None Req.	7 Days
Chlorine	200	P,G	Det. on site	No Holding
Cyanides	500	P,G	Cool, 4°C NaOH to pH 12	24 Hrs.
Fluoride	300	P,G	None Req.	7 Days
Iodide	100	P,G	Cool, 4°C	24 Hrs.
Nitrogen				
Ammonia	400	P,G	Cool, 4°C H_2SO_4 to pH<2	24 Hrs.
Kjeldahl, Total	500	P,G	Cool, 4°C H_2SO_4 to pH<2	24 Hrs.[f]
Nitrate plus Nitrite	100	P,G	Cool, 4°C H_2SO_4 to pH<2	24 Hrs.[f]
Nitrate	100	P,G	Cool, 4°C	24 Hrs.
Nitrite	50	P,G	Cool, 4°C	48 Hrs.

(continued)

TABLE 1. (continued)

Measurement	Vol. Req. (ml)	Container[b]	Preservative	Holding Time[c]
Dissolved Oxygen Probe	300	G only	Det. on site	No Holding
Winkler	300	G only	Fix on site	4-8 Hrs.
Phosphorus Ortho-phosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	24 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to pH<2	24 Hrs.[f]
Total	50	P,G	Cool, 4°C H_2SO_4 to pH<2	24 Hrs.[f]
Total, Dissolved	50	P,G	Filter on site Cool, 4°C H_2SO_4 to pH<2	24 Hrs.[f]
Silica	50	P only	Cool, 4°C	7 Days
Sulfate	50	P,G	Cool, 4°C	7 Days
Sulfide	500	P,G	2 ml zinc acetate	24 Hrs.
Sulfite	50	P,G	Det. on site	No Holding
Routine Organics				
BOD	1000	P,G	Cool, 4°C	24 Hrs.
COD	50	P,G	H_2SO_4 to pH<2	7 Days[f]
Oil & Grease	1000	G only	Cool, 4°C H_2SO_4 or HCL to pH<2	24 Hrs.
Organic Carbon	25	P,G	Cool, 4°C H_2SO_4 or HCL to pH<2	24 Hrs.
Phenolics	500	G only	Cool, 4°C H_3PO_4 to pH<4 1.0 g $CuSO_4$/1	24 Hrs.

(continued)

TABLE 1. (continued)

Measurement	Vol. Req. (ml)	Container[b]	Preservative	Holding Time[c]
MBAS	250	P,G	Cool, 4°C	24 Hrs.
NTA	50	P,G	Cool, 4°C	24 Hrs.

a. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.

b. Plastic (P) or Glass (G). For metals polyethylene with a polypropylene cap (no liner) is preferred.

c. It should be pointed out that holding times listed above are recommended for properly preserved samples based on currently available data. It is recognized that for some sample types, extension of these times may be possible while for other types, these times may be too long. Where shipping regulations prevent the use of the proper preservation technique or the holding time is exceeded, such as the case of a 24-hour composite, the final reported data for these samples should indicate the specific variance.

d. If the sample is stabilized by cooling, it should be warmed to 25°C for reading, or temperature correction made and results reported at 25°C.

e. Where HNO_3 cannot be used because of shipping restrictions, the sample may be initially preserved by icing and immediately shipped to the laboratory. Upon receipt in the laboratory, the sample must be acidified to a pH < 2 with HNO_3 (normally 3 ml 1:1 HNO_3/liter is sufficient). At the time of analysis, the sample container should be thoroughly rinsed with 1:1 HNO_3 and the washings added to the sample (volume correction may be required).

f. Data obtained from National Enforcement Investigations Center-Denver, Colorado, support a four-week holding time for this parameter in Sewerage Systems. (SIC 4952).

Source: Manual of Ground-Water Sampling Procedures, 1981, M. R. Scalf, J. F. McNabb, W. J. Dunlap, and R. L. Cosby, National Well Water Association, Worthington, Ohio.

Attachment SOP-VHB-035-2

Surface Water Sample Collection Record

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Standard Operating Procedure for Geologic Description of Sediments

INTRODUCTION:

A description of geologic sediments is often part of site investigations, particularly during the installation of groundwater monitoring and observation wells. During drilling, samples of the sediment are typically collected with a subsurface coring device, such as a split-spoon sampler or direct push technology. These samples are then described to aid in well construction and in making interpretations about the site geologic and hydrologic characteristics. The parameters of major importance for this type of description are: color, mottling, texture (e.g., grain size, sphericity, and roundness), consistence, structure, moisture content and concretions. Site specific methods for the format and level of detail in relation to description of soil and sediment may vary, field personnel should be familiar with the Site Specific Work Plan for each investigation.

PROCEDURES:

1. Separating Significant Layers
   Each distinct sediment layer in the sample should be described separately. Layers may be differentiated based on any of the parameters listed above. It may be most appropriate to describe layers less than 0.5 inch thick as laminae. Color, texture, and consistence, as well as other significant and apparent properties, should be recorded for any such laminae. In a situation where several laminae of similar properties are located in a sample, it may be best to describe them as a group. For example, "8 gray silty clay laminae between 5.5 and 7.0 feet, each about 1/4 inch thick."

2. Color
   The color of the sediment should be described such that if available, they can be described according to the standardized charts such as Munsell Soil Color Charts. However, project-specific conditions may preclude availability. If standardized are unavailable, the color of the sediment of interest shall be described according to the best judgement of the recorder.

3. Mottling
   Mottles (color changes indicative of seasonal moisture level variations) present in the sediment will be described as to their color, abundance, size and contrast to the matrix color. The color of the mottle will be described to the best judgement of the describer, or if available the Munsell Soil Color standardized charts.

   Abundance and size classes of mottles are as follows:

   Abundance: Few (F) - less than 2% of the surface
   Common (C) - 2% to 20%
   Many (M) - more than 20%

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Size: Fine (1) - smaller than 5mm
Medium (2) - 5mm to 15mm
Coarse (3) - larger than 15mm

The contrast of mottles can be determined by use of the tabulation in Attachment 1.

4. Texture

Texture (grain size distribution) of the sediment will be described according to the estimated percentages of sand, silt and clay in the sample. Textural classes and the percentage ranges of the particle sizes associated with these classes are shown in Attachment 2.
Also to be included in the description of the texture is a description of the rock fragments in the sediment sample. These will include particle sizes over approximately 5 mm in diameter. The volumetric percentage of rock fragments will be estimated. The general shape of the rock fragments will be described according to the following terminology: rounded or subrounded, angular or subangular, as illustrated in Attachment 4. The mineralogy of the rock fragments will also be defined whenever possible.

5. Consistence

The consistence of the sediment will be described whenever it remains evident in the sample. Frequently the sample is compacted excessively during sampling and the natural consistence is not evident during examination. If the consistence can be characterized, it will be described according to the terms and definitions listed in Attachment 5.

The key distinction for this parameter is the change from friable to firm. When handling a sample that is friable, it will take only slight pressure between the thumb and fingers to cause a clod or ped of the sediment to break apart. A sample that is firm will resist crushing to the point where it finally seems to explode when it is subjected to adequate pressure. A friable clod or ped will not explode or pop, it will simply fall apart when pressure is applied. The other descriptive classes, as shown in Attachment 5, will be applied to add more information as to the degree of soil stability, or lack thereof.

6. Structure

Observations of structure will include a characterization of the orientation and continuity of contacts, angularity of grains (see Attachment 4), and a qualitative description of the degree of sorting. Additional description of such as features as crossbeds, grading, and varves will aid in identifying the depositional environment.

7. Moisture

The soil moisture will be described on the basis of visual and manual field observations. The moisture class will be based on the percent water in the soil as follows:

Moisture Class	Moisture Content, percent	Field Description

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Dry	0	No dampness is evident in sample
Humid	1-25	Slight damp mark seen on hand when sample is squeezed
Moist	26-75	Grading from humid to wet
Wet	76-99	Water is released when sample is squeezed
Saturated	100	Water drips freely from sample

8. Concretions

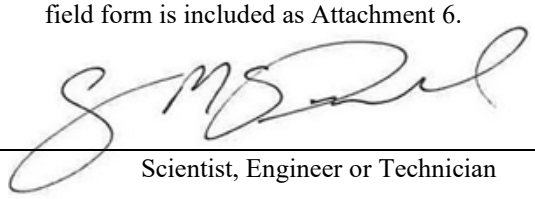
When iron or manganese concretions are present in a soil sample, they will be noted as to their abundance and size. The use of dilute hydrochloric acid is helpful in identifying the presence of carbonate minerals, though its use is not required.

9. Other Parameters

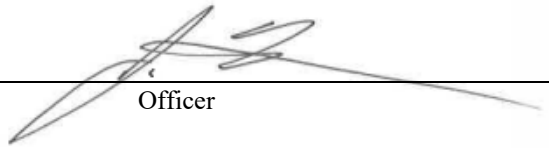
The protocols or work plan for a given study may require the determination of other parameters, e.g., screening for the presence of volatile organic compounds with a photoionization detector at a hazardous waste investigation. In such cases, the protocol, work plan, additional SOPs, or other authority or regulation will describe the additional or alternate method of sediment evaluation.

10. Data Recording

The information included in this SOP will be recorded either electronically (field computer based log), in field logbooks (SOP-VHB-034) or on field forms (Form VHB-Hydro-001) during the investigation. The information should include: the project name and number, well number, name of describer, well driller's name, date and other relevant information as indicated. A copy of the field form is included as Attachment 6.

Author:  Date: 3/14/22
Scientist, Engineer or Technician

Reviewed: _____ Date: _____
Technical Reviewer

Approved:  Date: 3/14/22
Officer

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Attachment SOP-VHB-036-001

"Determination of Soil Color"
Ref.: From Munsell Soil Color Charts
Baltimore: Kollmorgen Corporation, 1975,
3 pp.

MUNSELL®

SOIL COLOR

CHARTS

1975

EDITION

MUNSELL COLOR
MACBETH A DIVISION OF
KOLLMORGEN CORPORATION
2441 NORTH CALVERT STREET
BALTIMORE, MARYLAND 21218

DETERMINATION OF SOIL COLOR

quoted in part from

U.S. Dept. Agriculture Handbook 18—Soil Survey Manual

Soil colors are most conveniently measured by comparison with a color chart. The collection of charts generally used with soils is a modified version of the collection appearing in the Munsell Book of Color and includes only that portion needed for soils, about one-fifth of the entire range found in the complete edition.

The seven charts in the Soil Collection display 199 different standard color chips systematically arranged according to their Munsell notations, on cards carried in a loose leaf notebook. The arrangement is by the three simple variables that combine to describe all colors and are known in the Munsell system as Hue, Value, and Chroma.

The Hue notation of a color indicates its relation to Red, Yellow, Green, Blue, and Purple; the Value notation indicates its lightness; and the Chroma notation indicates its strength (or departure from a neutral of the same lightness).

The colors displayed on the individual Soil Color Charts are of constant Hue, designated by a symbol in the upper right-hand corner of the card. Vertically, the colors become successively lighter from the bottom of the card to the top by visually equal steps; their value increases. Horizontally they increase in Chroma to the right and become grayer to the left. The Value notation of each chip is indicated by the vertical scale in the far left column of the chart. The Chroma notation is indicated by the horizontal scale across the bottom of the chart.

As arranged in the collection the charts provide three scales: (1) radial, or from one chart to the next in hue; (2) vertical in value; and (3) horizontal in chroma.

"The nomenclature for soil color consists of two complementary systems: (1) Color names, and (2) the Munsell notation of color. Neither of these alone is adequate for all purposes. The color names are employed

in all descriptions for publication and for general use. The Munsell notation is used to supplement the color names wherever greater precision is needed, as a convenient abbreviation in field descriptions, for expression of the specific relations between colors, and for statistical treatment of color data. The Munsell notation is especially useful for international correlation, since no translation of color names is needed. The names for soil colors are common terms now so defined as to obtain uniformity and yet accord, as nearly as possible, with past usage by soil scientists. Bizarre names like 'rusty brown,' 'mouse gray,' 'lemon yellow,' and 'chocolate brown' should never be used."

The soil color names and their limits are given in the diagrams which appear opposite each chart.

"The Munsell notation for color consists of separate notations for hue, value, and chroma, which are combined in that order to form the color designation. The symbol for hue is the letter abbreviation of the color of the rainbow (R for red, YR for yellow-red, Y for yellow) preceded by numbers from 0 to 10. Within each letter range, the hue becomes more yellow and less red as the numbers increase. The middle of the letter range is at 5; the zero point coincides with the 10 point of the next redder hue. Thus 5YR is in the middle of the yellow-red hue, which extends from 10R (zero YR) to 10YR (zero Y)."

"The notation for value consists of numbers from 0, for absolute black, to 10, for absolute white. Thus a color of value 5/ is visually midway between absolute white and absolute black. One of value 6/ is slightly less dark, 60 percent of the way from black to white, and midway between values of 5/ and 7/."

"The notation for chroma consists of numbers beginning at 0 for neutral grays and increasing at equal intervals to a maximum of about 20, which is never really approached in soil. For absolute achromatic colors (pure grays, white, and black), which have zero chroma and no hue, the letter N (neutral) takes the place of a hue designation."

"In writing the Munsell notation, the order is hue, value, chroma with a space between the hue letter and the succeeding value number, and a virgule between the two numbers for value and chroma. If expression beyond the whole numbers is desired, decimals are always used. never fractions. Thus the notation for a color of hue 5YR, value 5, chroma 6, is 5YR 5/6, a yellowish-red. The notation for a color midway between the 5YR 5/6 and 5YR 6/6 chips is 5YR 5.5/6; for one midway between 2.5YR 5/6 and 5YR 6/8, it is at 3.75YR 5.5/7. The notation is decimal and capable of expressing any degree of refinement desired. Since color determinations cannot be made precisely in the field—generally no closer than half the interval between colors in the chart —expression of color should ordinarily be to the nearest color chip."

"In using the color charts, accurate comparison is obtained by holding the soil sample directly behind the apertures separating the closest matching color chips. Rarely will the color of the sample be perfectly matched by any color in the chart. The probability of having a perfect matching of the sample color is less than one in one hundred. It should be evident, however, which colors the sample lies between, and which is the closest match. The principal difficulties encountered in using the Soil Color Chart are (1) in selecting the appropriate hue card, (2) in determining colors that are intermediate between the hues in the chart and (3) in distinguishing between value and chroma where chromas are strong. In addition, the chart does not include some extreme dark, strong (low value, high chroma) colors occasionally encountered in moist soils. With experience, these extreme colors lying outside the range of the chart can be estimated."

The use of a mask will facilitate color matching. Two masks are included in the binder pocket: a black mask for use with dark samples and a gray mask for intermediate and light samples. The aperture in the mask allows exposure of four adjacent color chips; the remaining colors in the field are covered to eliminate the possibility of disturbing contrasts.

Attachment SOP-VHB-036-002

CONTRAST OF MOTTLES

Identification Table

Hue	Value	Chroma	Contrast
Same	Same	1 unit	Faint
	Same	2	Distinct
	Same	3	Prominent
	Same	4 or more	Prominent
	1 unit	Same	Faint
	1	1 unit	Faint
	1	2	Distinct
	1	3	Distinct
	2	Same	Faint
	2	1	Faint
	2	2	Distinct
	2	3	Distinct
	3	Same	Distinct
	3	2	Distinct
	3	3	Distinct
	4 or more	--	Prominent
2.5 units	Same	Same	Faint
	Same	1 unit	Distinct
	Same	2 or more	Prominent
	1 unit	Same	Distinct
	1	1	Distinct
	2	Same	Distinct
	2	1	Distinct
	3 or more	--	Prominent
5 units or more	--	--	Prominent

Attachment SOP-VHB-036-003

SOIL COMPONENTS AND FRACTIONS[1]

and

SOIL DESCRIPTION FORMAT[1]

TABLE I
SOIL COMPONENTS AND FRACTIONS

	SOIL COMPONENT	DEFINITION	FRACTIONS	SIEVE LIMITS* (Grain Size Range) UPPER	SIEVE LIMITS* (Grain Size Range) LOWER	FAMILIAR EXAMPLES
GRANULAR SOILS	Boulders	Materials too large to pass through an opening 8-in. square.	———	———	8-in.	Basketball
	Cobbles	Materials passing through an 8-in. square opening and retained on the 3-in. sieve.	———	8-in.	3-in.	Softball, baseball
	Gravel	Material passing the 3-in. sieve and retained on the No. 4 sieve.	coarse	3-in.	3-in.	Grape to baseball
			fine	3/4-in.	No.4(4.76mm)	Pea to grape
	Sand	Materials passing the No. 4 sieve and retained on the No. 200 sieve.	coarse	No.4(4.76mm)	No.10(2.0mm)	Road Salt
			medium	No.10(2.0mm)	No.40(0.42mm)	Sugar-table salt
			fine	No.40(0.42mm)	No.200(0.074mm)	finer than table salt
COHESIVE SOILS	Silt	Material passing the No.200 sieve that is nonplastic or very slightly plastic and exhibits little or no strength when air dried.	———	No.200 (0.074mm)	———	flour or powdered sugar
	Clay	Material passing the No.200 sieve that can be made to exhibit plasticity within a range of water contents and which exhibits considerable strength when air dried.				

* U.S. Bureau of Standards

SOIL DESCRIPTION FORMAT

Component	Identifying Term or Format	Defining Range of Percentage by Weight
Principal	Upper case letters	Generally > 50%
Secondary Component	Use adjective (ex. silty, sandy)	35% ------> 50%
Third Component	some	20% ------> 35%
Third Component	little	10% ------> 20%
Other	trace	5% ------> 10%

[1] *Hunter/BCA. Standard Operating Procedure No. 6006, Identification of Soils*

Attachment SOP-VHB-036-004

ANGULARITY OF ROCK FRAGMENTS[1]

(1) Angular: having sharp corners and edges, and therefore showing little or no effects of abrasion or wear.

(2) Subanglar: having edges and corners slightly rounded, so that wear is evident.

(3) Subround: having most of the corners and edges worn down to smooth curves, and thus showing extensive abrasion.

(4) Round: having all edges and corners smoothed off to gentle curves by prolonged wear.

[1]Source: Laboratory Manual for Hand Specimen Petrology, Department of Geology, University of Texas [Undated].

Attachment SOP-VHB-036-005

CONSISTENCE CLASSES

	Class	Description
Dry Soil:		
	dl ---	loose - usually associated with sands
	ds ---	soft - fragile
	dsh ---	slightly hard - breaks easily
	dh ---	hard - bearly breakable with fingers
	deh ---	extremely hard - can't break with fingers
Moist Soil:		
	ml ---	noncoherant
	mvfr ---	very friable - very easily broken apart
	mfr ---	friable - easily broken apart
	mfi ---	firm - resists crushing, "explode" when finally crushed
	mvfi ---	very firm - barely breakable with fingers
	mefi ---	extremely firm - can't crush with fingers
Wet Soil:		
	wso ---	nonsticky
	wss ---	slightly sticky - soil sticks to one finger when squeezed and separated
	ws ---	sticky - soil sticks to both thumb and finger, stretches slightly
	wvs ---	very sticky - soil stick strongly, resists separation of thumb and finger
	wsp ---	slightly plastic - forms wire when rolled between hands, but breaks easily
	wp ---	plastic - forms wire when rolled between hands, doesn't break easily
	wvp ---	very plastic - forms a very durable wire when rolled between hands

Attachment SOP-VHB-036-006

Form VHB-Hydro-001c
Drilling Log Input Form

Site Name:
Site Location:
Installation Date:

Boring ID/Well ID:					
Boring Depth		Depth to Water (ft BGS)		VHB Staff	
Well Depth (ft BTC)				Drilling Company	
Screen Diameter (in)		Depth Range (ft)		Sample Method	
Screen Type/Size				Reference Point (location)	
Riser Diameter (in)		Depth Range (ft)			
Riser Type/Size (ft)					
Notes					

Depth (ft bgs)	Sample Depth (ft bgs)	Recovery (ft)	Sample Description and Notes (Moisture, Color, Texture, Redox, Inclusions, Staining, Olfactory/ Visual Evidence of Contamination)	PID (PPM)	Stratum Change	Well Profile

Standard Operating Procedure for Calibration and Operation of the MiniRAE 3000 Portable Handheld VOC Photo-Ionization Detector

INTRODUCTION

A photo-ionization detector (PID) is a field instrument used to check for volatile organic vapors in air. Measurements are used for investigations and safety and health monitoring. This Standard Operating Procedure addresses use of the MiniRAE 3000 PID and other similar models. All PIDs operate on the same principle. A fan or pump pulls air into a chamber that is bathed in ultra-violet light. This light excites the electrons of the outer shell of the atoms of the organic molecule, creating positively charged ions. These ions pass through a detector that measures the energy level of the gas. The more organic molecules present, the more ions, and the more energy detected. In most cases, the reading is displayed in units of parts per million or parts per billion by volume (ppmV or ppbV).

PID have individual limitations regarding linear detection range and field. The ability to detect a chemical depends its ionization potential (IP). The IP of the target chemical must be compared to the energy generated by the ultra-violet (UV) lamp of the instrument. PIDs will typically detect compounds with IPs lower than the energy of the PID lamp. The energy of lamps available are 9.8, 10.6, and 11.7 eV (electron volts). In most cases a 10.6 eV lamp will be adequate and will detect most petroleum and chlorinated VOCs. Some specific compounds (such as acetylene, methanol, formaldehyde, and others) have and IP that is greater than 10.6 and won't be detected by that lamp. Prior to initiation of each project the user should determine what the VOCs of concern are and select the appropriate lamp. Lamp selection guidance may be found in the MiniRAE user manual.

EQUIPMENT AND SUPPLIES

- MiniRAE 3000 equipped with appropriate gas discharge lamp
- 100 ppm Isobutylene span gas or other calibration gas canister with a 500 cubic centimeter per minute (cc/min) flow-limiting regulator, or a >500 cc/min regulator and a T-connection that allows excess flow to escape, or a Tedlar bag
- Instrument logbook
- Field calibration forms
- External filter; i.e. moisture trap

PROCEDURE

A. General Considerations

Weather: The MiniRAE 3000 has limited function capabilities in damp and extreme cold conditions. Detector specifications include a temperature range of -4 to 113º F and a humidity range from 0 to 95%. In atmospheres outside of these ranges, the instrument may produce erroneous readings. Care should be taken to avoid exposure to damp atmospheres, if possible. In damp conditions the moisture trap should be inserted onto the probe tip to prevent moisture from entering the unit. Operating the MiniRAE 3000 with the moisture trap delays the reading because longer retention times are required. The extra time required will depend upon a number of variables and cannot be quantified.

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Extreme cold can stop the fan. Keep the MiniRAE 3000 in a dry, warm, secure station whenever the instrument is not in use.

Personnel Safety: The MiniRAE 3000 is Intrinsically Safe for use in Class I, Division I Groups A, B, C, D (i.e., environments where the following types of explosive gases, vapors, and liquids are present at all times: acetylene, hydrogen, ether, and hydrocarbons/fuels/solvents). **DO NOT CHARGE THE INSTRUMENT OR CHANGE THE BATTERY IN EXPLOSIVE ENVIRONMENTS.** The MiniRAE 3000 will not indicate low oxygen atmospheres or the presence of explosive gases that cannot be ionized and is not to be used in place of a Multi-gas Indicator for detection of potentially dangerous atmospheres..

B. MiniRAE 3000 Photo-Ionization Detector Operation, Maintenance, and Calibration

The instruction manual kept with the MiniRae 3000 details how to operate, maintain, and calibrate the instrument. Read the manual before operating the unit.. The general guidelines for using the instrument are discussed below.

B.1. Instrument Check

1. Check the operational status of the MiniRAE 3000 while in the office, before mobilizing to the field site. The RAE systems logo should appear after pressing and holding the [MODE] key. Once start-up and self-test procedures are complete, the instrument will show a numerical reading with icons on the display screen. A calibration icon will display if calibration is required either due to a lamp change, sensor replacement, change of calibration gas type, or if more than 30 days have elapsed since the last calibration. Note that many project-specific work plans and/or site-specific safety and health plans (SSHPs) mandate daily, or sometimes twice-daily, calibration.
2. Press the flashlight key to ensure that the built-in flashlight turns on.
3. Hold a xylene-containing marker, such as a Sharpie®, in front of the detection probe. The instrument is operating if there is a visible sensor reading.
4. The MiniRAE 3000 may have residual contamination from previous use. Draw the Sharpie® marker away from the detection probe and check the MiniRAE 3000 screen. The screen should return to "0.00 ppm" or a reasonable background concentration (0.00 ppm to 1.5 ppm) without fluctuations. If the display fluctuates between "0.00 ppm" and a reading larger than a typical background level the lamp may need cleaning. Refer to the instruction manual cleaning instructions.
5. Perform a calibration check sequence with the span gas. With the MiniRAE 3000 on, connect the instrument to the span gas cylinder or a Tedlar bag filled with span gas. If the response is +/- 2%, calibration may not be needed. Record this procedure and the results in your field book along with the background reading. If calibration is required, follow the steps provided in B.2, below.
6. Press and hold the [MODE] key for five seconds to turn the lamp and pump off.

B.2. Standard Two-Point Calibration Procedure – Perform at "background location" on the subject site (e.g., upwind, in a dry area, away from equipment operating at the site).

A two-point calibration uses a zero ("fresh") air and a span gas of known concentration. "Fresh" air is clean, dry air without organic impurities and an oxygen value of 20.9 percent. The zero calibration can be bypassed by selecting "Span Calib" in the calibration menu. The default span gas is 100 ppm isobutylene (isobutene).

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1. Press and hold the [MODE] and the [N/-] buttons until the Password screen appears (no password is required). Press [Mode] to enter calibration.
2. Press [Y/+] to begin zero (fresh air) calibration, selecting the highlighted "Zero Calib".
3. Press [Y/+] to start the calibration process; the message "Please apply zero gas" is displayed.
4. Connect the instrument to a "fresh" air source such as a zero gas cylinder or carbon filtration cartridge, or place the instrument in a background location at the site to measure ambient air.
5. Turn on the zero gas if necessary, press [Y/+] to start calibration.
6. Zero calibration occurs over a 30-second period; the message "Zeroing…" is displayed until zero calibration is complete. The message "Zeroing is done! Reading = 0.0 ppm" will be displayed.
7. The screen will return to the calibration menu; press [Y/+] to enter Span Calibration. The name of the default span gas, "Isobutene Span = 100 ppm", will appear.
8. Connect the instrument to a cylinder of the span gas with a 500 cc/min flow-limiting regulator, to a Tedlar bag filled with the gas, or to a >500 cc/min regulator attached to tubing with a T-connection that will allow excess gas to escape.
9. Press [Y/+] to initiate span gas calibration. Calibration occurs over a 30-second period; the message "Calibrating…" will be displayed until span gas calibration is complete. The message "Span 1 is done! Reading = ###.# ppm" will then be displayed where the displayed number "###.#" should be 100.0 ppm or very close to this value.
10. Record this value on the field calibration forms.
11. Press [MODE]; the message "Updating settings…" is displayed until the screen returns to the main display and begins or resumes monitoring.

See the MiniRAE 3000 User's Guide for instructions to conduct a three-point calibration.

B.3. Operation

1. Consult the Site Specific Safety and Health Plant (SSHP), particularly the permissible vapor exposure limits, before performing any field work.
2. *ALARM SETTING*. Set the alarm (a loud buzzer and/or red flashing LED) to the action level appropriate for upgrading personal protective equipment as stated in the SSHP using the following procedure:
   a. Press the [MODE] and [N/-] keys.
   b. Press [N/-] twice to cycle to the Alarm Setting menu; press [Y/+] to select Alarm Settings. The alarm settings are High Alarm, Low Alarm, STEL Alarm, TWA Alarm, Alarm Type, and Buzzer & Light.
   c. Press the [N/-] to scroll through the Alarm Limit menu until the display shows the limit to be changed.
   d. Press [Y/+] to select the alarm type to be changed.
   e. Press [Y/+] to increase each digit's value; press [N/-] to scroll to the next digit.
   f. Press [MODE] when all digits are entered.
   g. Press [Y/+] to save the changes, or press [N/-] to undo the changes and return to the previous settings.
   h. When alarm types have been changed or bypassed, press [MODE] to return to the Programming Menu.

3. *MAXIMUM CONCENTRATION*. Peak readings are recorded when the instrument is running in Hygiene Mode (the default mode that provides basic functionality). This reading is displayed by pressing [N/-] from the main screen displaying current detections. Continuing to press [N/-] will allow the user to scroll through the other instrument

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settings and data, such as the date, time, temperature, span gas settings and lamp settings, before returning to the main screen.

4. *BATTERY INDICATOR.* When the instrument's battery is fully charged, a battery icon shows a full battery and does not flash. A fully charged battery runs the instrument for approximately 16 hours. When the battery is low, the battery icon will blink and the instrument will beep once and flash every minute to indicate that the machine should be turned off within 10 minutes. To charge, turn the instrument off by holding the [MODE] button for 5 seconds. Reconnect the instrument to its cradle and connect the cradle to a wall electrical outlet via its AC/DC adapter. The instrument will lock in place and the LED in the cradle will glow. The charging time is less than 8 hours for a fully discharged battery. All units should have a spare battery pack within the carrying case if needed.

5. HEALTH AND SAFETY MONITORING.
   a. Enter the work area from the "background location," frequently viewing the display to monitor breathing space readings.
   b. Conduct the tasks required according to the appropriate protocols (in accord with approved SSHP and/or work plans) frequently observing breathing zone readings and recording them in the job specific field book, along with the time and location of the reading.
   c. If the PID behaves erratically, periodically confirm calibration by connecting the span gas canister or filled Tedlar bag to the MiniRAE 3000 as described in Section B.2, and note the concentration reading. Write this calibration confirmation in the instrument log book and in the job specific field book.

6. FIELD SCREENING. Field screening of soils may be done directly over soil cores or excavator buckets or using a bag or jar headspace technique. Refer to Site Specific Workplans to determine the appropriate method for each Site.
   a. Direct Screening: Using and hand tool make a small indentation in the sample area. Place the PID tip approximately 1 inch above the soil surface and cover the area with a nitrile gloved hand. Monitor the PID readings and record both the range and maximum reading on the soil boring log or field logbook.
   b. Bag or Jar Headspace: Collect a representative soil sample from the soil core or other sample collection tool and place it within a clean zip top bag or clean glass jar. The soil should occupy approximately ½ the volume of the container. Seal the bag or jar closed. If using the jar method aluminum foil or laboratory parafilm should be used. After sufficient equilibration time (refer to Site Specific Workplan for equilibration time and environmental requirements) pierce the bag or jar with the PID tip being careful to not push the tip into the sample media. Monitor the readings and record on the appropriate field form or log book.

REFERENCES

Rae Systems, 2007, MiniRAE 3000 User's Guide, Revision A, accessed at http://www.raesystems.com/~raedocs/manuals/MiniRAE3000_manual_v4.pdf. November 2007.

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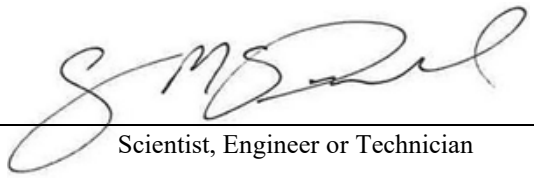
Author:  Date: 3/14/22
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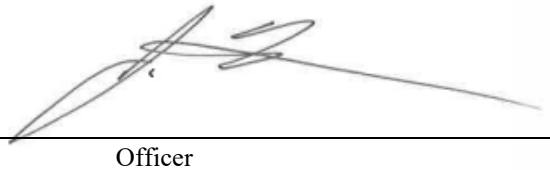
Technical Reviewer

Approved:  Date: 3/14/22
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Standard Operating Procedure for Low Stress Groundwater Sampling of Monitoring Wells and Piezometers

INTRODUCTION

A primary goal of a groundwater sampling program is to accurately assess the quality of the groundwater that occurs under the study site. To accomplish this goal, specific measures must be taken during groundwater sampling to ensure that the sample from each well is representative of the aquifer water in that locality. Measures must also be taken so that the samples are not altered or contaminated during the sampling and handling procedures. Low Stress Groundwater Sampling methods are generally more effective than bailer sampling techniques at accomplishing this goal.

For the purposes of this discussion, there are three zones of water in a well: the stagnant water in the casing, the water in the screened interval, and the formation water drawn into the well by purging. Stagnant water is unlikely to be representative of groundwater quality and should not be included in the sample. The concept of Low Stress sampling is to pump out the water in the screened interval at a low pumping rate (purging), drawing in the formation water while leaving the stagnant casing water relatively undisturbed and in place. Purging is continued until indicator parameters measured in the purge water are stable (usually accomplished after purging less than two well volumes). Dedicated tubing and pumps are preferred.

Data from previous sampling events are often helpful in order to successfully perform Low Stress Groundwater Sampling of Monitoring Wells. At a minimum these data should include:

- A description of the reference measuring point (usually the top of casing)
- The length and depth of the screen interval
- The depth from the measuring point to the sampling point (usually the center of the screen)
- Other potentially useful data are listed in the Ground Water Monitoring Well Sample Collection Record presented as Attachment VHB-SOP-053-2.

When practical, the wells should be sampled in order of increasing probable chemical concentrations beginning with the least contaminated well and ending with the most contaminated well.

Any field deviations from this SOP must be documented and technically justified in accordance with SOP-VHB-018 Standard Operating Procedure for Deviation from Protocols or Standard Operating Procedures and for Notation, Correction, and Documentation of Unforeseen Circumstances.

PROCEDURE

A. Water Level Measurement

A water level measurement should be taken before well purging (refer to SOP-VHB-009 for procedure). *Extreme care should be taken to avoid disturbing the water column in the well.* No attempt should be made to measure the total depth of the well until AFTER sampling is completed. If water levels are measured automatically with a transducer and data-logger (or other sub-aqueous method), manual measurements are still necessary before and after purging and sampling.

If volatile organic compound (VOC) contamination is suspected to be present at a new well or new site, or if the presence of groundwater-related VOCs in the breathing zone is a known risk at a characterized site, a photoionization detector (PID) measurement of the headspace of the well

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should be recorded immediately upon opening the well cap and the results used to assess potential health risks and the possible need to upgrade personal protective equipment (PPE) per the Site Health and Safety Plan. This measurement should be made with a calibrated PID at the rim of the well. Calibration and use of photoionization detectors is described in SOP-VHB-041 and SOP-VHB-044.

B. Well Purging/Sampling Equipment

There are a variety of sample withdrawal mechanisms that can be used to purge wells and collect water samples. Ideally, the sampling equipment coming in contact with the groundwater should be of a material that is completely inert to the analytes of interest. However, materials such as polyethylene, which can sorb contaminants, may be acceptable if the equipment is dedicated to a single well.

If a gasoline or diesel generator or compressor is used, it should be located a minimum of 30 feet downwind from the well head in order to prevent cross contamination by airborne exhaust constituents.

During purging and sampling, the intake of the sampling equipment (i.e., the end of the tubing or the intake of the downhole pump) should be located at the same depth for each sampling event, generally near the center of the well screen. In cases where appropriate data are available, the intake of the sampling equipment should be located at the center of primary flow zones, such as those with elevated permeability or contaminant concentrations relative to the rest of the screened interval. The intake of the sampling equipment should be at least 2 feet above the bottom of the well in order to avoid disturbing silt in the well sump. There should not be extra coils of tubing at the surface. The tubing should have as large a diameter as practical, taking into account the well recharge rate, well diameter, etc. Generally, tubing inside diameter should be 3/8 inch or less in order to ensure a continuous water column in the tubing.
The tubing should remain full of water (absent of air bubbles) during purging and sampling to avoid changes to the water chemistry by contact with the atmosphere.

Bailers are not suitable for low stress sampling. Positive displacement ("WaTerra" type) check valve pumps are also generally not suitable for low stress sampling.

Acceptable low stress sampling pumps include: down-hole electric pumps, down-hole bladder pumps, and top-of-well peristaltic pumps. Each has its own advantages and disadvantages.

1. Down-hole submersible electric pumps. Down-hole electric pumps should be dedicated to each well, as lowering the pump into the well will disturb the static water column. Grundfos and other high volume down-hole submersible electric pumps are satisfactory in large diameter wells and in aquifers with high hydraulic conductivity. For most monitoring wells these types of pumps typically cannot maintain the low flow rates required for slow purging. Low volume downhole electric pumps may be satisfactory if the components are of suitable materials. One type of down-hole submersible pump is the Grundfos Redi-Flo, which is operated with an AC power source and can pump at rates between 100 milliliters per minute (mL/min) and 190 liters per minute (L/min). Discharge rates for down-hole electric pumps shall not be controlled through the use of a valve or other type of flow-restricting device when collecting water samples for VOC analysis. In that scenario, a loss of VOCs from the water sample may occur due to the sudden decrease in pressure across the valve or other flow-restricting device.

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2. Down-hole bladder pumps. If feasible, bladder pumps should be dedicated to each well, as lowering the pump into the well will disturb the static water column. If the bladder pump is not dedicated, the pump should be lowered slowly and carefully into the wells to minimize disturbance of the water column. For aquifers where the depth to groundwater is greater than 20 feet (the approximate suction limit of suction pumps), bladder pumps often provide the best available technology for low stress sampling. The bladder pump discharge rate is commonly 50 mL/min or greater.

   One type of bladder pump is manufactured by Q.E.D. (with Teflon or dedicated, disposable polyethylene bladders; (http://www.qedenv.com/QED_Environmental_Systems), which can be installed permanently in each well for sites requiring long-term, repeated sampling, or can be decontaminated (or the bladder replaced) for use at multiple wells. A portable compressor and control box connected at the well head to the air line of the sampling pump provide the mechanism for well water evacuation. The pump operates by compressed air squeezing the bladder in the pump casing, thereby forcing the column of water in the water line up and out of the well.

   The effectiveness of the bladder pump is based on the proper placement of the pump within the well. If the bladder is not entirely submerged within the water column, the possibility of air entering the sampling train is distinct. Therefore, when installing the pumps, care must be taken to ensure that the bladder is entirely submerged.

3. Top-of-well peristaltic pumps. In many cases, peristaltic pumps provide the least expensive and most versatile option for low stress sampling. Peristaltic pumps may cause loss of volatile organic compounds due to the vacuum used to draw the water to the ground surface, although the significance of the loss is disputed among experts (Puls and Barcelona, 1995). Peristaltic pumps are limited to locations where the groundwater level is within about 20 feet of the ground surface. The lower the groundwater surface is (up to about 20 feet), the higher the potential for volatilization of contaminants during purging/sampling. Volatilization can be reduced by placing in-line sample bottle holders on the suction side of the pump.

   One type of peristaltic pump is made by Geotech (http://www.geotechenv.com/). This pump has a variable controller to adjust the rate of flow. It can be powered by a 12 volt DC battery, such as a deep cell marine battery. The peristaltic pump discharge rate is commonly 125 mL/min or greater.

C. Well Purging

Purge Rate - The purge rate should be kept consistent during each sampling event and between sampling events. The purge rate should not be greater than the rate of pumping used to initially develop the well. Purge rates should be set to maintain the water level in the well within 10 centimeters (~0.3 feet) of the normal water level measured prior to purging, if possible. If the initial purge rate draws down the water level more than 0.3 feet, the rate should be reduced, if possible. If the static water level is above the screened interval, care should be taken to avoid drawing the water level down below the top of the screened interval.

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Pumping the well dry should be avoided. If previous sampling data indicate that the well is unable to sustain low-flow purging without being pumped dry, the sampler should attempt to purge that well early in the day to maximize the likelihood of collecting a sample the same day. If the well is pumped dry using the lowest pumping rate possible, the sampler should leave the sampling equipment in the well, proceed with other wells, and return to the well later for sample collection within eight hours, if possible. If the well is still dry after allowing reasonable time to recharge, a note should be made stating why the sample was not collected. If the well has re-charged sufficiently, the sample should then be collected directly from the first water drawn from the well without waiting for parameter stabilization.

Purged well water should be collected in a graduated cylinder or bucket (after passing through the flow-through cell- see Indicator Parameters below) in order to quantify the volume of water that is evacuated. Purge time, water level, and purge rate/volume should be recorded every 3-5 minutes. Longer recording times are acceptable if purge time exceeds 30 minutes and purge rates remain constant. The volume of water to purge is determined by the stabilization of field measured indicator parameters during purging; however, the minimum final purge volume must be greater than the stabilized drawdown volume plus the extraction tubing volume (i.e., the sample must not be composed of the stagnant water stored in the well).

Indicator Parameters - Field measured indicator parameters should be measured and recorded every 3-5 minutes with a flow-through cell during purging, and include at a minimum: dissolved oxygen, specific conductance, temperature, pH, turbidity, and oxidation/reduction potential (ORP). A longer measurement cycle is acceptable if the purge time exceeds 30 minutes without stabilization. The time between readings should be long enough that the volume of water within the flow-through cell is fully exchanged at least once between readings. The flow-through cell should be located downstream from the peristaltic pump. A T-type valve should be located in-line between the pump and flow-through cell. Samples to be measured for turbidity should be collected from this T valve, before the purge water enters the cell, to prevent turbidity settling out within the flow-through cell.

Stabilization is defined as three consecutive readings collected at 3-5 minute intervals which are within the following limits:

- pH ± 0.1 unit
- Temperature ± 0.2º C
- Specific Conductance ± 3%
- ORP ± 10 millivolts
- dissolved oxygen ± 10%
- turbidity ± 10% for values > 1 NTU or less than 1 NTU.

EPA or project-specific requirements may not match these recommendations.

Flow-through cells should be clear (except for turbidity measurements), and designed to be kept full of water (without bubbles) during operation. They should not allow water to return to the well when the pump is turned off. They should not leak air, and the probes should be completely submerged. One such flow-through cell system is a YSI™ 556MPS handheld multiparameter system (see SOP-VHB-055 for calibration and use instructions). This system allows for chemical parameters to be monitored during the well purging process, within the flow-through cell.

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If indicator parameters have not stabilized after four hours of purging, proceed with sample collection as described below. Record all data and attempts to achieve stabilization, and include a comment on the chain of custody reflecting the fact that stabilization was not achieved.

D. Sample Collection

The sample is collected after the well has been purged and, if applicable, the field parameters are stable. The same equipment used for purging should be used for sampling. Sampling may be accomplished by two methods, from an in-line sample port or from an in-line sample bottle holder. An acceptable variation of the sample port method is the collection of samples directly from the discharge end of the sample tubing. The in-line bottle holder method is preferred, as the sample does not come in contact with air, thereby minimizing the potential for loss of volatile contaminants. Sampling should be accomplished from an in-line sample port or sample bottle holder (i.e., sample jig) up-stream of the flow-through cell. If a bottle holder is used for sampling, it should be located upstream of the peristaltic pump, if one is used. The rate of pumping during sampling should be the same as that used for purging. If in-line sample bottle holders are used, the pump should be shut off and the bottles should be removed one at a time. Additional water can be collected in the bottle cap if necessary to completely fill the bottles. If an in-line sample port is used, sample containers should be filled by allowing the pump discharge to flow down the side of the container with minimum turbulence. VOC samples should be collected first.

E. Sample Size and Preservation

Parameter-specific requirements are given for preservation, sample size, holding time, and analytical methodology in Attachment SOP-VHB-053-1 or in the Site-specific work plan. Alternative requirements may be necessary for specific projects.

1. Major Ions: For major ionic species, plastic bottles are used (the size will vary with the laboratory's requirements). Collect the sample by removing the cap carefully from the sample bottle, taking care not to touch the inside of the cap or bottle with the sampling tube/spigot. The sample container is filled and the bottle is capped tightly.

2. Volatile Organic Compounds (VOC): Samples shall be collected in 40 mL glass vials with screw-on caps equipped with Teflon lined septa. Ordinarily clear glass is adequate, but if the parameters of concern are typified by a high photolysis potential, brown glass should be utilized. Often the vials are pre-preserved with hydrochloric acid (HCl) to a pH of less than 2; however, if a sampling jig is used, the samples must be preserved by adding HCl to reduce the pH to less than 2. The sample vial should be filled slowly and carefully. Care must be taken to minimize turbulence. The vial shall be filled to a positive meniscus and the cap screwed on firmly. The vial should be inverted and tapped gently with a finger to check for air bubbles. There should be no bubbles in the sample vial immediately after sample collection. In some cases bubbles may form in the vial over time. Vials should be stored in the cooler with the cap downward to prevent loss if bubbles form.

3. Dissolved Metals: Metals samples will be preserved with nitric acid (HNO_3) to a pH less than 2. Generally, filtering samples for dissolved metals analysis is not necessary or desirable. If filtering is required (if the objective is to compare filtered and un-filtered samples, for instance) filtering should be performed in the field using a dedicated in-line filter with 0.45-micron aperture. Filters should be flushed with 25 to 50 milliliters of

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groundwater prior to sample collection. If filtering is performed, do not acidify samples until AFTER they are filtered.

4. Pesticides, SVOCs, and PCBs: The pesticide, SVOC, and PCB samples should be collected in clean 1 liter amber glass bottles with Teflon lined septa. The sample is collected by removing the cap from the sample bottle, taking care not to touch the inside of the cap or bottle with the sampling spigot or any other apparatus. For some pesticides, a pre-rinse with isopropyl alcohol is recommended. The sample bottle is then filled entirely, taking care not to introduce air bubbles. The bottle is tightly capped. The pesticide samples should be sealed with tape.

F. Documentation of Sample

Documentation of the sampling event will consist of logging data on the Ground Water Monitoring Well Sample Collection Record presented as Attachment VHB-SOP-053-2.

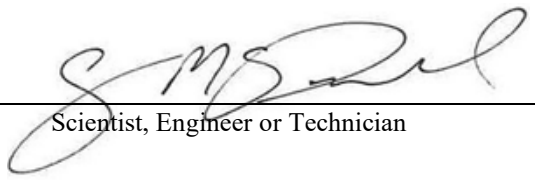
G. Transportation/Packing

Upon collection, a sample label is fixed to each bottle. The label will include data written in permanent ink. The label data will include the site name, the well name, the date and time, the type of analysis, the preservative, and the sampler's initials. The sample is then packed in an insulated cooler with ice or cold packs to maintain a temperature of 4 degrees Celsius (ºC) ± 2 ºC and delivered to the laboratory for analysis as soon as is reasonable, ensuring that hold times will not be exceeded. A completed Chain-of-Custody Form (SOP-VHB-007) will accompany the sample set.

References

ASTM D4448-01 (2013), Standard Guide for Sampling Ground-Water Monitoring Wells, November 2013.

Puls, R.W. and Barcelona, M.J., 1995, Low-Flow (Minimal Drawdown) Ground-water Sample Procedures, EPA Ground Water Issue EPA/540/S-95/504

Author:  Date: 3/14/22
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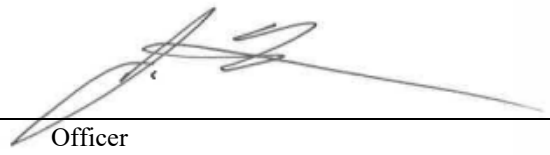
Approved:  Date: 3/14/22
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Attachment SOP-VHB-053-1

Sample Parameters, Methods, Volumes, Containers, Preservation and Holding Times
(following pages)

Source: "Water Quality Division Field Methods Manual, April 2006", Table 4.1.7. Parameter Summary Chart: Required Containers, Preservation and Holding Times. Vermont Department of Environmental Conservation

PARAMETER	SAMPLE TYPE	CONTAINER	PRESERVATION	MAXIMUM HOLD TIME	NOTE
METALS					
Cations, (calcium, magnesium, potassium, sodium)	Water	P, 125mL	HNO3 to pH < 2, filter on-site 0.45µm for dissolved	6 months	a, b
Mercury, Low Level	Water	G, 125mL, Teflon lined cap, acid rinsed or 500mL Teflon bottle	12N HCl (5mL/L) and BrCl solution (5mL/L); preserved at the laboratory in original bottle	28 days to preservation – 90 days to analysis	o
Metals, (all metals and cations)	Water	P, 250 mL	HNO_3 to pH < 2, filter 0.45µm for dissolved	28 days for Mercury, 6 months for remainder	a, b
Metals (TCLP - see footnote l)	Liquid, Sludge	P, 1000 mL or P, 2000mL (2 containers/ sample) Sample size depends on % solids. Consult Lab Supervisor.	Cool 4°C	6 months, Mercury as soon as possible	l
MICROBIOLOGY					
Coliform-Total	Water	P, 250 or 120 mL sterile	Cool, 4°C	8 hours (30 hours drinking water)	c, d, n
E. Coli	Water	P, 250 or 120 mL sterile	Cool, 4°C	8 hours (30 hours drinking water)	c, d, n
INORGANIC CHEMISTRY					
Alkalinity	Water	P, 250 mL	Cool, 4°C	14 days	
BOD Uninhibited 5-Day	Water	P, 2 L	Cool, 4°C	24 hours	c, f
Chloride	Water	P, 50 mL centrifuge tube	none required	28 days	
Chlorine	Water	G, 125mL amber	Cool, 4°C	analyze immediately	
Chloride-(Ion Chromatography)	Water	P, 50 mL centrifuge tube	Cool, 4°C, filter 0.45µm	28 days	j
Chlorophyll-a	Water	glass fiber filter, Whatman GF/A, 4.7mm, 7µm pore size stored in black jar	Freeze filter in black jar - 20 to -70°C	21 days	
COD	Water	P, 50 mL centrifuge tube	Cool, 4°C, H_2SO_4 to pH < 2	28 days	e
Conductance	Water	P, 250 mL	Cool, 4°C	28 days	
Cyanide, Total	Water	P, 2 L brown and P, 250 mL, with preservative added	Cool, 4°C, NaOH to pH >12, ascorbic acid	n	g, d, n
Dissolved Oxygen, Probe	Water	G, 300 mL D.O. bottle	none required	analyze immediately	
Dissolved Oxygen, Winkler	Water	G, 300 mL D.O. bottle	MnSO4, I-/Azide, dark	8 hours	h
Ignitability	Solid or Liquid	G, 250 mL	Cool, 4°C		
Nitrogen, Ammonia	Water	P, 250 mL	Cool, 4°C, H_2SO_4 to pH < 2	28 days	e

PARAMETER	SAMPLE TYPE	CONTAINER	PRESERVATION	MAXIMUM HOLD TIME	NOTE
Nitrogen, Nitrate (Ion Chromatography)	Water	P, 50 mL centrifuge tube	Cool, 4°C filter (0.45µm) DO NOT ACIDIFY	48 hours	j
Nitrogen, Nitrate + Nitrite	Water	P, 250 mL	Cool, 4°C, H_2SO_4 to pH < 2	28 days	e
Nitrogen, Nitrite	Water	P, 50 mL centrifuge tube	Cool 4°C, filter DO NOT ACIDIFY	48 hours	c, j
Nitrogen, Total Kjeldahl	Water	P, 250 mL	Cool 4°C, H_2SO_4 to pH < 2	28 days	e
Nitrogen, Total Persulfate	Water	P, 50 mL centrifuge tube	Cool 4°C, H_2SO_4 to pH < 2	28 days	e
pH	Water	**analyze immediately**	none required	analyze immediately.	
Phosphorus-Ortho	Water	G, 75 mL tubes	Filter immediately (0.45µm), Cool, 4°C	48 hours	c, j
Phosphorus, Total	Water	G, 75 mL tubes	none	28 days	k
Phosphorus, Total Dissolved	Water	G, 75 mL tubes	Filter immediately (0.45µm)	28 days	j, k
Silica	Water	P, 50 mL centrifuge tube	Filter immediately for dissolved (0.45µm) Cool, 4°C	28 days	
Solids - Total Dissolved	Water	P, 250 mL	Cool, 4°C	7 days	
Solids - Total Suspended	Water	P, 1 L	Cool, 4°C	7 days	
Solids-Total Volatile	Water	P, 250 mL	Cool, 4°C	7 days	
Sulfate (Ion Chromatography)	Water	P, 50 mL centrifuge tubes	Cool, 4°C, filter 0.45µm	28 days	j
Turbidity	Water	P, 250 mL	Cool, 4°C	48 hours	
ORGANICS					
Pesticides & PCBs	Water	G, 2.6 L amber, Teflon lined cap, methylene chloride rinsed	Cool, 4°C, Pesticides & PCBs need to be pH 5-9	7 days to extraction, 40 days after	d
Semi-volatiles or TPH (nonvolatile)	Water	G, 2.6 L amber, Teflon lined cap, methylene chloride rinsed	Cool, 4°C	7 days to extraction, 40 days after	d
TPH Immunoassay Screen	Water	P, 50 mL centrifuge tube	Cool, 4°C	14 days	c
Volatile Organics or TPH (volatile)	Water	G, two to three 40 mL vials per sample, Teflon lined septa. Trip blanks.	Cool, 4°C, HCl to pH <2	14 days (7 days without HCl)	i, d

G = Glass
P = Plastic (Nalgene)

NOTES:

a "Dissolved" samples or turbid samples must first be filtered on-site through a 0.45 µm membrane filter followed by acid preservation. A filter blank must be submitted with each batch of filtered samples.

b 2 mL concentrated HNO_3/1000 mL and 0.5 mL/250 mL of sample; 0.2 mL/125 mL.

c Lab needs prior notice for this parameter.

d Chlorinated samples need to be collected in sample bottle containing 0.008% $Na_2S_2O_3$ (0.0032g for 40mL; 0.16g for 2000mL; 0.208g for 2600mL). Sample bottles (250mL) for microbiology tests contain 0.25mL of 10% $Na_2S_2O_3$.

e 0.5 mL concentrated H_2SO_4/250 mL and 0.1 mL/50mL centrifuge tube.

f Hold time is from the end of sample collection period for composite samples. Maximum composite time is 24 hours.
g Test for sulfides in the field prior to preservation. Test kit and instruction provided with sample bottles. If positive for S, bring sample to lab in brown bottle for filtration. If negative, collect in bottle containing ascorbic acid and NaOH.
h Samples must be "fixed" in field with 2 mL of manganese sulfate (DO #1) and then 2 mL of iodide-azide (DO #2), stored in the dark and analyzed within 8 hours.
i No head space, vials pre-acidified with 0.4 mL 1:1 HCl. Two trip blanks must be brought into the field with each sample set.
j Samplers must filter sample through 0.45 um membrane filter. For ion chromatography, a single sample container of 50 mL is sufficient for all three anions (chloride, sulfate and nitrate).
k Samplers must fill tubes to 50 mL line.
l TCLP work requires two separate samples of the listed volume. Sample must undergo TCLP extraction within the following time periods (Table 4.1.8.)
m Sample handling procedures are described in the Air Toxic Quality Assurance Plan.
n Sampling instructions provided by lab must be followed.
o Sampling instructions outlined in the following method should be followed: Method 1669, "Method for Sampling Ambient Water for Determination of Metals at EPA Ambient Criteria Levels", U.S. Environmental Protection Agency, April 1995 with January 1996 revisions.

Source: "Water Quality Division Field Methods Manual, April 2006", Table 4.1.8. Sample Maximum Holding Times (Days). Vermont Department of Environmental Conservation

	From: Field Collection To: TCLP Extraction	**From: TCLP Extraction To: Preparative Extraction**	**From: Preparative Extraction To: Analysis**	**Total Elapsed Time (Days)**
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals except mercury	180	NA	180	360

NA = Not Applicable

Attachment SOP-VHB-053-2

Groundwater Sampling Data Form

(following two pages)

VHB, INC.
100 State Street, Suite 600
Montpelier, VT 05602 USA **www.vhb.com**

Ground Water Monitoring Well Sample Collection Record

Well ID:_____

Project Name: _____ Project #: _____

Site Location: _____ Date:_____

Weather Conditions:_____ Time On Site:_____

1. WATER LEVEL DATA: (from TOC)

Description of measuring point (MP)_____ Depth to water below MP (ft):_____

Total well depth (ft):_____ Well Dia.(in):_____ Length of water column in well (ft):_____

Gallons per foot[1]:_____ Well volume (gal):_____

2. PURGING DATA: Method:_____ Stabilized intake depth (ft):_____

Purge Volume @_____ well volumes:_____ (gal) Purge Rate:_____ (mL/min)

Parameter equipment:_____

Time	Depth (ft.)	volume removed (liters)	Flow Rate (mL/min)	Temp (C)	Spec Cond. (uS/cm) @25ºC	Dissolved Oxygen (mg/L)	pH. (Std)	ORP (mV)	Turb. (NTU)

Project Number:_____ Well Name:_____ Date:_____ Sampler's Initials: _____

Purge Water Disposal Method _____ Comments (e.g. color, odor):_____

3. SAMPLE COLLECTION: Method:_____ Sample Time:_____

Sample Matrix	Chain-of-Custody#	Shipper ID#	Container Qty/type	Preservation	Analytical method/Lab

[1]well volumes for various diameters in gal./ft.

0.50" = 0.01	0.75" = 0.023	1.00" = 0.041	1.25" = 0.064	1.50" = 0.09
2.00" = 0.16	3.00" = 0.32	3.50" = 0.50	4.00" = 0.65	6.00" = 1.47

Comments:

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Time Off Site_____

STANDARD OPERATING PROCEDURE
FOR CALIBRATION AND USE OF THE YSI MODEL 556 MPS/Pro Quatro

The YSI 556 MPS is used for monitoring field parameters in groundwater and surface water. The 556 MPS is being phased out by YSI and replaced by the Pro Quatro Model over time. Both units are very similar in construction and use and this SOP applies to both. It is commonly used during Low Flow/Low Stress Groundwater Sampling (as discussed in SOP–VHB-053 ("*Standard Operating Procedure for Low Stress Groundwater Sampling of Monitoring Wells and Piezometers*"). This SOP is designed to meet the requirements outlined in The U.S. Environmental Protection Agency – Region 1 "*Standard Operating Procedure for Calibration of Field Instruments*" dated June 3, 1998 and most recently revised March 23, 2017. Different states, regulatory districts and/or agencies may have their own required calibration procedures. Therefore, it is important to review the project specific workplan and/or applicable regulatory standards to ensure this SOP meets the criteria for each specific project.

The YSI 556 MPS is a multi-parameter data sonde used to track field parameters in water. The YSI 556 MPS is equipped with the following sensors: pH; Temperature; Specific Conductance; Oxidation-Reduction (Redox) Potential; and Dissolved Oxygen.

PROCEDURE:

Prior to taking the YSI into the field, check the calibration solutions to ensure they have not passed the expiration date(s). Replacement calibration solutions are located in the equipment storage room. Ideally, the temperature of the calibration solutions should be kept as close as possible to the ambient water temperature that is being measured. However, given the variability of field conditions this may not always be possible. Calibrations should not be performed using solutions of extreme temperature (less than 0ºC or greater than 40ºC). The 556 MPS is powered by 4 C-cell batteries which are accessed by loosening the four screws on the back of the handset. Prior to calibration, turn on the instrument and note the battery life; this is located in the lower right corner of the screen. If the battery life in the instrument is low, change the batteries prior to calibrating. Remove the storage cup from the sonde and check each of the sensors for dirt, scale, and/or biological fouling. Clean any dirty sensors with a cotton swab and mild detergent solution (such as Alconox®). Refer to the YSI 556 MPS User's Manual for more information on sensor cleaning and storage. A copy of the manual is included with each YSI field kit and is also available on the internet at www.ysi.com. Some of the calibration standards may contain hazardous constituents. Always wear appropriate personal protective equipment (nitrile gloves and safety glasses) during calibration. Copies of the MSDS sheets for each calibration standard are kept at the office.

Equipment Check List:

- YSI 556 MPS (handset and sonde)
- YSI calibration form (attached)
- YSI calibration cup
- Calibration kit which contains: pH standard buffer solutions (pH4.01, 7.00, and 10.00), specific conductivity standard (1.413 ms/cm), oxidation/reduction potential solution (Zobell), zero dissolved oxygen solution, and thermometer traceable to National Institute for Standards and Technology (NIST).
- Distilled or deionized water for rinsing the probe between solutions and tap water for probe storage
- Paper towels
- Nitrile gloves and safety glasses
- YSI Flow-through cell

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CALIBRATION PROCEDURES

A. Temperature

Calibration of the temperature sensor is completed at the factory; however, the accuracy of the sensor should be periodically checked. This check should be performed at least once per year, and documentation is kept with each calibration kit. If documentation of the latest temperature check can not be located, or if it was performed more than one year ago, perform the following temperature verification utilizing the NIST traceable thermometer included with the calibration kit.

1. Fill a container with water and add ice to make a cold water bath.
2. Place both the NIST thermometer and the YSI sonde into the ice bath and wait for the temperature readings to stabilize (usually 1 to 2 minutes).
3. Compare the two readings. They should be within ± 0.2 ºC. Record the readings on the calibration sheet. If the readings are not within ± 0.2 ºC, the temperature sensor may not be working correctly and needs to be replaced. Consult the User's Manual and/or contact YSI customer service for further information regarding a faulty temperature sensor.
4. Prepare a second water bath, this time using warm water, and repeat steps 2 and 3.

B. Specific Conductance

Conductance is a measure of a liquid's ability to conduct an electrical current. The YSI should be set to display specific conductance (unless otherwise required by the project specific workplan), which is the conductivity value corrected to 25ºC. Specific conductance is usually calibrated to a single standard (generally 1.413 ms/cm) which ideally is close to the specific conductance of the water being sampled. If the sampler has prior knowledge that the sampled water will have significantly higher or lower specific conductance, a reagent which more closely matches the Site's conditions should be used. If specific conductance is a critical parameter at a given Site, it is recommended that a second standard solution be used to perform a linearity check of the calibration. The two standards should be far enough apart to bracket the Site water's specific conductance.

1. Rinse the sonde with distilled or de-ionized water. Gently shake to remove any excess water and pat the sensors dry using paper towels.
2. Immerse the sonde in the specific conductance standard.
3. From the calibration menu, select conductivity, then specific conductance using the arrow keys and the enter button.
4. Enter the value of the conductivity standard (usually 1.413 ms/cm) and press enter.
5. Wait for the specific conductance readings to stabilize (usually 1 to 2 minutes), then press enter. The word "calibrated" will be displayed at the top of the screen, and the specific conductance value will change to match (or nearly match) the calibration standard. Record the post calibration specific conductance value on the calibration form. If the "out of range, accept anyway?" message appears, the conductivity standard may need to be replaced, or the conductivity sensor may need to be replaced. If new conductivity standard is available, try to recalibrate, otherwise note the calibration error on the calibration form and move on to the next parameter.

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C. pH

PH is determined electronically using a glass electrode. Typically a three point calibration is performed using buffer solutions of 4.01, 7.00, and 10.00. On some Sites a two point calibration may be acceptable.

1. Rinse the sonde with distilled or de-ionized water. Gently shake to remove any excess water and pat the sensors dry using paper towels.
2. Immerse the sonde in the first calibration standard (e.g., pH 7.00). From the calibration menu select pH, then select 3-point calibration, then enter the value of the first pH standard and press enter.
3. Wait for the pH and temperature readings to stabilize (usually 1 to 2 minutes), then press enter. Wording on the upper portion of the screen will change from "pH calibration" to "calibrated" and the pH value will change to match the value of the pH standard. The pH millivolts reading should be noted as well during calibration with the pH 7.00 standard. If the pH millivolts value is less than -70 or greater than 70, the pH electrode needs to be replaced. Contact VHB's Equipment Manager if this is the case. Note the calibrated pH value on the calibration form and move on to the next standard by pressing enter. If the "out of range, accept anyway?" message appears, the pH electrode may need to be cleaned, the pH standard may need to be replaced, or the pH electrode may need to be replaced. Refer to the instrument use section of this document for cleaning instructions. If new pH standard is available, try to re-calibrate with the new standard. If not, note the error on the calibration form, move on to the next calibration standard and continue with the calibration.
   NOTE: Do not press escape after the first calibration is complete or the YSI will return to the calibration menu and the first pH calibration will have to be repeated.

4. Repeat steps 1 through 3 for the remaining pH buffer standards.

D. Oxidation/Reduction Potential (ORP)

Oxidation/reduction potential (ORP) is the electrometric difference measured in an aqueous solution between an inert indicator electrode and a reference electrode. It is measured in millivolts and varies with temperature. It is calibrated using a standard Zobell solution.

1. Rinse the sonde with distilled or de-ionized water. Gently shake the sonde to remove any excess water and pat the sensors dry using paper towels.
2. Immerse the sonde in the ORP standard. From the calibration menu select ORP, and then enter the ORP standard value which corresponds to the temperature of the solution. A chart of the standard values (Ag/AgCl 4M KCl in millivolts) vs. temperature is included with each YSI calibration kit.
3. Wait for the ORP and temperature readings to stabilize (usually 1 to 2 minutes), then press enter. The upper portion of the screen will change from "ORP calibration" to "calibrated" and the ORP value will change to match the value of the ORP standard. Note the calibrated ORP value on the calibration form. If the "out of range, accept anyway?" message appears, the ORP standard may need to be replaced, or the ORP electrode may need to be cleaned or replaced. If new ORP standard is available, try to recalibrate, otherwise note the calibration error on the calibration form and move on to the next parameter. Refer to the instrument use section of this document for sonde cleaning instructions.

E. Dissolved Oxygen (DO)

Dissolved oxygen content is measured using a diffuse membrane stretched over an electrode. The probe membrane must be periodically replaced and the electrode periodically cleaned to ensure accurate operation. Unstable, erroneous readings, and/or inability to calibrate properly, are indications that DO probe maintenance is required.

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Instructions for membrane replacement and/or electrode cleaning are provided in the YSI User's Manual and with each YSI DO kit. The YSI must be turned on for at least 20 minutes prior to calibration to polarize the DO sensor. Following calibration the dissolved oxygen sensor is checked by submersion in a zero oxygen solution.

1. Rinse the sonde with distilled or de-ionized water. Gently shake to remove any excess water and pat the sensors dry using paper towels.
2. Create a 100% water-saturated air environment by placing a wet sponge or a small amount of water in the bottom of the calibration cup. Ensure that the DO membrane is dry and **loosely** screw the calibration cup onto the sonde. The interior of the calibration cup must be vented to the atmospheric during the calibration process.
3. Allow the air in the calibration cup to become saturated with water vapor. This will take 10 to 15 minutes.
4. From the calibration menu select dissolved oxygen, then type in the barometric pressure, which is located in the lower right hand corner of the screen, and press enter. Record the barometric pressure on the calibration form.
5. Once the temperature and dissolved oxygen readings stabilize press enter to complete the calibration. The wording on the top of the screen will change from "DO calibration" to "calibrated". Record both the percent and mg/L values on the calibration form. The instrument should read 100% (± 0.2 mg/L). An oxygen solubility chart is included with each YSI kit to correlate mg/L and % DO at various temperatures and pressures. If the "out of range, accept anyway?" message appears, the DO membrane likely needs to be changed. Follow the instructions included with each DO kit to change the DO membrane and repeat the calibration process.
6. Once the calibration process is complete, press escape until the main menu is reached. Select run to view the instrument readings.
7. Open the zero oxygen solution and immerse the sonde in the solution making sure the DO probe is completely submerged.
8. Observe the dissolved oxygen and temperature readings. Once they stabilize, record the dissolved oxygen reading on the calibration form. If the instrument reads above 0.5 mg/L, the zero DO solution may be defective, or the DO sensor may need to be replaced. If new zero DO solution is available, repeat the process with the new solution, otherwise, replace the DO membrane and repeat steps 2 through 8.

INSTRUMENT USE

Once calibration is complete the instrument is ready for use. Detailed information on data logging and other capabilities of the 556 MPS may be found in the User's Manual. During use, either the flow-through cell or slotted probe guard should be used to prevent damage to the sensors. Damage to the sensors may also occur if exposed to pure solvents, oils, petroleum products, or very high concentrations of these compounds. The YSI should not be used if any of these conditions are known or suspected. Following use, the sonde should be decontaminated with a mild detergent solution (such as Alconox®) and rinsed with distilled or de-ionized water. For more thorough cleaning, the sonde may be soaked in a diluted solution of Simple Green® and water. The sonde should be stored in the calibration cup with either a wet sponge or small amount of tap water ensuring that the sensors remain moist, but are not submerged in the water.
NOTE: Do not store the sonde in distilled or de-ionized water as this may result in sensor damage.

POST CALIBRATION CHECK

A post calibration check should be performed at the end of each day to determine if any calibration drift has occurred. A post calibration check may not be required in all instances so be sure to check the project specific workplan to determine its necessity. The post calibration check is performed by placing the YSI in run mode and

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immersing the sonde in each of the calibration standards. Care must be taken to ensure the sonde is properly rinsed with distilled or de-ionized water and patted dry with paper towels in between each parameter to prevent cross contamination of the calibration solutions. The sonde should be kept in each standard until the temperature and parameter of interest have stabilized (approximately 1 to 2 minutes for each liquid standard and 10 to 15 minutes for the 100% dissolved oxygen check) prior to recording the value on the calibration form. Below are the post calibration check criteria. If the post calibration check readings fall outside of these parameters notify the Project Manager, as the collected data for that parameter may need to be qualified.

POST CALIBRATION CHECK CRITERIA

Measurement	**Post Calibration Criteria**
Dissolved Oxygen	±0.5 mg/L of 100% saturation value <0.5 mg/L for the zero oxygen solution, but not a negative value
Specific Conductance	±5% of the standard or ±10 µs/cm (whichever is greater)
pH	±0.3 pH units
ORP	±10 mv

REFERENCES:

1. U.S. EPA Region 1, 2010. Standard Operating Procedure – Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential (ORP), and turbidity, January 19, 2010.

2. Operations Manual for YSI 556 MPS

3. VHB, Inc., 2022. Standard Operating Procedure for Low Stress Sampling of Groundwater Monitoring Wells and Piezometers, 2022.

Author:_____ Date: 3/14/22
Scientist, Engineer or Technician

Reviewed:_____ Date:_____
Technical Reviewer

Approved:_____ Date: 3/14/22
Officer

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ATTACHMENT 1
YSI CALIBRATION FORM

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100 State Street, Suite 600
Montpelier, VT 05602

Phone: (802) 778-1299
www.vhb.com

YSI CALIBRATION SHEET

Job Name: **Job # :** **YSI #:** **Serial #:**

Brand of Standard		----------	YSI							YSI	YSI		
Lot #		----------	N/A							N/A	N/A		
Expiration Date		----------	N/A							N/A	N/A		
Date	Time	Initials	YSI Temp. - °C	Specific Cond. 1.413 ms/cm	Specific Cond. _____ ms/cm	pH 7.00	pH 4.01	pH 10.00	ORP-Zobell Solution (200-275mV)	Barometric Pressure (mmHg)	100% D.O. (%)	100% D.O. (mg/L)	Zero O_2 Solution (mg/L)
Calibration													
End of Day Check													
Calibration													
End of Day Check													
Calibration													
End of Day Check													
Calibration													
End of Day Check													
Calibration													
End of Day Check													
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End of Day Check													
Calibration													
End of Day Check													
Calibration													
End of Day Check													
Calibration													
End of Day Check													

NIST Certified Thermometer Check (Date/Results):_____ (must be completed at least once per year)

Notes: Calibration order is left to right on chart. The optimum pH mV range for pH7 is -70 to +70 after calibration.

APPENDIX B: FIELD FORMS

TEST BORING/MONITORING WELL LOG

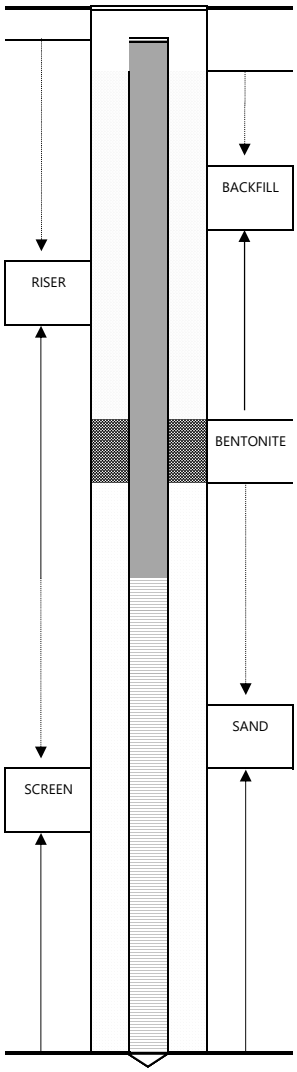
CLIENT:	
PROJECT:	
PROJECT #:	
DIGSAFE:	
DRILLER:	
DRILLING DATE:	
DRILLING METHOD:	
SAMPLING METHOD:	
PRE-CLEAR DEPTH:	
LOGGED BY:	

LOCATION ID:

ESTIMATED DEPTH TO WATER (ft.):	
TOTAL BORING DEPTH (ft.):	
BOTTOM OF WELL DEPTH (ft.):	
PVC DIAMETER, SLOT:	
RISER LENGTH (ft.):	
SCREEN LENGTH (ft.):	
FINISH:	
REFUSAL ENCOUNTERED:	

NOTES/SKETCH:

DEPTH (ft.)	LAB SAMPLE	BLOWS (per 6-in)	RECOVERY (in.)	PID (ppmV)	SOIL DESCRIPTION	WELL CONSTRUCTION (ft.)



SOIL DESCRIPTIONS:

1) PRIMARY GRAIN SIZE
(BOULDERS, COBBLES,GRAVEL, SAND (COARSE, FINE) SILT, CLAY)
2) SECONDARY GRAIN SIZE
(TRACE=0-10% LITTLE=10-20% SOME=20-35% AND=35-50%)
3) PLASTICITY (VERY HIGH, HIGH, MED., LOW, SLIGHT, NON-PLASTIC)
4) MOISTURE (WET, MOIST, DRY)
5) DENSITY (LOOSE, MEDIUM DENSE, HARD)
6) ANGULARITY
(V. ANGULAR, ANG, SUB ANG, SUB ROUNDED, ROUNDED, WELL ROUNDED)
7) COLOR (GREY, BROWN, etc.)
8) STRUCTURES, STAINING, ALTERATION
(LAMINATED, BEDDED, IRON STAINED, ETC.)
9) ODORS/ORGANIC CONTENT (PETROLEUM, SEPTIC)
10) GEOLOGICAL INTERPRETATION (I.E. FILL/TILL, GLACIAL CLAY,CHANNEL DEPOSIT, etc.)

VHB-LOW FLOW GROUNDWATER SAMPLING RECORD

PROJECT NAME _____ PROJECT# _____ WELL ID _____

LOCATION _____

SAMPLING CREW _____ DATE _____ SAMPLING TIME _____

_____ _____ _____

_____ _____ _____

PURGE DATA

REFERENCI PVC Steel Casing Ground Surface PURGING DEVICE: _____ FIELD FILTERED: Y___ N___ N/A___

DEPTH TO WATER: _____ (FT) WELL DEPTH: _____ (FT) FLOW RATE: _____

HEIGHT OF REFERENCE POINT FROM GRADE: _____ ROADBOX: _____ STANDPIPE: _____

Clock Time hr:min	Static Depth ft. (< 0.3 ft.)	Purge Rate ml/min. (100-400)	Cum. Volume Purged L	Temp. C (3%)	SP.Cond (uS/cm) (3%)	Cond. (uS/cm) (3%)	pH (s.u.) (+ -0.1 unit)	ORP/Eh (mv) (+ - 10 mv)	DO (mg/L) (10%)	Turbidity (NTU) (10%)	Water Depth (ft) (<0.33)	Flow (l/min) (<0.5)	COMMENTS

FINAL FIELD DATA

Temp. _____ (C) ORP _____ (mv) COMMENTS: _____

Conductivity _____ (uS/cm) DO _____ (mg/L) _____

pH _____ (s.u.) Turbidity _____ (NTU) _____

ODOR AND PHYSICAL APPEARANCE OF SAMPLE _____

WEATHER CONDITIONS _____

WELL CONDITION DATA

Protective Casir ` Y N Concrete pad present: Y N Cap on riser: Y N

Protective Casir ` Y N Standing Water: Y N Visible Heaving: Y N

Physical Damage: Y N

If yes, Describe: _____

SAMPLER'S SIGNATURE: _____

APPENDIX C: HEALTH & SAFETY PLAN

Health and Safety Plan Addendum

Phase III Remedial Investigation

Kennecott Mines and Mill Town Site

Kennecott, AK

PREPARED FOR

National Park Service
Wrangell-St. Elias National Park and Preserve
Kennecott, AK
EDL Number 5AKR3347

PREPARED BY

500 Southborough Drive
Suite 105b
South Portland, ME 04106

February 21, 2024

Health and Safety Plan Addendum

Phase III Remedial Investigation

Kennecott Mines and Mill Town Site

Kennecott, AK

Adopted By:	**Date:**
Jonathan Ordway, Cleanup Lead	3/18/24
Hannah Gnoza, Site Safety Officer	3/18/24

Health and Safety Plan Acknowledgment Form

I have read, understand and will abide by the procedures set forth in the August 2, 2021 Health and Safety Plan (HASP) for the Kennecott Mines and Mill Town Site and this HASP Addendum. I have also been briefed on the contents of this plan and been given sufficient opportunity to ask any questions that have arisen and have received answers which I believe are appropriate and complete which are incorporated in this HASP or its Addenda.

Printed Name	Signature	Organization	Date

1.1 Introduction

This Health and Safety Plan (HASP) Addendum for the Kennecott Mines and Mill Town Site was prepared for the Phase III Remedial Investigation (RI) scheduled for summer 2024. Because activities to be conducted during the Phase III RI are mostly described in the August 2, 2021 Phase I RI HASP, a new HASP has not been developed and the safety procedures outlined in the 2021 HASP will be followed. The Phase III RI scope of work, job tasks, additional Phase III RI training requirements, and current training certificates for the Phase III RI field staff are summarized below.

1.2 Scope of Work

The Phase III RI will include the following activities:

- The collection of surficial soil samples to estimate the concentrations of Contaminants of Potential Concern (COPCs) in surface soil that represent a reasonable estimate of exposure to human and ecological receptors at residential properties within the Mill Town.
- The collection of surface water and sediment samples from National Creek to evaluate if concentrations of COPCs exceed PALs and what concentrations of COPCs are a reasonable estimate of the exposure to human and ecological receptors.
- Identification of nearby groundwater seeps and tailings deposits near National Creek for assessing how Site-related contaminants may migrate to and impact surface water and sediment quality.
- The installation of monitoring wells and collection of groundwater samples to assess if COPC concentrations in groundwater in the vicinity of the Mill Town are greater than PALs and estimate the horizontal and vertical extent of groundwater with COPC concentrations that exceed PALs.
- The drilling of soil borings and collection of subsurface soil samples to assess if COPC concentrations in subsurface soil within the vicinity of the Mill Town are greater than PALs and estimate the horizontal and vertical extent of subsurface soils with COPC concentrations that exceed PALs.

One VHB team will perform and oversee the Phase III RI field work. The team will consist of four VHB personnel that will complete two 16-day shifts (including mobilization and demobilization).

1.3 VHB Site Activities

Table 1 in the 2021 HASP identifies tasks completed during the Phase I RI for which safety protocols were established in the 2021 HASP. The Job Tasks identified in the 2021 HASP that will be conducted during the Phase III RI include:

- Job Task 6: surface water and sediment sample collection. Six new surface water and sediment sampling locations are proposed for the Phase III RI sampling program as part of Data Quality Objective (DQO) 9: Background Study.
- Job Task 7: equipment and personnel decontamination.

In addition to the tasks outlined above, the Phase III RI field program will also include the following tasks:

Table 1 – Job Tasks

Job Task #	Activity Description
8	DQO 2/3 Surface Soil Investigation – Incremental sampling methodology (ISM) soil sampling in the vicinity of the Silk Stocking Cottages.
9	DQO 7: Groundwater Investigation – Installation of monitoring wells and groundwater sample collection around the Mill Town.
10	DQO 8: Subsurface Soil Investigation – Drilling of subsurface soil borings and subsurface soil sample collection around the Mill Town.
11	DQO 9: Background Study – Discrete and ISM soil sample collection at four sampling areas believed to not be impacted by mining and milling operations.

1.4 Other Hazard Information

Heavy Equipment

Working around heavy equipment presents hazards such as physical injury and hearing loss. Heavy equipment that may operate at the Site includes a drill rig. Before drilling begins, drilling locations will be cleared for the presence of underground utilities such as gas, electricity, and water by a private utility locator. The location of overhead power lines will be identified, and equipment will be set up a safe distance from the utility lines. Site specific practices to reduce heavy equipment hazards include:

- Wearing high visibility vests, steel-toe boots, hard hats, and safety glasses
- Using hearing protection in the vicinity of operating heavy equipment
- When approaching equipment, make eye contact with the operator before entering the swing radius of equipment or passing in front of or behind running equipment

UTV Safety

A Utility Vehicle (UTV) will be used during the Phase III RI to facilitate movement of field equipment and personnel around the Site. Two or more VHB field personnel will complete UTV safety training before operating the UTVs. VHB personnel who do not receive the training will not operate or use the UTVs.

1.5 Accident Reporting

This HASP Addendum clarifies that VHB's Incident Response Team (reachable via the Incident Reporting Hotline) will report any work-related or suspected work-related fatalities or inpatient hospitalization of three or more workers within 8 hours by telephone to OSHA at 1-800-321-6742.

1.6 Current Training Certificates and Qualifications

Personnel Verification of Clearance/Credentials

Employee Name	Training Certifications (e.g., OSHA HAZWOPER, First Aid/CPR, etc.) (Type, Date)	Issues/Concerns
Adam Robtoy	40-Hour HAZWOPER Training for General Site Workers, April 10, 2000 8-Hour Hazardous Waste Operations Refresher, March 13, 2024 Ionizing Radiation Hazards Training, June 21, 2023 Adult First Aid/CPR/AED with Anaphylaxis and Epinephrine Auto-Injector Administration, November 4, 2021 OSHA – OHV (UTV, ATV), June 29, 2023	
Benjamin Bliss	OSHA 40 Hour HAZWOPER Training, May 28, 2019 8 Hour HAZWOPER Refresher Training, February 19, 2023 Ionizing Radiation Hazards Training, June 19, 2023 OSHA – OHV (UTV, ATV), June 29, 2023	
Eric Sirkovich	OSHA 40 Hour HAZWOPER Training, 2021 8 Hour HAZWOPER Refresher Training, March 5, 2024 Ionizing Radiation Hazards Training, July 3, 2023 Adult First Aid/CPR/AED with Anaphylaxis and Epinephrine Auto-Injector Administration, August 15, 2023 OSHA – OHV (UTV, ATV), July 3, 2023	
Hannah Gnoza	OSHA 40 Hour HAZWOPER Training, May 11, 2020 8 Hour HAZWOPER Supervisor Refresher Training, April 14, 2023 Ionizing Radiation Hazards Training, June 15, 2023	

Health and Safety Plan

Phase I Remedial Investigation

Kennecott Mines and Mill Town Site

Kennecott, AK

PREPARED FOR

National Park Service
Wrangell-St. Elias National Park and Preserve
Kennecott, AK
EDL Number 5AKR3347

PREPARED BY

500 Southborough Drive
Suite 105b
South Portland, ME 04106

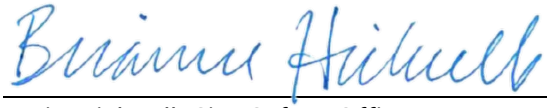
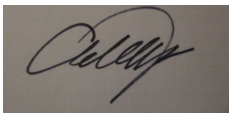
August 2, 2021

Health and Safety Plan

Phase I Remedial Investigation

Kennecott Mines and Mill Town Site

Kennecott, AK

Adopted By:	**Date:**
	8/2/2021
Tracy Planinsek, Project Manager/Cleanup Lead	
	8/2/2021
Brie Hicknell, Site Safety Officer	
	8/4/2021
Adam Robtoy, Site Safety Officer	
	8/2/2021
Ben DeJong, Corporate Health & Safety Representative	

Health and Safety Plan Acknowledgment Form

I have read, understand and will abide by the procedures set forth in this Health and Safety Plan (HASP) and any amendments thereto. I have also been briefed on the contents of this plan and been given sufficient opportunity to ask any questions that have arisen and have received answers which I believe are appropriate and complete which are incorporated in this HASP or its Amendments.

Printed Name	Signature	Organization	Date

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List of Acronyms

ANSI	American National Standards Institute
ATSDR	Agency for Toxic Substances & Disease Registry
CDC	Centers for Disease Control and Prevention
CHSR	Corporate Health and Safety Representative
CPR	Cardiopulmonary Resuscitation
DQO	Data Quality Objective
GFCI	Ground-Fault Circuit Interrupter
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
ISM	Incremental Sampling Methodology
NIOSH	National Institute for Occupational Safety & Health
NPS	National Park Service
OSHA	Occupational Safety and Health Administration
PCOPC	Preliminary Contaminants of Potential Concern
PPE	Personal Protective Equipment
RI	Remedial Investigation
SOP	Standard Operating Procedure
SSO	Site Safety Officer
XRF	X-ray Fluorescence

1

Introduction

This Health and Safety Plan (HASP) was developed by VHB for use for the Phase I Remedial Investigation (RI) at the Kennecott Mines and Mill Town Site in Kennecott, Alaska (the Site). The Site is located within Wrangell-St. Elias National Park and Preserve (WRST). This HASP describes the health and safety guidelines that will be implemented during this work to protect on-Site personnel, visitors, and the public from physical harm and exposure to hazardous materials.

The information in this HASP will be revised, as needed, when new information is received or conditions change. Every effort has been made to address the physical and chemical hazards that may be encountered during the implementation of the proposed RI. Should changes arise in the Site conditions or scope of work, or if new information becomes available regarding potential Site hazards, an addendum to the HASP may be issued. HASP addenda must be reviewed and approved by the Corporate Safety and Health Representative (CSHR). Sign-off forms will accompany each addendum and must be signed by all personnel covered by the addendum.

1.1 Background Information

The Kennecott Mines and Mill Town were located, developed, operated, and closed during the first four decades of the twentieth century (1900 to 1938). The initial discovery of the Kennicott copper deposits were made in 1900 on Bonanza Ridge. The Kennecott Mill Town was built in 1907 by the Kennecott Copper Corporation to receive, concentrate, store, and transport the ore mined from Bonanza, Jumbo, Erie, and Glacier Mines. Mill Town processing of ore and shipment of processed ore began in 1911. A tramway transported material from the mines to the Mill Town, and the railroad was used to transport the concentrated ore from the mill to Cordova, Alaska (Gilbert et al., 2001).

Substantial and sophisticated facilities were developed around the mining operation to advance the work and support the workforce. The largest facilities were the local Power Plant, a large gravity concentrator plant, and an ammonia Leaching and Floating Plant, all of which supported the milling operation. Machine shops, repair shops, administrative offices, material stores, large bunk houses, single staff housing, and private residences for management were developed around the main milling facilities (Gilbert et al., 2001).

Copper production declined in the 1930s, and the mines were temporarily shut down between 1932 and 1935. Low prices for copper and the depletion of the ore body finally caused the closure of the mines in 1938 and abandonment of the town (Gilbert et al., 2001).

In 1965, the Consolidated Wrangell Mining Company acquired rights to mine copper from surface deposits below the Bonanza Mine. The company installed a bunkhouse and a heavy media separation plant, but the operation proved unsuccessful and the plant was dismantled

and removed. Two years later, the company installed a more traditional mechanical separation unit and again attempted operations for a brief time. Most of the milling machinery was left behind at the closure in the mid-1970s and still remains, scattered throughout the Mill Town (Gilbert et al., 2001).

The land rights were eventually acquired by the Great Kennicott Land Company in 1976. The land was subdivided for sale to the public and within the Mill Town, almost every building was designated its own lot. During this time of ownership, several renovations and demolitions occurred (Gilbert et al., 2001). Although the WRST was created in 1980 by the United States Congress, the Kennecott Mill Town was not acquired by the Federal Government until 1998. Preservation measures have been taken by the National Park Service (NPS) and Friends of Kennicott to draw greater visitor numbers throughout the years (Gilbert et al., 2001; NPS, 1996).

1.2 Nature and Extent of Contamination

Known types of waste generated as a result of Site operation include lead- and arsenic-based paint, mill tailings, blasting caps, boiler ash, asbestos-containing material, and petroleum-related waste from spills. The groups of preliminary contaminants of potential concern (PCOPCs) identified for this field investigation include metals, volatile organic compounds, semivolatile organic compounds, polychlorinated biphenyls, asbestos, and explosives. These types of PCOPCs were detected during previous sampling efforts at the Site and/or cannot be conclusively excluded as PCOPCs using existing data.

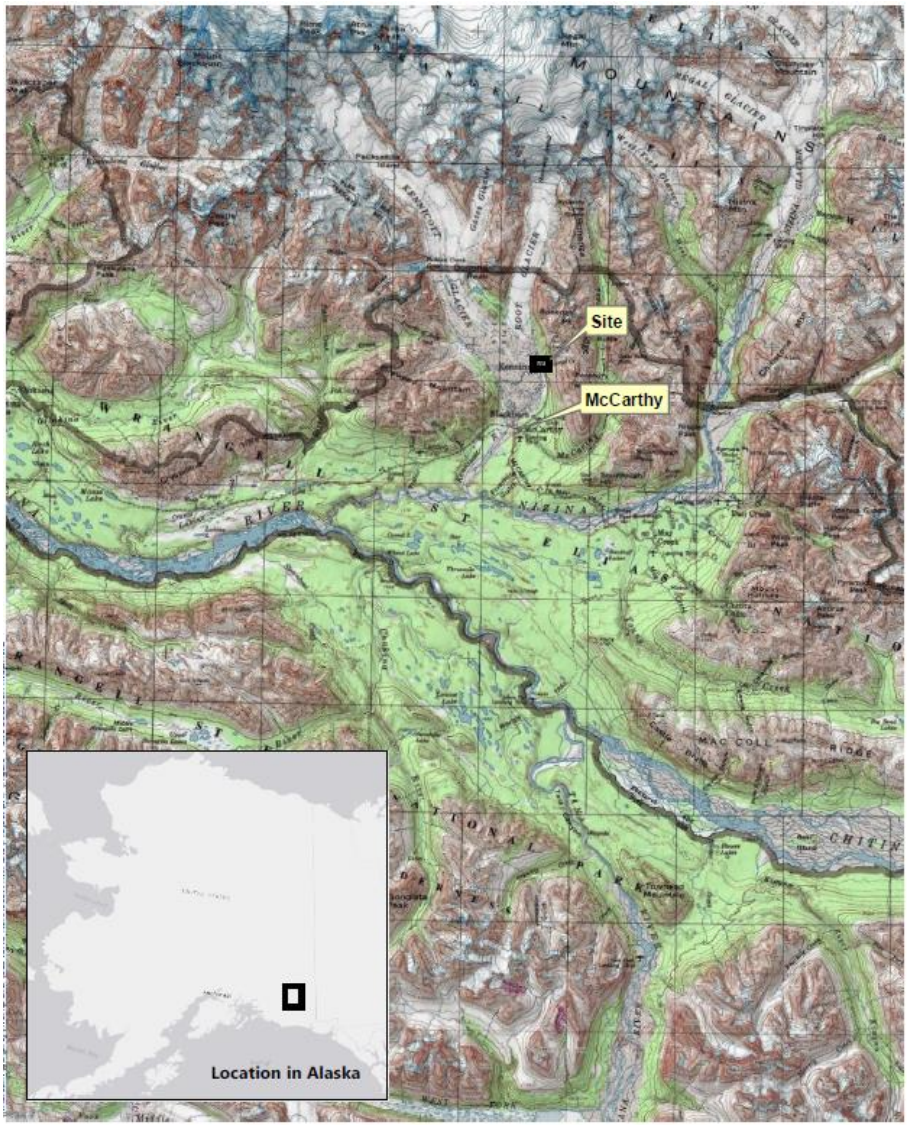
1.3 Site Location Map

The Site is located in the 13.2 million acre WRST in the southcentral portion of Alaska, approximately 100 miles from the Pacific coast, and 230 miles from Anchorage (see Figure 1). The Mill Town can be found on a valley wall near the confluence of the Root and Kennicott Glaciers, about 3.5 miles north of the town of McCarthy.

Site Location:

- Site Entrance is located at coordinates 61.485167, -142.888722, McCarthy Road, Kennecott, Alaska 99566.
- Government employee, contractor, and local community vehicle access via private toll bridge (permit required).

Figure 1: Site Location Map



2

Site Activities

2.1 Scope of Work

The goal of the Site-wide RI is to provide sufficient data to characterize the nature and extent of contamination in environmental media and determine if concentrations are present at levels that pose an unacceptable risk to human and ecological receptors. The Phase I RI will address the following data gaps for the Mill Town portion of the Site:

- Nature of PCOPCs in ambient outdoor air;
- Nature and horizontal extent of PCOPCs in surface soil;
- Extent of tailings and ore/ore concentrates;
- Nature of PCOPCs in National Creek surface water and sediment in the vicinity of the Mill Town; and
- Location(s) of groundwater seeps, springs, or storm water run-off conveyances that could act as PCOPC sources to National Creek in the vicinity of the Mill Town.

The Phase I RI will include the following activities:

- The collection of air samples and surficial soil samples for assessing ambient (outdoor) air quality as it relates to fugitive dust emissions.
- The collection of surficial soil samples for assessing the nature and extent of lead- and arsenic- based paint impacts around current and historical structures.
- The collection of surficial soil samples for estimating metal concentrations and assessing the horizontal extent of mill tailings and ore/ore concentrates across the Mill Town.
- The collection of surficial soil samples for assessing the horizontal extent of blasting cap disposal areas and concentrations of related contaminants.
- The collection of surface water and sediment samples from National Creek and identification of nearby groundwater seeps for assessing how Site-related contaminants may migrate to and impact surface water and sediment quality.

Two VHB teams will perform and oversee the Phase I RI field work. One team will consist of three VHB personnel and will complete two 10-day shifts (including mobilization and demobilization) separated by a 4-day break. A second team will consist of two VHB personnel and will complete one 12-day shift (including mobilization and demobilization).

2.2 VHB Site Activities

The provisions of this HASP apply to all VHB personnel who may be exposed to safety and/or health hazards while performing investigative activities at the Site. Specifically, the Phase I RI field sampling program will include the following tasks:

Table 1 – Job Tasks

Job Task #	Activity Description
1	Data Quality Objective (DQO) 1: Ambient Air Investigation – Composite soil sample collection and co-located air sample collection.
2	DQO 2: Historical Paint Investigation – Soil sample collection and screening using X-ray fluorescence (XRF) in the vicinity of Mill Town Buildings
3	DQO 3: Mill Tailings and Ore/Ore Concentrates Investigation – Soil sample collection and screening using XRF on and around tailings piles
4	DQO 4: Blasting Caps Investigation – Incremental sampling methodology (ISM) soil sampling while coordinating with abandoned explosives anomaly avoidance expert
5	DQO 5: Transformer House Investigation – Soil sample collection around and under the Transformer House
6	DQO 6: National Creek Investigation – Surface water and sediment sample collection
7	Equipment and personnel decontamination

This HASP only addresses specific tasks being implemented by VHB and does not apply to VHB subcontractors, NPS staff, or other NPS contractors. VHB subcontractors, if utilized, will be provided a copy of this HASP for reference. All subcontractors performing intrusive work at the Site must prepare and follow their own site-specific HASP, which must be consistent with this VHB HASP. VHB or NPS staff providing direct oversight of the work may direct subcontractors to stop work if unsafe practices are observed.

Any non-VHB personnel who wish to enter the exclusion zone (defined in section 6.2), including subcontractors, oversight personnel, NPS personnel, and other visitors, are required to receive a briefing on this HASP and have appropriate training. If a visitor to the Site does not comply with the requirements of this HASP, all work will stop until the visitor has left the work area. Emergency responders who must enter the controlled work area (as defined in section 6.2) will be verbally informed of health and safety hazards in accordance with procedures in this HASP.

3

Roles and Responsibilities

3.1 Team Organization

The following Project Team functions have the Safety and Health responsibilities as listed below:

NPS Co-Project Supervisors/Federal Government Leads – Veronica Dickerson & Sarah Venator

The NPS Project Manager has the responsibility to direct all operations, and the authority to suspend or modify work practices for any reason.

Project Manager/Cleanup Lead – Tracy Planinsek

The Project Manager is responsible for:

Project oversight;

Implementing the project HASP,

- Providing an adequate supply of safety equipment;
- Ensuring that all Site workers have received safety training for this project, and document that they have read and understand the HASP.

Site Safety Officer (SSO) – Brie Hicknell, Adam Robtoy

The SSO is responsible for:

- Advising the Project Manager of safety and health matters, and implementing the HASP;
- Providing Safety Data Sheets or other chemical information to the Project Manager regarding chemical hazards on Site;
- Establishing safe work zones;
- Preparing HASP amendments when needed at the Site;
- Ensuring that all field personnel have become familiar with the HASP contents through a documented training session;
- Daily safety meetings; and
- Accident reporting.

The SSO has the authority to:

- Request compliance with the HASP;
- Suspend or modify work practices for safety reasons; and
- Exclude from the Site any individual whose on-Site conduct endangers themselves or others.

Corporate Health and Safety Representative (CHSR) – Ben DeJong

The CHSR is responsible for the overall safety and health of all Site workers, and for overall regulatory compliance. In addition, the CHSR is responsible for:

- Reviewing the HASP to ensure compliance with all appropriate regulations and compliance with Company Policies;
- Responding to significant changes in working conditions by issuing a written amendment to this HASP.
- Making final decisions with regard to any safety and health dispute or concerns that arise at the Site; and
- Any mandatory incident reporting.

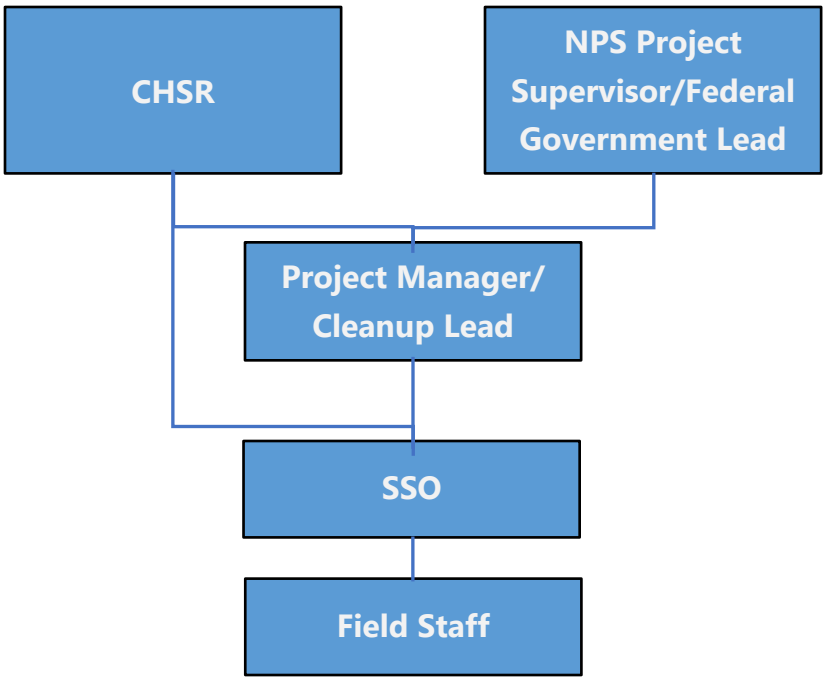
Other Project Professionals:

All VHB personnel on Site are responsible for conducting work in compliance with the HASP and for bringing safety and health concerns to the attention of the SSO. Some of the specific responsibilities of the personnel are as follows:

- Reading the HASP in its entirety before starting on-Site work;
- Completing the HASP acknowledgment form before starting work;
- Attending the required pre-entry briefing (before on-Site work) and all subsequent safety meetings conducted during the field program;
- Bringing forth any questions or concerns regarding the content of the HASP to the Project Manager or the SSO before starting or continuing on-Site work;
- Reporting all accidents, injuries, illnesses, regardless of the severity, to the SSO; and
- Complying with the requirements of this HASP and the requests of the SSO.

3.2 Chain of Command

The following diagram illustrates the Chain of Command with regard to safety and health responsibilities listed above in Section 2.



4

General Safe Work Practices

The following are general safe work practices to be used at the Site:

1. The "buddy system" shall be used at all times by field personnel in restricted areas.
2. Visual, voice, or radio communications shall be maintained at all times.
3. Dermal contact with contaminated surfaces, waters, soils, and equipment shall be avoided whenever possible. Do not walk through discolored surfaces or kneel on the ground, nor lean, sit, or place equipment on chemical containment drums or containers.
4. Eating, drinking, and smoking are prohibited in exclusion and contamination reduction zones (defined in Section 6.2). Alcoholic beverages are prohibited during working hours.
5. Hands and face must be thoroughly washed upon leaving the contamination reduction area. Further, whenever decontamination procedures for outer garments are in effect, the entire body should be thoroughly washed as soon as possible after the protective garment is removed.
6. All equipment must be decontaminated or discarded upon exiting the exclusion zone.
7. Personal protective equipment (PPE) appropriate for work conditions shall be required for all field personnel and may include, but is not limited to respiratory protection, hard hat, chemical resistant coveralls, boots, gloves, safety glasses, ear protection, insect repellant, and bear spray.
8. Anyone reporting to work under the influence of alcohol and/or illicit drugs will be subject to disciplinary action. Any employee taking prescription medication or under a physician's care that could affect or inhibit job performance must notify the SSO one week before reporting to the work Site.
9. All employees shall listen for and yield the right-of-way to heavy equipment and vehicles.
10. Employees are responsible to clean and maintain the protective equipment issued to them. Any and all defects or failures of the equipment shall be reported immediately to the SSO, and the SSO must decide if replacement is necessary.
11. All personnel shall report all injuries and/or illnesses to the SSO regardless of severity.
12. Personnel shall avoid any potentially dangerous environmental situations such as entering a confined space without proper supervision, training, and equipment.
13. Alaska 811: Digline must be notified not less than 15 business days prior to all drilling and excavation operations.

14. Personnel encountering a potentially hazardous environment (e.g., noticing strong vapor levels of unidentified substances) shall instruct all other on-Site personnel to leave the Site and shall call the project manager and SSO to report the condition and await instruction. Personnel shall not re-enter the Site without proper protective clothing and shall not work at the Site until the unknown substance is further identified and/or abated.
15. A hard hat shall be worn when working around heavy equipment or when overhead hazards exist.
16. A shirt and long pants shall be worn at all times.
17. Safety Glasses shall be worn when handling contaminated media and chemicals that may damage the eyes.
18. Skin abrasions must be thoroughly protected to prevent chemical exposure.
19. Each employee is responsible for his or her health and safety.

4.1 COVID-19 Pandemic

Various state and federal restrictions may apply as a result of their respective responses to the COVID-19 pandemic. VHB has adopted specific COVID-19 Field Work Protocols to provide guidance and protocols to support the completion of work in a safe manner. VHB's current protocols are available in Appendix F.

5

Hazard Assessment

5.1 Summary of Potential Hazards

A summary of the potential hazards to workers are identified in Table 2. The Physical, Biological, and Chemical hazard control and mitigation strategies are discussed in the following sections.

Table 2 – Potential Hazards to Workers

Hazard	Job Task[1]						
	1	2	3	4	5	6	7
Harmful Dust	√	√	√	√	√	√	√
Confined Space							
Heavy Equipment							
Moving Parts							
Heavy Lifting	√	√	√	√	√	√	√
Overhead Hazards							
Hand Tools	√	√	√	√	√	√	√
Fall over 6 feet							
Cuts and Lacerations	√	√	√	√	√	√	√
Chemical Exposure	√	√	√	√	√	√	√
Steep, Uneven Terrain	√	√	√	√	√	√	√
Ionizing Radiation		√	√		√		
Abandoned Explosives		√	√	√			
Traffic	√	√	√	√	√	√	√
Electrical Hazards	√	√	√	√	√	√	
Excavation/Trenching							
Noise							
Slip, Trip, Falls	√	√	√	√	√	√	√
Hazardous Plants	√	√	√	√	√	√	√
Dangerous Animals	√	√	√	√	√	√	√
Heat Stress	√	√	√	√	√	√	√
Cold Stress	√	√	√	√	√	√	√
Extreme Weather	√	√	√	√	√	√	√
Burns							

[1] See Table 1 for Activity list

Surface Water						√	
Corrosives	√	√	√	√	√	√	

5.2 Physical Hazard Information

Physical Hazard Mitigation and Controls for this Project

Harmful Dust	Airborne particulates may be inhaled during sampling activities during dry conditions and high winds. Controls: Wear respiratory protection (e.g. N95 masks) when there are high levels of particulates in the air. Use water mist to control dust as needed when collecting soil samples.
Moving Parts	Hand drills, and other power tools may be used during investigation activities. Hazards include rotating and moving parts, potential breakage of parts. Controls: Awareness of equipment location and usage. Maintain distance from equipment that is in use, following standard operating procedures (SOPs), as described later in this document. Safety glasses are required when operating any power tools.
Heavy Lifting/ Hand Tools	Movement of heavy equipment, or misuse of tools such as a hand auger, could result in back strains or sprains. Controls: Enlist the help of others or use manual assist methods (dollies, carts, etc.) as needed to move large objects. Transport heavy items using vehicles or wagons as much as possible. Follow hand auguring SOPs.
Cuts and Lacerations	Cutting tools or other potentially penetrative equipment may be used during site activities. Debris scattered in sampling areas may also present a cut/laceration hazard. Controls: Wear appropriate clothes, gloves and footwear when using cutting tools and collecting samples in areas of debris, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip.
Steep, Uneven Terrain	Sampling locations will take place on and around the Site's mill tailings, or waste pile/debris. Controls: Work on steep slopes must be conducted by a two-person team. Take a less steep path when possible. Exercise caution on steep slopes by wearing suitable footwear, avoiding slippery areas, and carrying lighter loads.

Ionizing Radiation	XRF Field Screening Instruments are used on soil prior to soil sample collection. XRF Instruments create X-rays that are ionizing radiation. Controls: Remain aware of equipment location and usage. Maintain distance from equipment that is in use, and follow SOPs as described later in this document.
Traffic	Air sampling equipment will be set up and utilized on the main road that runs through the Mill Town. Roads are only utilized by NPS staff, but VHB staff should be aware of traffic when working at the Site. Controls: NPS staff will be notified of sampling locations. Changes to traffic patterns will be established by using tape or cones to designate lanes prior to sampling activities. Wear a reflective traffic vest when present in traffic areas.
Electrical Hazards	Electrical equipment will be used during site sampling (including but not limited to: power tools, XRF, Air sampling pumps, YSI, photoionization detector, peristaltic pump) Controls: Plug-in components shall be used with a ground-fault circuit interrupter (GFCI) and an extension cord at all times in potentially hazardous conditions such as wet environments or other exterior locations.
Noise	Noise – Operation of equipment may result in sound pressure levels exceeding 85 decibels. Controls: Wear ear muffs or plugs with noise reduction rating of 23 or above.
Slip, Trip, Falls	These may result from uneven terrain, tripping over materials or tools on the ground, or tripping on uneven ground surfaces. Controls: Good housekeeping; maintain orderliness at the Site. Wear stable footwear and beware of uneven terrain.

Cold Stress

Cold stress is caused by lowering the body core temperature to a level that causes lowered mental output, fatigue, unconsciousness, coma, and ultimately death. Exposure of flesh to cold may cause physical damage to extremities through the onset of frost bite. Conditions for low temperature injuries can be created by low ambient temperature, chilling winds, and perspiration or emersion, causing heat to be lost from the body by evaporation of water. During mornings and evenings the ambient temperature can drop below 45 degrees Fahrenheit; during these conditions, the following cold stress prevention controls will be implemented:

- If deemed appropriate by the SSO, all personnel shall work in 50-minute cycles separated by a minimum of 10 minutes in a warm environment. All work shall stop if the ambient temperature or the wind chill temperature results in serious discomfort, deterioration in function, or other signs of hypothermia in personnel.
- Monitoring personnel for signs of decreased mental function or physical signs of cold stress (discoloration of lips, excessive shivering, discoloration with no shivering).

The SSO should also ensure that the following steps are taken:

- Dress properly: Wear sufficiently warm clothes that do not cause restriction in blood flow or movement. Dress in layers with loose clothes. Wear a warm hat or hard hat liner. Care should be taken not to spend too long in a warm environment while dressed for cold conditions. This may cause excessive sweating or even heat stress (see below).
- Keep dry: Evaporation of perspiration or water from emersion can cause rapid cooling of the body, lowering the body temperature further. Care should be taken to avoid short periods of hard labor followed by periods of inactivity.
- Provide shelter (heated, if possible) to protect personnel during rest periods.

Heat Stress

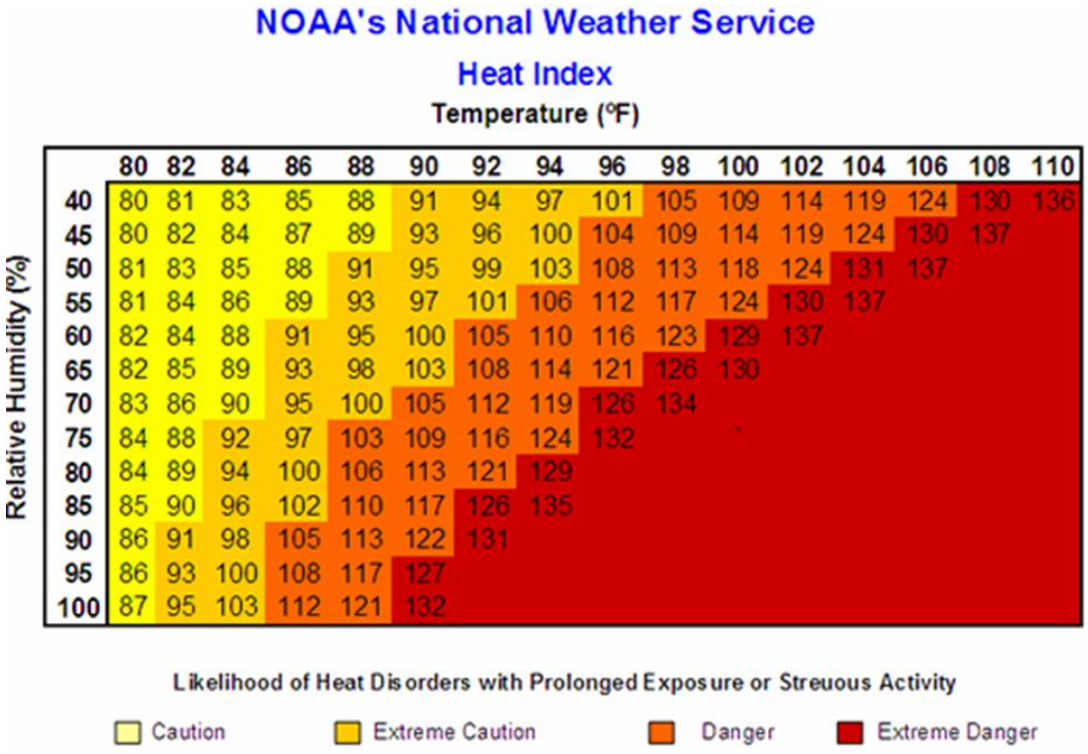
Heat-related illnesses include heat rash, heat cramps, heat exhaustion, and heat stroke. Heat rash can occur when sweat isn't allowed to evaporate, leaving the skin wet most of the time and causing irritation. Heat cramps are painful muscle spasms due to excessive salt loss associated with profuse sweating. Heat exhaustion results from the loss of large amounts of bodily fluids and excessive loss of salts due to profuse sweating. Heat stroke occurs when the body's temperature regulatory system fails and no longer effectively cools the body. Heat stroke is a medical emergency that can lead to permanent brain damage or death.

The risk of workers developing heat-related illness is dependent on several factors, including weather conditions, type of work being performed, and type of work clothes being worn. Due to the daily variability of these factors, the risk of heat-related illness and associated appropriate protective measures must be assessed daily and continually monitored throughout the workday. The SSO will evaluate the potential for heat-related illness each morning before work activities begin by using the attached heat stress guidance while considering the daily weather forecast and anticipated project work tasks. Field staff must be familiar with the symptoms of heat-related illness so they can monitor themselves and other field staff for these symptoms while working in the field.

The attached heat stress guidance uses the National Oceanic and Atmospheric Administration "heat index" to identify four risk levels and provides recommended protective measures for workers. The four risk levels are defined as lower, moderate, high, and very high to extreme (corresponding to the caution, extreme caution, danger, and extreme danger ranges, respectively, in the heat index table below). Working in direct sun or while wearing impermeable clothing increases the risk level beyond the default level identified by the heat index table alone.

Common sense protective measures (i.e., periodic rest, drinking water, etc.) are adequate for the lower and moderate risk levels. When risk levels are high the SSO needs to assess the need for increased rest periods and/or implement physiological monitoring (i.e., monitor pulse and/or body temperature). A very high to extreme risk level requires physiological monitoring and triggers additional protective measures to prevent heat-related illness. A smart phone app is available at the Google Play Store, the Apple App Store, or OSHA (Occupational Safety and Health Administration) (https://www.osha.gov/SLTC/heatillness/heatindex/heatapp.html) that can be used by workers if cellular phone coverage is available at the work location. This phone app will calculate the heat index based on nearby weather station data and provide links to relevant OSHA heat stress information and guidance. Physiological monitoring tools, such as the Hot Dot Body Temperature Alert Patch, may be used to augment heart rate and body temperature monitoring in the field. The inclusion of the Hot Dot or similar physiological monitoring tools must be discussed with the CSHR before being used in the field.

Figure 2 Heat Index



5.3 Biological Hazard Information

Biological Hazard Mitigation and Control for this Project

Insects

cdc.gov

Bees, wasps, mosquitoes, fleas, yellowjackets and other stinging or biting insects could be encountered on site or when entering vegetative areas.

Controls: These insects can cause irritating bites, can carry disease and can even cause death. Be aware that insect contact is possible, and that both overhead and ground level nest of bees and wasps may be found. If you know you are allergic to these insects, let the SSO know and verify that you have a sting kit with you. Keep lunch and snack foods covered. Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.

Hazardous Plants (to eat)

Exposure to toxic plants may produce symptoms ranging from dermatitis, local mucous membrane irritation, upset stomach, convulsions, irregular heartbeat, serious illness, and death in rare cases. Several toxic species of plants and mushrooms are present in the Park. **Baneberry, Monkshood, purple larkspur, devil's club, and some mushroom species** are poisonous to eat.

Controls: Don't eat anything in the wild.

Monkshood

Poison.org

Devil's Club

Fs.fed.us

Baneberry

Hort.extension.wisc.edu

Purple Larkspur

Fs.fed.us

Hazardous Plants (irritants)

Fws.gov

Cow parsnips, devil's club, and monkshood are topically toxic/irritants. Cow Parsnips are common in Alaska. If Cow Parsnip leaves are bruised and contacted, they can deposit a chemical on the skin that cause extreme sun sensitivity, resulting in sunburns that blister in short time and cause rashes that may persist for several months.

Controls: Wear long pants, long sleeve shirts when working in areas containing these plants. If you believe you have come in contact with them, keep the affected area covered from the sun and wash the area

	thoroughly with cool water and soap as soon as possible. Report any rashes or skin irritation to the SSO.
Dangerous Animals	The Park and surrounding area is home to a large variety of wildlife including **bears, wolves, coyotes, foxes, moose, caribou, bats, and rodents**. While a variety of animal species inhabit the region around the Kennecott Mill Town Site, only bears and moose are expected to potentially create a significant hazard. Black and brown/grizzly bears are present in the McCarthy area. Controls: Rodents in the area can spread disease through bites, scratches, and their waste. Maintain a safe distance from rodents, as they may carry rat bite fever, rabies and other diseases. Avoid contact with both dead and living animals. If bitten or scratched, get medical attention immediately. The following **safety precautions regarding bears** are summarized on the NPS Wrangell-St. Elias National Park and Preserve website https://www.nps.gov/wrst/planyourvisit/be-bear-aware.htm. The following **safety precautions regarding moose** are summarized on the NPS Wrangell-St. Elias National Park and Preserve website https://www.nps.gov/wrst/planyourvisit/moose-safety.htm. Britannica.com Bear.org Anchorage.net

Bear Safety

- Always work with a partner.
- Defensive aerosol sprays which contain capsaicin (red pepper extract) have been used with some success for protection against bears. These sprays may be effective at a range of 6-8 yards. If discharged upwind or in a vehicle, they can disable the user. Take appropriate precautions. If you carry a spray can, keep it handy and know how to use it. do not use pepper spray as a pre-emptive deterrent, since it may attract bears.
- Be aware you are in bear country and make noise, particularly in areas where the terrain or vegetation make it difficult to see. Loud noise can be made using bear bells, banging metal or other loud items periodically, or shouting every few minutes.
- Avoid thick brush. If you can't, try to walk with the wind at your back so your scent will warn bears of your presence. Contrary to popular belief, bears can see almost as well as people, but trust their noses much more than their eyes or ears.
- Give bears plenty of room. Maintain a distance of at least 100 yards, more if cubs are present. Identify yourself and let the bear know you are human. Talk to the bear in a calm voice. Wave your arms. Help the bear recognize you. If a bear cannot tell what you are, it may come closer or stand on its hind legs to get a better look or smell. A standing bear is usually curious, not threatening. Stand your ground. If the situation permits, back away slowly, keeping a close eye on the bear. Otherwise, you may wish to detour around the bear, but in this case, detour upwind so that the bear can get your scent but if the bear follows, stop and hold your ground, and try to appear larger.
- Do not make eye contact; look ahead and at the ground. Be aware of signs of distress, such as drooling, huffing/barking sounds, pretending you are not there after it has already seen you. A distressed bear might charge, but most charges are bluffs. Do not run. Continue waving your arms and talking to the bear. If the bear gets too close, raise your voice and be more aggressive. Never imitate bear sounds or make a high-pitched squeal.
- If a **brown bear/grizzly bear** is threatening, staying on your feet may allow you to dodge, or divert an attack. Play dead at the last moment, dropping into a fetal position with your hands clasped behind your neck and your elbows shielding your face. Typically, a bear will break off its attack once it feels the threat has been eliminated. Remain motionless for as long as possible. If you move, and the bear sees or hears you, it may return and renew its attack. If the attack is prolonged and the brown bear tries to feed on you, fight back vigorously! The encounter has now likely changed from a defensive one to a predatory one.
- If an attack by a **black bear** escalates and the bear physically contacts you, fight back with anything that is available to you. In rare instances, particularly with black bears, an attacking bear may perceive a person as food. If the bear continues biting you long after you assume a defensive posture, it likely is a predatory attack. Fight back vigorously. Fighting back may scare black bears off. In addition, if a bear is stalking you then you are in a predatory situation and fighting back is your only option. This also applies to any attack at night as these may also be considered predatory in nature.

Moose Safety

- Do not approach moose. Cow moose are extremely defensive of their young. Male moose may also be very aggressive during the rutting season.
- Be aware. In the summer months, moose blend in well to their environment and can be surprisingly hard to see for such large animals. They are likely to stand their ground even when they hear people approaching, so pay close attention to your surroundings, especially in prime moose habitat such as willow thickets or around streams or ponds.
- If the moose knows you are there, talk softly and move away slowly. Avoid aggressive movements.
- Signs of stress include laid back ears and raised hackles and indicate an imminent charge. Most of the time, when a moose charges it is a 'bluff', or warning for you to get back. If possible, get behind something solid (*a tree or a car*). A moose will use its feet to kick on both sides of a tree. Do not fight back, but use pepper spray, aiming for the face.
- **Unlike with bears, it is okay to run from a moose**. They usually won't chase you and if they do, it's unlikely that they'll chase you very far. If a moose knocks you down, curl up in a ball and protect your head with your arms and keep still.

5.4 Chemical Hazard Information

Documentation exists regarding a variety of contamination types, sources, and levels. Site-related contaminants are reported to be present in the following:

- Mine tailings and soil (heavy metals such as antimony, arsenic, copper, lead, cadmium, chromium, and mercury)
- Lead- and arsenic-based paint
- Fuel oil (Bunker C or similar) is present in floor trenches in the Power Plant and potentially in Site soils
- Boiler ash (note: boiler ash has been contained under a cap southwest of the Power Plant); and
- Remaining asbestos-containing material (note: most known asbestos-containing material has been removed or encapsulated).

Although several investigations have been conducted, there may still be unknown contaminants at the Site.

Site-related contaminants are ubiquitous throughout the Mill Town, and for the purposes of this analysis, all tailings, soils, surface dusts, and historical paints/paints of unknown age are assumed to be hazardous. Levels of contamination are expected to vary across the Mill Town and from structure to structure. Inhalation, dermal contact, and ingestion are considered potential routes of exposure for the Site-related contaminants. Previous air sampling conducted to date has not shown a clear and immediate danger to employees, contractors, visitors, or residents. Results of the most recent exposure sampling study conducted at the end of the 2019 season indicated that employee exposures to target airborne contaminants were below the referenced occupations' exposure limits for the work tasks evaluated. Target airborne contaminants were also well below occupational exposure limits during a simulated

Mill Building Tour and thus not expected to present an airborne hazard to either child or adult visitors (W&C, 2020).

The following are descriptions of each contaminant detected at the Site and its properties, including symptoms, in alphabetical order.

Antimony

Antimony is a silvery-white metal that is found in the earth's crust. Antimony ores are mined and then mixed with other metals to form antimony alloys or combined with oxygen to form antimony oxide. Antimony is released to the environment from natural sources and from industrial processes. In the air, antimony is attached to very small particles that may stay suspended in the air for many days. Most antimony ends up in soil, where it attaches strongly to particles that contain iron, manganese, or aluminum. Antimony can be found at low concentrations in some river, lake, and stream water.

Because antimony is found naturally in the environment, the general population is exposed to low levels of it every day, primarily in food, drinking water, and air. It may be found in air near industries that process or release it, such as smelters, coal-fired plants, and refuse incinerators. In polluted areas containing high levels of antimony, it may be found in the air, water, and soil.

Exposure to antimony at high levels can result in a variety of adverse health effects. Breathing high concentrations for an extended period can irritate your lungs and can cause heart and lung problems, stomach pain, diarrhea, vomiting, and stomach ulcers. Ingesting large doses of antimony can cause vomiting; other effects are unknown. Long-term animal studies have reported liver damage and blood changes when animals ingested antimony. Antimony can irritate the eyes if exposed and the skin with extended dermal contact. [Source: Agency for Toxic Substances & Disease Registry (ATSDR) Fact Sheet: Antimony, https://wonder.cdc.gov]

Arsenic

Arsenic is a naturally occurring element that is found in combination with either inorganic or organic substances to form many different compounds. Inorganic arsenic compounds are found in soils, sediments, and groundwater. These compounds occur either naturally or as a byproduct of mining, ore smelting, and industrial use of arsenic. Organic arsenic compounds are found mainly in fish and shellfish. In the past, inorganic forms of arsenic were used in pesticides and paint pigment. They were also used as wood preservatives and as a treatment for a variety of ailments.

People are most likely to be exposed to inorganic arsenic through drinking water and to a lesser extent through various foods. Water sources in some parts of the United States have higher naturally occurring levels of inorganic arsenic than other areas. Other sources of inorganic arsenic exposure include contact with contaminated soil or with wood preserved with arsenic. Unusually large doses of inorganic arsenic can cause symptoms ranging from nausea, vomiting, and diarrhea to dehydration and shock. Long-term exposure to high levels of inorganic arsenic in drinking water has been associated with skin disorders and increased

risks for diabetes, high blood pressure, and several types of cancer. Inorganic arsenic and arsenic compounds are considered to be cancer-causing chemicals.

Forms of organic arsenic (for example, arsenobetaine) found in seafood are not known to be toxic to humans. [Source: The Centers for Disease Control and Prevention (CDC): Arsenic Factsheet, https://epa.gov]

Asbestos

Asbestos is a naturally occurring mineral fiber. It was used in numerous building materials and vehicle products for its strength and ability to resist heat and corrosion before its dangerous health effects were discovered. Individual asbestos fibers cannot be seen by the naked eye, which puts workers at an increased risk. Asbestos fibers are released into the air during activities that disturb asbestos-containing materials. The asbestos fibers can then be inhaled without knowing and trapped in the lungs. If swallowed, they can become embedded into the digestive tract as well. Asbestos is a known human carcinogen and can cause chronic lung disease as well as lung and other cancers.

Symptoms and/or cancer may take many years to develop following exposure. [Source: OSHA Fact Sheet: Asbestos, https://osha.gov]

Cadmium

Cadmium is a natural element in the earth's crust. It is usually found as a mineral combined with other elements such as oxygen, chlorine, or sulfur. Most soil and rocks, including coal and mineral fertilizers, contain some cadmium. Cadmium is used in many products, including batteries, pigments, metal coatings, and plastics, and it is found in cigarette smoke. Cadmium enters the environment through mining operations and the action of wind and rain. Forest fires and volcanoes also release some cadmium to the air.

People are exposed to cadmium through their diet, since cadmium is absorbed into plant and animal foods that people eat. People are exposed to higher amounts of cadmium by breathing cigarette smoke. In the workplace, people are exposed where cadmium is used or generated, such as in battery manufacturing, metal soldering, or welding. When eaten, large amounts of cadmium can severely irritate the stomach and cause vomiting and diarrhea. Breathing high levels of cadmium damages people's lungs and can cause death. Exposure to low levels of cadmium in air, food, water, and particularly in tobacco smoke over time may build up cadmium in the kidneys and cause kidney disease and fragile bones. Cadmium is considered a cancer-causing agent. [Source: CDC Cadmium Factsheet, https://cdc.gov]

Fuel Oil

Fuel oil Nos. 4, 5, and 6 are commonly known as "residual oils" since they are manufactured in whole or in part from distillation residues from refinery processing. Residual oils are complex and variable mixtures of relatively high molecular weight compounds and are difficult to characterize in detail. Molecular composition includes asphaltenes, polar aromatics, naphthalene aromatics, aromatics, saturated hydrocarbons and heteromolecules containing sulfur, oxygen, nitrogen and metals. Dimethyl and trimethyl naphthalenes are

important components of Fuel No. 6. The viscous residuum fuel oils, No. 5 and 6, sometimes referred to as bunker fuels, usually must be preheated before being burned.

Fuel oil No. 6 contains about 15% paraffins, 45% naphthenes, 25% aromatics, and 15% non-hydrocarbon compounds; the hydrocarbons contain 30 and greater carbon atoms. It consists of straight-run and cracked distillates and residuals and contains aliphatics and aromatics. Appreciable concentrations of polycyclic aromatic hydrocarbons (PAHs) are present in residual fuels because of the common practice of using both uncracked and cracked residues in their manufacture.

Fuel oil #6 would be expected to be a skin, eye and respiratory irritant and a central nervous system depressant from inhalation of large amounts of the vapor or mist. Prolonged or repeated contact with the skin may produce a defatting dermatitis with dryness and cracking. Chronic effects of some of the constituents in Fuel No. 6 (such as naphthalenes) include changes in the liver and kidney. Exposure to petroleum in soil is predominantly of concern through a number of possible exposure pathways, including dermal contact with soil, ingestion of soil, inhalation of soil particulates, and ingestion of contaminated groundwater. The PAHs in this product include both carcinogenic and noncarcinogenic compounds (Source: NPS, 1997).

Chromium

Chromium is a naturally occurring element found in rocks, animals, plants, and soil. It can exist in several different forms. Depending on the form it takes, it can be a liquid, solid, or gas. The most common forms are chromium (0), chromium(III), and chromium(VI). No taste or odor is associated with chromium compounds. The metal chromium, which is the chromium (0) form, is used for making steel. Chromium(VI) and chromium(III) are used for chrome plating, dyes and pigments, leather tanning, and wood preserving.

Chromium can be found in air, soil, and water after release from the manufacture, use, and disposal of chromium-based products, and during the manufacturing process. Chromium does not usually remain in the atmosphere but is deposited into the soil and water. Chromium can easily change from one form to another in water and soil, depending on the conditions present. Exposure to chromium can occur by eating food containing chromium(III), breathing contaminated workplace air or skin contact during use in the workplace, drinking contaminated well water, or living near uncontrolled hazardous waste sites containing chromium or industries that use chromium.

Breathing high levels of chromium(VI) can cause irritation to the lining of the nose, nose ulcers, runny nose, and breathing problems, such as asthma, cough, shortness of breath, or wheezing. The main health problems seen in animals following ingestion of chromium(VI) compounds are irritation and ulcers in the stomach and small intestine and anemia. Skin contact with certain chromium(VI) compounds can cause skin ulcers. Some people are extremely sensitive to chromium(VI) or chromium(III). Allergic reactions consisting of severe redness and swelling of the skin have been noted. [Source: ATSDR ToxFAQs™ for Chromium, https://atsdr.cdc.gov]

Copper

Copper is a metal that occurs naturally throughout the environment, in rocks, soil, water, and air. Copper is released into the environment by mining, farming, and manufacturing operations and through wastewater releases into rivers and lakes. Copper is also released from natural sources, like volcanoes, windblown dusts, decaying vegetation, and forest fires. Copper released into the environment usually attaches to particles made of organic matter, clay, soil, or sand. Copper does not break down in the environment. Copper compounds can break down and release free copper into the air, water, and foods.

You may be exposed to copper from breathing air, drinking water, eating foods, or having skin contact with copper, particulates attached to copper, or copper-containing compounds. Lakes and rivers that have been treated with copper compounds to control algae, or that receive cooling water from power plants, can have high levels of copper. Soils can also contain high levels of copper, especially if they are near copper smelting plants. You may be exposed to copper by ingesting copper-containing fungicides, or if you live near a copper mine or where copper is processed into bronze or brass. You may be exposed to copper if you work in copper mines or if you grind metals containing copper.

Breathing high levels of copper can cause irritation of your nose and throat. Ingesting high levels of copper can cause nausea, vomiting, and diarrhea. Very-high doses of copper can cause damage to your liver and kidneys, and can even cause death. [Source: ATSDR ToxFAQs™ for Copper, https://atsdr.cdc.gov]

Lead

Lead is a soft, dense, blue-gray metal. Lead occurs naturally in the earth's crust, where it combines with other elements such as oxygen and sulfur. It is used to make batteries and metal mixtures.

Because of health concerns, lead is no longer added to gasoline and house paints. Workers may be exposed in industries that involve lead, such as smelting and battery manufacturing. Workers can also secondarily expose household members by bringing lead home on their clothes. People can be exposed to small amounts of lead by breathing air, drinking water, eating food, or swallowing dust or dirt that contains lead. For adults, the diet is the source of most general low-level environmental exposure to lead. Children can be exposed to more lead than adults. Children are commonly exposed to lead from hand-to-mouth activities involving contaminated dust and soils around older homes that contain lead-based paint or from eating paint chips that contain lead.

No safe blood lead level has been identified. For infants and young children, lead levels of 10 micrograms or more in a deciliter of blood are levels of concern and can damage ability to learn. (A microgram is one millionth of a gram. A deciliter is about half a cup of liquid.) Of all people, young children face the most danger from exposure to lead because their growing bodies are more prone to harm; also, children absorb lead more easily than do adults' bodies. Pregnant women and women of childbearing age should avoid exposure to lead because lead ingested by a mother can affect the unborn child. At much higher blood lead

levels lead can damage people's kidneys, blood, and nervous system and progress to coma, convulsions, or death. [Source: CDC Lead Factsheet, https://cdc.gov]

Mercury

Mercury is an element and a metal that is found in air, water, and soil. It exists in three forms that have different properties, usage, and toxicity. The three forms are called elemental (or metallic) mercury, inorganic mercury compounds, and organic mercury compounds. Organic mercury is not a factor at the Site.

Elemental mercury: People may be exposed when they breathe air containing elemental mercury vapors. Vapors may be present in such workplaces as dental offices, smelting operations, and locations where mercury has been spilled or released. In the body, elemental mercury can be converted to inorganic mercury. The human health effects from exposure to low environmental levels of elemental mercury are unknown. Very high mercury vapor concentrations can quickly cause severe lung damage. At low vapor concentrations over a long time, neurological disturbances, memory problems, skin rash, and kidney abnormalities may occur.

Inorganic Mercury: People may be exposed if they work where inorganic mercury compounds are used. When eaten in large amounts, some inorganic mercury compounds can be very irritating and corrosive to the digestive system. If repeatedly eaten or applied to the skin over long period of time, some inorganic mercury compounds can cause effects similar to what is seen with long term mercury vapor exposure, including neurological disturbances, memory problems, skin rash, and kidney abnormalities. [Source: CDC Mercury Factsheet, https://cdc.gov]

Nickel

Nickel is a naturally occurring element. Pure nickel is a hard, silvery-white metal used to make stainless steel and other metal alloys. People are exposed to nickel by: eating food containing nickel, which is the major source of exposure for most people; skin contact with soil, bath or shower water, or metals containing nickel, as well as by handling coins or touching jewelry containing nickel; drinking water that contains small amounts of nickel; or breathing air or smoking tobacco containing nickel.

The most common harmful health effect of nickel in humans is an allergic reaction. Approximately 10-20% of the population is sensitive to nickel. People can become sensitive to nickel when jewelry or other things containing it are in direct contact with the skin for a long time. Once a person is sensitized to nickel, further contact with the metal may produce a reaction. The most common reaction is a skin rash at the site of contact. The skin rash may also occur at a site away from the site of contact. Less frequently, some people who are sensitive to nickel have asthma attacks following exposure to nickel. Some sensitized people react when they consume food or water containing nickel or breathe dust containing it. People working in nickel refineries or nickel-processing plants have experienced chronic bronchitis and reduced lung function. These persons breathed amounts of nickel much higher than levels found normally in the environment. Workers who drank water containing

high amounts of nickel had stomachache and suffered adverse effects to their blood and kidneys. [Source: ATSDR ToxFAQs™ for Nickel, https://atsdr.cdc.gov]

Vanadium

Vanadium is an element that occurs in nature as a white-to-gray metal compound and is often found as crystals. It usually combines with other elements such as oxygen, sodium, sulfur, or chloride.

Vanadium and vanadium compounds can be found in the earth's crust and in rocks, some iron ores, and crude petroleum deposits. Exposure to vanadium occurs by: eating foods containing vanadium; breathing air near an industry that burns fuel oil or coal - these industries release vanadium oxide into the air; working in industries that process vanadium or make products containing vanadium; breathing contaminated air or drinking contaminated water; or breathing cigarette smoke. Vanadium is not readily absorbed by the body from the stomach, gut, or contact with the skin. Exposure to high levels of vanadium pentoxide in air can result in lung damage. Nausea, mild diarrhea, and stomach cramps have been reported in people who have been exposed to some vanadium compounds. A number of effects have been found in animals ingesting vanadium compounds including decreases in the number of red blood cells, increased blood pressure, and mild neurological effects. [Source: ATSDR ToxFAQs™ for Vanadium, https://atsdr.cdc.gov]

5.5 Other Hazard Information

Abandoned Explosives Safety

Blasting caps and fuses have been found at various Site locations and present a hazard to VHB personnel working in these Blasting Cap Disposal Areas. While the exact number of remaining caps on Site is unknown, they have been found on the north side of the former Mill Building, downslope from the door to the crushing level, and on the moraine immediately west of the north end of the former leaching plant (Shields, 2011). A more recent report (Shields, 2015) states that there is a high density of fuses and caps throughout the debris slope on the north side of the former Mill Building, and that several fuse pieces were found on the south side slope. The report also describes the upper mill perimeter and ore bin as locations that should be treated as potentially containing explosives. The WRST Regional Blasting Officer (Tim George) will be present at the Site and will direct VHB to safe locations within the Blasting Cap Disposal Areas during sampling. VHB field personnel will complete abandoned explosives awareness training provided by the Mr. George and will follow his direction when working in an area where blasting caps and fuses may be present.

XRF Field Screening Safety

Nitrile gloves and steel toed boots are required as specified in Section 7. The general XRF radiation avoidance procedures will be followed as specified below:

- The XRF analyzer cannot be operated without a password. Do not share the password with anyone who has not been instructed in the proper use of the equipment or who has not received Ionizing Radiation Hazards Training.
- Turn the analyzer off before cleaning the exterior.
- Keep the battery pack separated from the instrument until all preparations are complete and screening is ready to begin.
- Always position the window centered on object intended for analysis. Do not attempt to use the analyzer on small soil piles or outcrops.
- Make sure no body parts are near or within the frame of the window.
- Use two hands while operating the instrument.
- The Deadman trigger, which will stop the instrument if the operator releases the trigger, should be set to "on" if the XRF analyzer is operated in unstable or slippery conditions.
- The XRF analyzer may require the operator to remain still for approximately one minute. Check surroundings carefully for biological hazards or nuisance, and trim overhanging vegetation, before beginning the test.
- Communicate with other people on-site to establish an exclusion zone of 10 feet in front of the beam and 3 feet to each front side.
- Do not use the analyzer if it has been dropped or physically damaged or is malfunctioning.

Driving Safety

Driving presents hazards to employees from road conditions, other drivers on the road, and even personnel's own behavior. While driving hazards can never be totally eliminated, the following steps can help reduce risk and keep everyone on the road safe

- Abide by all traffic laws.
- Seat belts must always be worn.
- Although using cell phones while driving is discouraged, hands-free cell phone devices may be used when necessary. The use of hand-held cell phones is strictly prohibited while driving.
- Practice defensive driving. Defensive driving starts from the moment a driver gets behind the wheel.
- Always be alert to the hazards around, including changing weather. Driving defensively means taking every possible precaution to avoid an accident, despite the hazards around.
- Inspect vehicle before a trip. Make sure tires are properly inflated and loads are securely tied down.
- Eliminate or reduce hazards whenever possible. Make sure that no loose items are on the dashboard or by feet, or that items are not piled too high on seats or floors.
- Keep a safe distance from the vehicle in front.
- Let problem drivers move ahead. Do not challenge them.
- Use extra caution when driving in the winter. Clear all snow and ice from the vehicle before driving, leave plenty of room for stopping, and drive slowly.
- All vehicles must be equipped with basic emergency response and safety equipment including:
    - Potable water;
    - First aid kit;
    - Flashlight with extra batteries;
    - Anti-bacterial wipes, gel, or solutions; and
    - PPE necessary for work at the destination, appropriate for the expected exposures.
- If your vehicle should break down:
    - Get off the roadway, out of the path of oncoming traffic, even if it requires driving on a flat tire. The tire is replaceable.
    - Turn on emergency flashers.
    - If a motorist stops to render assistance, it is better to remain in the car, and ask them to get help.

6

Contaminated Site Protocols

6.1 Exposure Controls

Based on available contaminant concentration data, expected locations of contamination, and types of activities involved in this project, it is currently anticipated that modified Level D PPE will be necessary to perform field work.

The main pathway for exposure to chemicals is through dermal contact, ingestion, and inhalation. Nitrile gloves and safety glasses will prevent contact exposure when handling contaminated media. Where conditions warrant (e.g., dry conditions and high winds), respiratory protection may be needed to prevent ingestion by airborne particulates. Refer to Section 7 for more information on PPE.

Investigation-derived waste will be produced during investigation activities and will require the same level of PPE for proper handling and disposal.

6.2 Work Zones

To prevent both exposure of unprotected personnel and migration of contamination due to tracking by personnel or equipment, work areas and associated PPE requirements will be clearly identified. The SSO will designate work areas or zones as suggested in the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities" (National Institute for Occupational Safety & Health [NIOSH], 1985). The areas surrounding each work area will be divided into three zones:

- Exclusion or "hot" Zone;
- Contamination Reduction Zone; and
- Support Zone.

Exclusion Zone

Based on the principal activities performed during this investigation, the establishment of an exclusion zone based on possible contaminant exposure is not necessary. However, an exclusion zone may be warranted in particular sampling areas to maintain little to no disturbance from residents or visitors as deemed necessary by the SSO.

Contamination Reduction Zone

A station will be established adjacent to work areas for decontamination of tools that are in contact with the potentially contaminated site media. Decontamination water will be

collected in 5-gallon buckets and transferred to a 55-gallon drum. Personnel will remove PPE and other disposable items and place them in a plastic bag before leaving the work area.

Support Zone

The support zone is the portion of the Site that does not lie within the exclusion zone or contamination reduction zones.

6.3 Restricted Areas

VHB previously coordinated with the Federal Government Lead and an on-Site Park representative to exchange safety information, address and receive authorization of on-Site traffic, and identify restricted areas. VHB will continue to coordinate with the Park, as necessary, as work progresses. The SSO may establish an exclusion zone at locations found necessary to prevent unauthorized personnel from entering the work areas.

6.4 Hazard Communication Program

When face-to-face communication is possible, field personnel will communicate verbally. In areas where verbal communication is not possible, field personnel will communicate with two-way radios or cellular phones to facilitate contact between team members, the SSO, and/or local emergency response personnel in the event of an emergency.

6.5 Decontamination Procedures

Decontamination will occur in the Contamination Reduction Zone.

Machinery and Equipment

All equipment that contacts potentially contaminated media will be decontaminated as appropriate on Site. All hand-held equipment will be decontaminated by removal of disposable wrappings or by washing in soapy water, followed by a clean water rinse on Site.

If the XRF analyzer becomes muddy or dusty during use, it will be wiped with a household cleaning wipe. The XRF analyzer will be cleaned with a cleaning wipe after use before leaving the Site. Do not wash the XRF analyzer with water.

Any coring and hand auguring equipment will be decontaminated by cleaning all parts of the equipment that were in contact with potentially contaminated soil.

Decontamination water will be collected in either 55-gallon drums or other appropriate containers.

Personnel Decontamination

All disposable gloves and Tyvek, if utilized, will be disposed properly as solid waste. Work boots will be washed of all nuisance contaminants. The employees' hands and faces will be washed before eating and as soon as possible at the end of each workday.

All disposable gear will be placed in a double lined garbage bag and securely sealed. The garbage bag will be delivered to a disposal location approved by NPS.

7

Personal Protective Equipment

Level D PPE will be used by VHB personnel to control physical and chemical hazards at the Site. Level D PPE will include:

- **Boots/shoes:** Heavy duty work boots or shoes with a safety toe and shank (compliant with the American National Standards Institute (ANSI) Z41 standard) are required if activities involve heavy equipment, heavy lifting, or in areas where debris is present. In wet environments, (or if preferred) these can be substituted with heavy duty rubber boots with safety toe and shank.
- **Gloves:** cut-resistant gloves are required when using knives or blades, or working with power tools and machinery; chemically-resistant gloves are required when collecting samples, handling investigation-derived waste, or performing work with potential chemical contact hazards (e.g., nitrile for short exposure times of less than five minutes and Viton for longer potential exposure times).
- **Hearing Protection:** OSHA-approved hearing protection shall be worn when exposed to noise levels greater than 85 decibels.
- **Coveralls:** Coveralls may be worn to protect clothes from nuisance contamination, if desired. No hood or booties are required.
- **Safety Glasses:** chemical splash goggles or face shield will be required when splash, dust, flying particles or other eye hazards are present (ANSI standard Z.87).
- **Traffic Vests:** High-visibility safety vests must be worn when working in high-traffic areas or near heavy equipment.
- **Hard Hats:** NIOSH-approved, (as necessary) when overhead hazards exist, and construction activities are ongoing.

It should be noted that respiratory protection such as N95 masks may be needed in dry and windy conditions that may cause a large particulate concentration in the atmosphere.

7.1 Additional Safety Equipment

The following should be available and on Site:

- Barricade tape or safety cones;
- Respiratory protection (e.g. N95 mask);
- Portable hand-held eyewash bottles with a minimum of six gallons of water available.
- Photoionization Detector

8

Training

All Site workers, including subcontractors, will be required to produce evidence of 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training, current annual 8-hour HAZWOPER refresher training, and evidence of enrollment in a medical surveillance program.

All personnel performing activities covered by this HASP must be active participants in a medical monitoring program that complies with 29 CFR 1910.120(f). Each individual must have completed an annual surveillance examination within the last year (if exposure criteria have been met) and an initial baseline examination prior to performing any work on the Site covered by this HASP. All VHB employees are enrolled in and have had a baseline Hazardous Materials worker medical exam. Documentation of these exams can be found at VHB's main office in confidential personnel files.

8.1 Safety and Health Training

HAZWOPER

All personnel covered by this HASP must have completed the appropriate training requirements specified in 29 CFR 1910.120(e). Each individual must have completed an annual 8-hour refresher training course and/or initial 40-hour training course not more than one year before performing any work covered by this HASP. Additionally, personnel must have completed at least three days of field training under the direction of a trained and experienced supervisor. Unless they are the sole representative of VHB on-site, the SSO must have completed an 8-hour HAZWOPER supervisor training course. Applicable clearance and credentials shall be documented for each VHB site worker on the form in Appendix E.

First Aid/CPR

The SSO will be trained in First Aid/Cardiopulmonary Resuscitation (CPR).

8.2 Pre-Entry Briefing

The SSO will conduct a pre-entry briefing to all personnel before Site activities begin. The HASP acknowledgement form must be signed by each VHB field employee before entry to the Site. Specific topics that will be discussed during the pre-entry briefing include:

- Site history;
- Work scope;
- Potential hazards associated with contaminants of concern and how these potential hazards will be controlled;
- PPE;
- Potential physical hazards associated with implementing the scope of work;
- Emergency egress and hospital location/directions;
- Decontamination procedures; and
- Communication systems (e.g., emergency signals).

The HASP acknowledgement form will be signed at this meeting. Daily safety meetings will be conducted in the morning throughout the duration of the field portion of the project. Attendance of the safety meetings is mandatory for workers who are assembled at the Site.

8.3 Abandoned Explosives Safety Awareness Training

VHB field personnel will attend training provided by the WRST Regional Blasting Officer prior to arrival at the Site. The WRST Regional Blasting Officer will provide additional daily training when sampling occurs in the blasting cap disposal areas.

8.4 Ionization Radiation Safety Training

VHB employees tasked with using a field portable XRF analyzer will complete an approved ionizing radiation safety training course prior to conducting field sampling activities. Documentation of applicable employee training for hazardous waste site work is provided in Appendix E.

9 Emergency Procedures

The SSO is responsible for responding to and correcting any emergency situation. These duties include the protection of employees, evacuating and securing the work area, ensuring the protection of the public and the environment, ensuring that appropriate decontamination procedures are implemented for all personnel, and determining the cause of any incident, recommending changes to prevent its reoccurrence, upgrading or downgrading the level of PPE, and notifying the appropriate Federal, State and local agencies.

The Project Manager is responsible for ensuring that the features of the emergency response contingency plan are in place, personnel are appropriately trained, and required safety and spill response equipment is maintained on the Site. At this Site, the Field Manager will also perform the duties of the SSO.

9.1 Personnel Injury

All personnel who are injured or become ill in the Exclusion Zone must be decontaminated to the maximum extent possible prior to exiting. If injuries are minor, a full decontamination procedure will be completed, and first-aid administered as needed prior to transport. If injuries are severe, a partial decontamination will be completed that will include the removal of all contaminated clothing and redressing in clean coveralls or appropriate materials. First-aid will be administered by trained personnel, as needed, prior to the arrival of medical personnel. Any person transported for further medical attention will be accompanied by information regarding the chemical(s) to which they may have been exposed.

Any vehicle and equipment used to transport contaminated personnel will be cleaned or decontaminated, as necessary.

9.2 Evacuations Routes and Procedures

In the event of an emergency that requires evacuation of the work area, a verbal command will be used to clearly inform all personnel of the emergency situation. At that time, all personnel will proceed to a point within the clean zone, upwind of any dust and proceed with decontamination procedures.

10

Accident Reporting

Any accident or injury to a VHB employee, subcontractor, or visitor must be reported by the worker to the SSO and/or the Project Manager immediately and to the VHB Incident Reporting Hotline (844)-407-0011. Local park personnel will be contacted in the event of a serious injury requiring emergency medical attention so the appropriate park resources can be mobilized. Injuries to subcontractors must be reported to the subcontractor's contact person immediately. The SSO will investigate as necessary to determine the cause of the incident and whether it was preventable. If the incident may have been preventable, the SSO will determine by what means future incidents of a similar nature can be avoided. The SSO will also investigate if applicable safety and health procedures were followed and whether required PPE was properly used. The SSO will complete the Accident Investigation Report Form (see Appendix D) and submit it to the Project Manager who will review the form and interview the employee and/or SSO if necessary.

Any work-related or suspected work-related fatalities or inpatient hospitalization of three or more workers must be reported within 8 hours by telephone to OSHA at 1-800-321-6742.

11

Emergency Information

11.1 Emergency Contact Information

Emergency Contacts	Telephone
Ambulance	911
Fire (Palmer Fire Station)	911 or 907.745.3709
Police (Palmer Police Department)	911 or 907.745.4811
Hospital (Mat-Su Medical Center)	911 or 907.861.6000
VHB	
Tracy Planinsek, Project Manager	207.536.2580
Brie Hicknell, SSO	207.889.3117
Adam Robtoy, SSO	802.778.1290
Ben DeJong, CHSR	802.829.0932
Client Contact	
Veronica Dickerson, Federal Government Lead	440.665.0915
Sarah Venator, Federal Government Lead	907.644.3573

11.2 Hospital Location and Directions

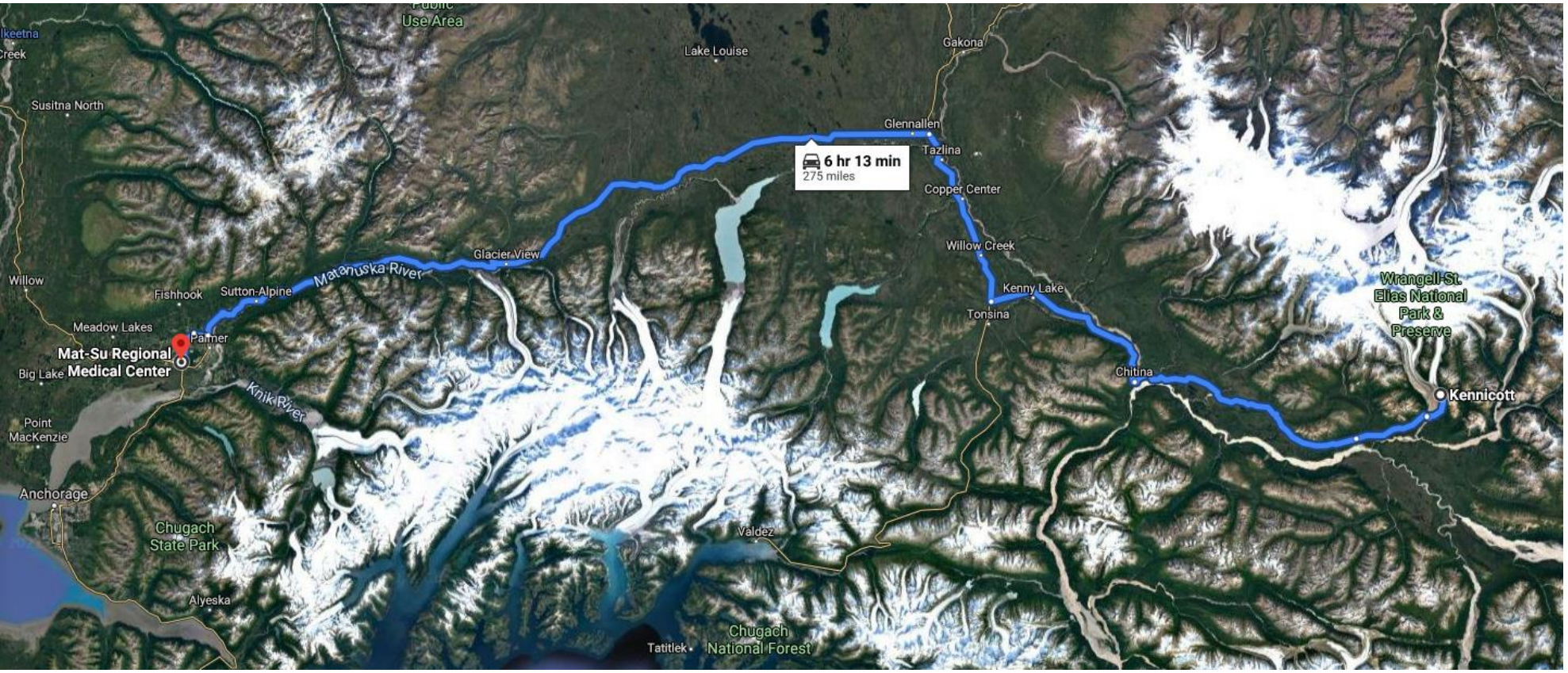


Figure 3 Route to Mat-Su Regional Medical Center

1. Follow McCarthy Road and Edgerton Highway to AK-4N for 2 hours and 57 min (98.1 miles)
2. Take AK-1 S to Fishhook-Willow Road/N Palmer Fishhook Rd in Farm Loop for 3 hours and 3 minutes (168 miles)
3. Follow Fishhook-Willow Rd/N Palmer-Fishhook Rd and N Trunk Rd to your destination for 13 minutes (8.6 miles)

Appendix A - Safety Meeting Sign-In Sheet

DAILY SAFETY MEETING ATTENDANCE FORM

Kennecott Mill Town Site

Kennecott, AK VHB Project #58345.43

Conducted by:	Date and Time Performed:

Topics Discussed:	›
	›
	›
	›

Printed Name	Signature	Representing

Appendix B – Hazard Assessment Form

Hazard Assessment Form – Job Task #1

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by:	Brie Hicknell	**Date:** 7/19/2021

Job Task: DQO 1: Ambient Air Investigation - composite soil sample collection and co-located air sample collection.

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in soil (heavy metals)
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
	Ionizing Radiation		›
	Abandoned Explosives		›
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including air pumps, Dust Traks, and weather station
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #2

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by:	Brie Hicknell	**Date:** 7/19/2021	
Job Task:	DQO 2: Historical Paint Investigation - Soil sample collection and screening using XRF around Mill Town Buildings		

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in soil (heavy metals)
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
√	Ionizing Radiation	2	› XRF Field Screening Instrument emits X-rays
√	Abandoned Explosives	3	› Paint sampling program overlaps into the blasting cap area of concern
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including XRFs, moisture meter, GPS, iPads
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #3

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by:	Brie Hicknell	**Date:** 7/19/2021	
Job Task:	DQO 3: Mill Tailings and Ore/Ore Concentrates Investigation – Soil sample collection and screening using XRF on and around tailings piles		

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in soil (heavy metals)
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
√	Ionizing Radiation	2	› XRF Field Screening Instrument emits X-rays
√	Abandoned Explosives	3	› Mill tailings sampling program overlaps into the blasting cap area of concern
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including XRFs, moisture meter, GPS, iPads
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #4

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by:	Brie Hicknell	**Date:** 7/19/2021
Job Task:	DQO 4: Blasting Caps Investigation – ISM soil sampling while coordinating with WRST Regional Blasting Officer	

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in soil (heavy metals)
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
	Ionizing Radiation		›
√	Abandoned Explosives	3	› Work will take place in blasting cap disposal area
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including GPS, iPads
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #5

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by:	Brie Hicknell	**Date:** 7/19/2021

Job Task: DQO 5: Transformer House Investigation – Soil sample collection around and under Transformer House

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		› No personnel will enter the space beneath the building; all samples will be collected with a pole or other device that prevents the need to enter confined space
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in soil (heavy metals)
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
√	Ionizing Radiation	2	› XRF Field Screening Instrument emits X-rays
	Abandoned Explosives		›
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including XRFs, moisture meter, GPS, iPads
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #6

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by: Brie Hicknell	**Date:** 7/19/2021

Job Task: DQO 6: Surface water and sediment sample collection from National Creek

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment to/between sampling locations
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in surface water and sediment
√	Steep, Uneven Terrain	1	› Sampling locations may be located in steep, uneven terrain
	Ionizing Radiation		›
	Abandoned Explosives		›
√	Traffic	1	› Potential traffic in sampling area, potential impact risk
√	Electrical Hazards	1	› Various electrical equipment including GPS, iPads, YSI
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Walking between locations, sometimes on steep terrain › Carrying equipment between sampling locations
√	Hazardous Plants/Insects	1	› Sampling locations may be located near hazardous plants/insects
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
√	Surface Water	1	› Sampling will occur in National Creek
√	Corrosives	1	› Preservatives in sample containers may be corrosive

Hazard Assessment Form – Job Task #7

Kennecott Mill Town Site Phase I RI

Kennecott, AK VHB Project #58345.43

Conducted by: Brie Hicknell **Date:** 7/19/2021

Job Task: Equipment and personnel decontamination

Place a check mark (√) next to every category that could pose a hazard to employees working in this area. Identify the sources responsible for contributing to this hazard. Upon completion, rate each hazard category on a scale of 0-4. (0=no hazard, 1=slight hazard, 2=moderate hazard, 3=high hazard, 4=extreme hazard).

Category:		Hazard Rating:	Sources:
√	Harmful Dust	1	› May experience dusty conditions during high winds and dry conditions
	Confined Space		›
	Heavy Equipment		›
	Moving Parts		›
√	Heavy Lifting	1	› Carrying equipment and pails of decontamination water
	Overhead Hazards		›
√	Hand Tools	1	› Hand tools may be sharp
	Fall over 6 feet		›
√	Cuts and Lacerations	1	› Hand tools may be sharp
√	Chemical Exposure	1	› Exposure to chemicals in decontamination water
√	Steep, Uneven Terrain	1	› May carry equipment and pails of decontamination water across steep, uneven terrain
	Ionizing Radiation		›
	Abandoned Explosives		›
√	Traffic	1	› Potential traffic in decontamination area, potential impact risk
	Electrical Hazards		›
	Excavation/Trenching		›
	Noise		›
√	Slip, Trip, Falls	1	› Carrying equipment and pails of decontamination water
√	Hazardous Plants/Insects	1	› Hazardous plants/insects may be near decontamination area
√	Dangerous Animals	2	› Dangerous animals may be present at the Site
√	Heat Stress	1	› Heat stress is possible in Alaska during August
√	Cold Stress	1	› Cold stress is possible in Alaska during August
√	Extreme Weather	1	› Extreme weather is possible in Alaska during August
	Burns		›
	Surface Water		›
	Corrosives		›

Appendix C – Activity Hazard Analysis

Activity Hazard Analysis : Job Task #1

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 1: Ambient Air Investigation › Composite soil sample collection › Air sample collection › Carrying sampling equipment › Setting up equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #2

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 2: Historical Paint Investigation › Soil sample collection and screening using XRF around Mill Town Buildings › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Radiation exposure	Review and follow the XRF safety procedures (described in Section 5.5) and the XRF Field SOP prior to the use of the XRF Instrument. Complete ionizing radiation safety training prior to using XRF.
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Abandoned Explosives	Attend required safety awareness training by WRST Regional Blasting Officer prior to working in blasting cap disposal areas. Always be accompanied by the WRST Regional Blasting Officer when in or near blasting cap disposal areas.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #3

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 3: Mill Tailings and Ore/Ore Concentrates Investigation › Soil sample collection and screening using XRF on and around tailings piles › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Radiation exposure	Review and follow the XRF safety procedures (described in section 5.5) and the XRF Field SOP prior to the use of the XRF Instrument. Complete ionizing radiation safety training prior to using XRF.
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Abandoned Explosives	Attend required safety briefing by WRST Regional Blasting Officer prior to working in blasting cap disposal areas. Always be accompanied by the WRST Regional Blasting Officer when in or near blasting cap disposal areas.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #4

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 4: Blasting Caps Investigation › ISM soil sampling while coordinating with WRST Regional Blasting Officer › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Abandoned Explosives	Attend required safety briefing by WRST Regional Blasting Officer prior to working in blasting cap disposal areas. Always be accompanied by the WRST Regional Blasting Officer when in or near blasting cap disposal areas.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #5

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 5: Transformer House Investigation › Soil sample collection around and under Transformer House › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Radiation exposure	Review and follow the XRF safety procedures (described in section 5.5) and the XRF Field SOP prior to the use of the XRF Instrument. Complete ionizing radiation safety training prior to using XRF.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #6

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
DQO 6: National Creek Investigation › Surface water and sediment sample collection from National Creek › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Activity Hazard Analysis : Job Task #7

Principal Activities	Potential Safety/Health Hazards	Recommended Controls
Equipment and personnel decontamination › Carrying equipment	*Physical Hazards:*	
	Slip, Trips, Falls/Steep, Uneven Terrain	Use safe work practices and good housekeeping: Keep tools and materials from underfoot. Route extension cords so they lay flat on the ground and do not cross heavily trafficked areas.
	Heavy Lifting	Use proper techniques when lifting or moving heavy objects: bend at the knees, do not twist, and keep the load as close as possible. Use others for assistance when possible or utilize manual assist methods (dollies, carts, etc.).
	Cuts and Lacerations/Hand Tools	Wear appropriate clothes, gloves, and footwear when using cutting tools, and wear eye protection when moving through trees and underbrush. When cutting, keep your body and appendages out of the way, use only sharp blades so you can reduce the force required, and secure thick material so it doesn't slip. Wear proper footwear in areas of debris/debris fields to protect from puncture.
	Electrical Hazards	Check that sampling pumps have a grounded multi-contact plug and receptacle. Always use a GFCI with an extension cord in potentially hazardous conditions such as wet environments or outside.
	Traffic	Be aware of surroundings, wear high-visibility clothing when working around roadways.
	Extreme Weather/Heat Stress/ Cold Stress	Dress accordingly for the weather. If extreme weather occurs, stop work and muster in a safe location (indoors, in vehicles). Know the symptoms of heat and cold stress and take frequent breaks to cool down or warm up as needed.
	Chemical Hazards:	
	Harmful Dust	If the dust content in the air is high, use respiratory protection PPE (e.g. dust mask) to limit the exposure. Use water mist to control dust during sample collection when possible. Review Section 5.4 for known or suspected PCOPCs.
	Chemical Exposure/Corrosives	Use PPE to limit contact risk. Review Section 5.4 for known or suspected PCOPCs.
	Biological Hazards:	
	Hazardous Plants/Insects	Wear long sleeve clothing and apply insect repellant (with 15 to 30% concentration of DEET) when necessary.
	Dangerous Animals	Maintain a safe distance from dangerous animals. Follow NPS protocols if bears or moose are encountered.

Appendix D – Accident Investigation Form

SSO'S ACCIDENT INVESTIGATION REPORT

Injured Employee _____ Job Title _____

Date/Time of Accident _____

Location of Accident_____

Witnesses to the Accident _____

Injury Incurred? _____ Nature of Injury _____

Engaged in What Task When Injured? _____

_____

_____

Will Lost Time Occur? _____ How Long? _____ Date Lost Time Began_____

Were Other Persons Involved/Injured? _____

How Did the Accident Occur? _____

_____

_____

_____

What Could Be Done to Prevent Recurrence of the Accident? _____

_____

_____

_____

What Actions Have You Taken Thus Far to Prevent Recurrence? _____

_____

_____

_____

SSO's Signature: _____ Date: _____

SSO's Printed Name: _____

Note: If the space provided on this form is insufficient, provide additional information on a separate page and attach.

Appendix E – Current Training Certificates and Qualifications

Personnel Verification of Clearance/Credentials

Employee Name	Training Certifications (e.g., OSHA HAZWOPER, First Aid/CPR, etc.) (Type, Date)	Issues/ Concerns
Benjamin Bliss	OSHA 40 Hour HAZWOPER Training, May 28, 2019 8 Hour HAZWOPER Refresher Training, May 11, 2021 Ionizing Radiation Hazards Training, July 30, 2021	
Brie Hicknell	OSHA 40 Hour HAZWOPER Training, August 7, 2017 8 Hour HAZWOPER Supervisor Refresher Training, July 9, 2021 Ionizing Radiation Hazards Training, July 22, 2021	
Don Maynard	OSHA CFR 1910.120 40-Hour Training Program for Hazardous Waste Operations, April, 1990 8-Hour Hazardous Waste Operations Refresher, February 16, 2021 Ionizing Radiation Hazards Training, July 30, 2021	
Adam Robtoy	40-Hour HAZWOPER Training for General Site Workers, April 10, 2000 8-Hour Hazardous Waste Operations Refresher, February 16, 2021 Ionizing Radiation Hazards Training, March 11, 2021	
Emily Rodriguez	OSHA 40 Hour HAZWOPER Training, July 31, 2020 8 Hour HAZWOPER Refresher Training, July 11, 2021 Ionizing Radiation Hazards Training, July 26, 2021	

Certificate of Completion

This certifies that

Benjamin R. Bliss

has successfully completed

OSHA 40 Hour HAZWOPER Training

Annual Refresher Training Required

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)

And State OSHA/EPA Regulations as well including 29 CFR 1926.65(e)

This course is approved for 40 Contact Hours (4 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044)

Julius P. Griggs

Julius P. Griggs
Instructor #892

1905281286476

Certificate Number

5/28/2019

Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley,CA 93063
(888) 309-SAFE (7233) or 805 306-8027
https://www.safetyunlimited.com

Scan this code or visit www.safetyunlimited.com/v to verify certificate.

Annual Refresher Training Required

Certificate of Completion

This certifies that

Benjamin R. Bliss

has successfully completed

8 Hour HAZWOPER Refresher Training

Refresher certification does NOT necessarily indicate initial 24 or 40 Hour HAZWOPER certification

In Accordance w/Federal OSHA Regulation 29 CFR 1910.120(e) & (p)

And all State OSHA/EPA Regulations as well including 29 CFR 1926.65 for Construction.

This course (Version 3) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044).

Safety Unlimited, Inc., Provider #5660170-2, is accredited by the International Association for Continuing Education and Training (IACET) and is accredited to issue the IACET CEU. As an IACET Accredited Provider, Safety Unlimited, Inc. offers CEUs for its programs that qualify under the ANSI/IACET Standard. Safety Unlimited, Inc. is authorized by IACET to offer 0.8 CEUs for this program.

Julius P. Griggs

Julius P. Griggs
Instructor #892

2105115286476

Certificate Number

5/11/2021

Issue Date

ACCREDITED IACET PROVIDER

Scan this code or visit safetyunlimited.com/v to verify certificate.

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley,CA 93063
(855) 784-2677 or 805 306-8027
https://www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training

COMPLIANCE TRAINING ONLINE.com

Certificate of completion

Presented to:

Benjamin Bliss

Ionizing Radiation Hazards

OSHA 29 CFR 1910.1096 Training

certificate number: 034E7BFA-F160-11EB-95C6-0050568D5CA3

Trained & Tested at: www.ComplianceTrainingOnline.com

HRS/CEU/CME: 2.0/0.2/2.0

Tested On: 7/30/21
Valid Thru: 7/30/22

Signed: CTO

Certificate of Completion

This certifies that

Brianne N Hicknell

Has Successfully completed

OSHA 40 Hour HAZWOPER Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)

And State OSHA/EPA Regulations as well including 29 CFR 1926.65(e)

This course is approved for 40 Contact Hours (4 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044)

Julius P. Griggs

Julius P. Griggs
Training Director

1708071207734

Certificate Number

8/7/2017

Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Annual Refresher Training Required

Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certificate of Completion

This certifies that

Brianne N. Hicknell

has successfully completed

8 Hour HAZWOPER Supervisor Refresher Training

This certification alone does NOT indicate INITIAL 8 Hour OSHA Supervisor Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)(8)

And all State OSHA/EPA Regulations as well including 29 CFR 1926.65 for Construction.

This course (Version 3) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044)

Safety Unlimited, Inc., Provider #5660170-2, is accredited by the International Association for Continuing Education and Training (IACET) and is accredited to issue the IACET CEU. As an IACET Accredited Provider, Safety Unlimited, Inc. offers CEUs for its programs that qualify under the ANSI/IACET Standard. Safety Unlimited, Inc. is authorized by IACET to offer 0.8 CEUs for this program.

Julius P. Griggs

Julius P. Griggs
Instructor #892

2107095207734

Certificate Number

7/9/2021

Issue Date

ACCREDITED IACET PROVIDER

Scan this code or visit safetyunlimited.com/v to verify certificate.

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley,CA 93063
(855) 784-2677 or 805 306-8027
https://www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training

COMPLIANCE TRAINING ONLINE.com

Certificate of completion

Presented to:

Brianne Hicknell

Ionizing Radiation Hazards

OSHA 29 CFR 1910.1096 Training

certificate number: 0DF6663F-EB06-11EB-95C6-0050568D5CA3

Trained & Tested at: www.ComplianceTrainingOnline.com

HRS/CEU/CME: 2.0/0.2/2.0

Tested On: 7/22/21
Valid Thru: 7/22/22

Signed: CTO

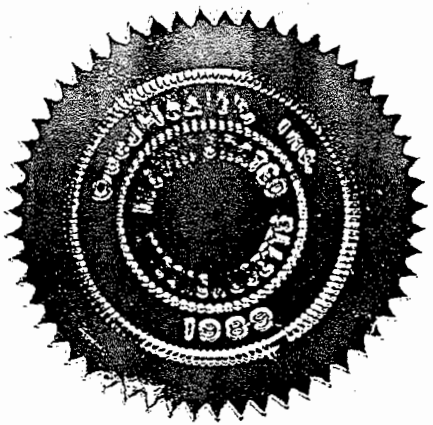
Certificate of Achievement
Presented
To

Don Maynard

For the successful completion of a 40 - hour training program
in health and safety, which meets OSHA requirements under
29 CFR 1910.120 for hazardous waste operations.
Course Given By OccuHealth, Inc., Mansfield, Mass. (508) 339-9119

Thomas E Hamilton

Thomas E. Hamilton, CIH
Course Director, President



April 1990

This is to certify that

Donald Maynard

has attended the 8-hour eLearning course by video conference

Hazardous Waste Operations Refresher

pursuant to OSHA 29 CFR Part 1910.120

Course Location

Zoom Video Conference

Institute for Environmental Education 16 Upton Drive Wilmington, MA 01887

February 11& 16, 2021
Course Dates

21-4115-182-274674
Certificate Number

February 16, 2021
Examination Date

February 16, 2022
Expiration Date

Training Director

16 Upton Drive, Wilmington, MA 01887 Telephone 978.658.5272 www.ieetrains.com

INSTITUTE FOR ENVIRONMENTAL EDUCATION

COMPLIANCE TRAINING ONLINE.com

Certificate of completion

Presented to:

Donald Maynard

Ionizing Radiation Hazards

OSHA 29 CFR 1910.1096 Training

certificate number: 518AD0BE-F137-11EB-95C6-0050568D5CA3

Trained & Tested at: www.ComplianceTrainingOnline.com

HRS/CEU/CME: 2.0/0.2/2.0

Tested On: 7/30/21
Valid Thru: 7/30/22

Signed: CTO

The UNIVERSITY of VERMONT

Adam Robtoy

has completed

40 hour HAZWOPER training for General Site Workers,

in accordance with the requirements of the
OSHA Hazardous Waste Operations and Emergency Response Standard, 29 CFR 1910.120.
Presented

April 10, 2000

by

Environmental Safety Facility
Risk Management Department
University of Vermont

Francis Churchill
Environmental Safety Specialist

Ralph Stuart, CIH
Environmental Safety Program Coordinator

This is to certify that

Adam Robtoy

has attended the 8-hour eLearning course by video conference

Hazardous Waste Operations Refresher

pursuant to OSHA 29 CFR Part 1910.120

Course Location

Zoom Video Conference
Institute for Environmental Education 16 Upton Drive Wilmington, MA 01887

February 11& 16, 2021
Course Dates

21-4115-182-274676
Certificate Number

February 16, 2021
Examination Date

February 16, 2022
Expiration Date

Training Director

16 Upton Drive, Wilmington, MA 01887 Telephone 978.658.5272 www.ieetrains.com

INSTITUTE FOR ENVIRONMENTAL EDUCATION

COMPLIANCE TRAINING ONLINE.com

Certificate of completion

Presented to:

Adam Robtoy

Ionizing Radiation Hazards

OSHA 29 CFR 1910.1096 Training

certificate number: C5751899-82A7-11EB-95C6-0050568D5CA3

Trained & Tested at: www.ComplianceTrainingOnline.com

HRS/CEU/CME: 2.0/0.2/2.0

Tested On: 3/11/21
Valid Thru: 3/11/22

Signed: CTO

Certificate of Completion

This certifies that

Emily E. Rodriguez

has successfully completed

OSHA 40 Hour HAZWOPER Training

Annual Refresher Training Required

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)

And State OSHA/EPA Regulations as well including 29 CFR 1926.65(e)

This course is approved for 40 Contact Hours (4 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044)

Safety Unlimited, Inc., Provider #5660170-2, is accredited by the International Association for Continuing Education and Training (IACET) and is accredited to issue the IACET CEU. As an IACET Accredited Provider, Safety Unlimited, Inc. offers CEUs for its programs that qualify under the ANSI/IACET Standard. Safety Unlimited, Inc. is authorized by IACET to offer 4 CEUs for this program.

Julius P. Griggs

Julius P. Griggs
Instructor #892

2007311338461

Certificate Number

7/31/2020

Issue Date

ACCREDITED IACET PROVIDER

Scan this code or visit safetyunlimited.com/v to verify certificate.

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley,CA 93063
(888) 309-SAFE (7233) or 805 306-8027
https://www.safetyunlimited.com

Annual Refresher Training Required

Certificate of Completion

This certifies that

Emily E. Rodriguez

has successfully completed

8 Hour HAZWOPER Refresher Training

Refresher certification does NOT necessarily indicate initial 24 or 40 Hour HAZWOPER certification

In Accordance w/Federal OSHA Regulation 29 CFR 1910.120(e) & (p)

And all State OSHA/EPA Regulations as well including 29 CFR 1926.65 for Construction.

This course (Version 2) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) (Accreditation # 044).

Safety Unlimited, Inc., Provider #5660170-2, is accredited by the International Association for Continuing Education and Training (IACET) and is accredited to issue the IACET CEU. As an IACET Accredited Provider, Safety Unlimited, Inc. offers CEUs for its programs that qualify under the ANSI/IACET Standard. Safety Unlimited, Inc. is authorized by IACET to offer 0.8 CEUs for this program.

Julius P. Griggs

Julius P. Griggs
Instructor #892

2107125338461

Certificate Number

7/12/2021

Issue Date

ACCREDITED IACET PROVIDER

Scan this code or visit safetyunlimited.com/v to verify certificate.

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley,CA 93063
(855) 784-2677 or 805 306-8027
https://www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training

COMPLIANCE TRAINING ONLINE.com

Certificate

of completion

Presented to:

Emily Elizabeth Rodriguez

Ionizing Radiation Hazards

OSHA 29 CFR 1910.1096 Training

certificate number: C3603E4A-EE2D-11EB-95C6-0050568D5CA3

Trained & Tested at: www.ComplianceTrainingOnline.com

HRS/CEU/CME: 2.0/0.2/2.0

Tested On: 7/26/21
Valid Thru: 7/26/22

Signed: CTO

Appendix F – VHB COVID Guidance

Updated July 30, 2020

COVID-19 | Field Work Protocol

During the COVID-19 pandemic, VHB employees and subcontractors will continue to perform field work, including survey, natural resource assessments, site walks, construction monitoring, and other tasks under contract that are allowed under local jurisdictional essential-work guidelines.

We have established guidance and protocols to make sure that all work—in VHB offices, on site, or in the field—is completed in a safe manner. Employees must follow VHB's Essential Work Protocol as well as the specific field work guidance below. These protocols are subject to change and Employees and subconsultants must adhere to the protocols that are published at the time that the work is being conducted.

In addition to the normal health and safety requirements that are appropriate to the specific task, VHB employees and subconsultants must follow the stricter of any local field work requirements or these VHB field work protocols.

Employees are required to sign off that this protocol was reviewed and adhered to for every field assignment. Documentation will be logged in the project folder. Email verification that the protocols have been read is acceptable.

Employees are required to complete daily health screenings to conduct field work and must notify Managing Directors for any work-related non-office trip such as a field visit. Unlike office visits, these requests will be auto approved unless there are any questions you are not able to confirm.

Employees performing field work in areas with local non-essential travel restrictions are required to carry a letter from VHB documenting the essential nature of the work.

Protocols & Guidance

Daily health screenings are required to complete field work. Click here to notify Managing Directors for any work-related non-office trip such as a field visit. Unlike office visits, these requests will be auto approved unless there are any questions you are not able to confirm.

In accordance with CDC's latest guidance, wear cloth face coverings (homemade or self-provided) in public settings where social distancing measures are difficult to maintain, especially in areas of significant community-based transmission. The cloth face coverings recommended by CDC are not surgical masks or N-95 respirators. Those critical supplies must be reserved for healthcare workers and other medical first responders. For guidance on making a cloth face covering, please click here.

Trip Planning: Think through the entire trip carefully before heading out: How to get there, where are you staying, and how you will eat. Assume stores will not be open and bring everything you need from home.

Limit field crews as much as possible to minimize interaction with others. Where work can be safely conducted by one person, please do so. Maintain a communication plan if you are working alone.

Identify field crews (preferably of two people) and ask them to partner on every project to minimize the potential for spreading a potential infection.

Equipment: To the extent possible, equipment should be assigned to each field crew and stay with them for the duration. As equipment supplies allow, assign individuals their own equipment for the duration of the need. If you need to purchase additional equipment, please contact your supervisor.

Properly sanitize VHB tablets, GPS units, and other shared field equipment.

Travel to sites in separate vehicles to maintain social distancing while commuting.

Follow sanitation and disinfecting procedures for VHB vehicles. This includes equipping each vehicle with disinfecting wipes and hand sanitizer and implementing a disinfecting protocol that includes wiping down the steering wheel, levers, knobs, handles, and keys before and after each use.

Rental vehicles should be avoided if possible.

While fueling vehicles, employees must wear gloves or wash hands after using the pump at a gas station before getting back into your vehicle.

Individuals and individuals with household members who develop cold or flu-like symptoms should stay home for a minimum of 14 days before returning to work in the field.

Field work requiring overnight stays, regardless of whether or not there are local non-essential travel restrictions in effect, must be approved by the Regional Manager. Overnight stays must be limited to national hotel chains that have published their cleaning protocols and have committed to following CDC guidance. Employees should wipe down high-contact surfaces in their hotel rooms and decline daily housekeeping services to prevent the spread of germs. All meals must either be brought from home or purchased from a retailer serving take-out food. Employees should maintain social distancing at all times and are encouraged to eat their meals individually in their hotel room or vehicles. (Hilton and Marriott chains appear to be following CDC guidance.)

Employees who feel uncomfortable due to a situation in the field where they are unable to maintain appropriate social distancing, whether due to the nature of the task or the behavior of other nearby people, should follow these protocols and contact their Managing Director for guidance. The safety of our people is the highest priority. Do not put yourself in a hazardous situation to expedite the completion of a work task.

Field crews are permitted to open manhole covers after participating a training held by the Health and Safety Group.

Be mindful of regular safety precautions in addition to COVID-19 field precautions.

Appendix G – Bear and Moose Guidance

Bear Safety
In Alaska's National Parklands

National Park Service
U.S. Department of the Interior

Bears and campers often frequent the same areas in Alaska's national parks. In coastal parks, both tend to spend time on the beach, the narrow band of land found between the sea and the brush, forest, or steep cliffs. Bears prefer these areas because they often contain abundant vegetation for grazing and make travel easy, while campers prefer these areas for cooking and because they offer easy access to kayak travel. Inland parks are also home to bears and it is important that campers respect their space.

It is likely that bears and campers will encounter one another, but by remaining calm and following the basic advice of experienced bear behaviorists, you increase the odds of a positive outcome for both you and the bear.

Some parks require campers to attend an orientation at the Visitor Information Station. During this orientation a park ranger will inform you about areas that are closed to camping due to high bear activity or recent bear/human encounters. If the park you are visiting does not have a bear safety orientation, take the time to read this brochure and learn how you can camp safely in bear country. Contact park staff to obtain current information on bear safety issues. Once in the backcountry you are on your own. Some parks require you to obtain a camping permit and may issue free bear-resistant food containers (BRFC).

Cover photo © Robert Sabin

Bear Signs to Watch for and Areas to Avoid

It is important to be "bear aware" when camping and hiking in Alaska's national parks and to avoid seasonal bear foraging areas (sedge meadows, berry patches, etc.). Bear signs are easy to find if you know what to look for. Select a campsite with the least amount of bear sign and away from seasonal bear foraging areas. Here are some signs to be on the lookout for:

Bear scat or tracks that seem very recent or abundant. Be alert.

Trails are formed because of consistent use. Bears will often follow the path of least resistance, for example, lakeshores and ridgelines.

A tree or log that has bear hair or claw marks may indicate that it is a repeatedly used bear rub-tree.

Large dug up areas could be forage sites, day beds, or belly holes.

Avoid salmon streams! Bears like fish. And a noisy stream may lessen your ability to hear a bear or for a bear to hear you.

Narrow beaches with steep cliffs or extremely dense brush do not allow a bear to pass safely. Remember that at high tide a bear will not have as much room to pass between your camp and the high water line.

Neither you nor the bear want to be surprised by the other. Avoid areas with restricted visibility and make noise when exiting your tent.

Ask yourself: Can a bear walk by and pass my campsite and cooking area unhindered?

Photo © John Schoen

Cooking and Storing Food

Keep all food and cosmetics in the BRFC when not in use. Place any snacks, wrappers, lip balm, sunscreen, etc. that were used while kayaking or hiking into the BRFC before entering your tent. It is not a good idea to store food in kayaks overnight. At night, store your BRFC and clean cooking gear off of main animal trails, (in coastal parks above high tide line) and at least 100 yards from your tent and hidden in thick brush or behind rocks.

Prepare and consume food at least 100 yards from your tent site and food storage area. Try to select cooking areas where you can see a comfortable distance to minimize the risk of a surprise encounter with a bear passing through the area.

National Park Service photograph

If camping in a coastal park, prepare and eat all food in the intertidal zone, that area below the seaweed debris line and the waterline. Cook and eat as close to the water as possible so cooking smells and any food particles will then be washed away by the next tide.

Be prepared to quickly stow all food back into the BRFC if a bear should suddenly approach. Keep your gear together — minimize the amount of space that you occupy. Always ask yourself, "Is there room for a bear to get around us?" Or "Can I quickly get all this gear under my control?"

Photo © Terry D. DeBruyn

Minimize Bear Disturbance and Displacement

If you see a bear and it does not see you, back away out of sight and change your course. Move out of the area or quietly observe the bear at a safe distance without approaching or otherwise disturbing it. Disturbance is evident whenever a bear changes its behavior because of you. If it stops eating and looks up, sniffs the air with ears erect, trying to locate you, you are too close! Bears only have 6-8 months to acquire the calories and fat reserves needed for the entire year. Give them space!

While many bears seem to be tolerant of human presence at distances farther than 100 yards, each animal and situation is different. Pay attention to the bear's behavior and respect its right to feed and travel undisturbed. Use telephoto lenses and binoculars. Allow bears to pass by your camp undisturbed. If you have made sure that the bear is aware of your presence so it is not surprised and have kept all your gear under your direct control, allow the bear to pass by unhindered. You may just be afforded the opportunity to safely observe this amazing creature in its natural environment.

Photo © John Hyde

Do not pursue or harass bears for the sake of a close encounter or photograph, either on land or from your watercraft.

Minimize the Risk of Having Your Gear Destroyed

Photo © Robert Sabin

When choosing your tent site avoid areas frequented by bears, camping in travel corridors (e.g., river corridors and trails), and do not camp in the intertidal zone.

Do not leave gear unattended. This includes tents, clothes, pads, water bottles, etc. Consider using a portable electric fence to discourage bears from investigating your camp.

Keep gear together and under your immediate control. In coastal areas minimize the amount of space you take up on a beach. Again, ask: "Is there room for a bear to get around our camp?" A Tip: set up tents after making dinner and take them down before breakfast in order to keep all gear with or very near you in the intertidal when cooking or packing up kayaks.

Elevate your reaction if a bear elevates its inquisitiveness. If a bear approaches make noise, wave your arms, etc. Stand your ground! Never surrender your gear to a bear!

Handling a Bear Encounter

National Park Service
U.S. Department of the Interior

Basic Bear Safety Tips

As the number of visitors to bear country in Alaska increases so does the number of human/bear encounters. The vast majority of these encounters do not result in human injury or fatality. However, a much larger proportion of these encounters do result in the bear's death. You can help prevent injury to yourself, to others, and to the bear by taking a few basic precautions.

Stay Alert
Stay constantly alert in bear country. Use your ears, eyes, and even your nose to detect the presence of a bear. The sooner you are aware of the bear, the more time you and the bear will have to react appropriately.

Be Visible, Make Noise
A surprise encounter with a bear is dangerous and can be terrifying. However, you can reduce the potential for such encounters. Avoid surprises by traveling in open areas with good visibility. Make noise as you walk, particularly in thick brush, or when rounding a blind corner — talk, clap, and sing. Be extra alert in windy conditions or near noisy streams or beaches that may mask your sounds. When possible, travel with the wind at your back.

Safety in Numbers
The larger your group is, the less risk of a bear attacking. Stay together as a group, particularly if visibility is poor.

Avoid Bears
If possible change your course to avoid the bear or move out of the area slowly. Never approach a bear, even from your boat or kayak. Approaching bears is dangerous and can cause undue stress and disturbance to the bear increasing the risk of attack.

Store Food Properly
Keep all food and scented items under your immediate control at all times. While camping, keep a clean camp and store food appropriately.

Report Bear Encounters
If you have an encounter, report it to a park ranger as soon as possible. This will alert others and enable park staff to respond appropriately to the situation if necessary.

Photo © Terry D. DeBruyn

What Should You Do if You Encounter a Bear?

First assess the situation. Does the bear see you? If the bear does not see you, simply move away from the bear and the encounter is over.

Non-defensive Bear Encounters

- If the bear is aware of you and either looking or not looking at you, or moving steadily along a route, this can be considered non-defensive behavior.

What if you are hiking or kayaking and you encounter a non-defensive bear?

- Change your course to avoid the bear. Increase your distance from the bear and stay alert to the bear's whereabouts.

What if you are camping, cooking, or eating and you encounter a non-defensive bear?

- Keep all of your gear under your direct control.
- Make sure the bear is aware of your presence. Talk calmly to the bear and stand your ground!
- If you are in a group, stay together without blocking the bear's route. If the bear is not focused on you allow the bear to pass peacefully.
- If the bear approaches and is focused on you, stand together and elevate your defensive actions by shouting, yelling, and waving your arms. Do not retreat from the bear. You can also use noisemakers like air horns or bang pots and pans.
- If the bear departs the encounter ends.
- If the bear continues to approach, stand your ground and remain assertive.
- If a bear charges you stand your ground and remain assertive. Make yourself look big. Most non-defensive charges do not end in contact.
- Now is the time to use bear pepper spray if you have it!
- If the bear makes contact fight back vigorously! This is likely a predatory attack. Kick, punch, or hit the bear's face, eyes and nose.

Defensive Bear Encounters

- Defensive behaviors arise when bears are defending food or female bears are defending their offspring. Defensive encounters usually occur suddenly and at close distances.
- Defensive behavior may include snorting, huffing, jaw popping, and charging.

Photo © John Hyde

- If you note any of these behaviors STOP AND STAND YOUR GROUND. Your safety lies in calming the bear.
- Talk calmly to the bear; move slowly away diagonally if the bear is stationary.
- Continue to monitor the bear as you move from the area.
- If the bear renews its advance stop and stand your ground again. Talk calmly to the bear.
- If the bear charges remain non-threatening and stand your ground. Most charges do not end in contact. Now is the time to use bear pepper spray if you have it!

What if the bear makes contact during a defensive encounter?

- IF IT IS A BROWN BEAR, PLAY DEAD: Lie face down with your hands clasped behind your neck and legs spread apart so the bear can't turn you over. Do not move until the bear leaves the area. If the attack is prolonged and the brown bear begins to feed on you, fight back vigorously! The encounter has now likely changed from a defensive one to a predatory one.
- IF IT IS A BLACK BEAR, DO NOT PLAY DEAD: Fight back vigorously!
- NEVER PLAY DEAD WITH A BLACK BEAR! Most black bear attacks are predatory.
- FIGHT ANY BEAR THAT ATTEMPTS TO ENTER YOUR TENT!!

Left: Standing brown bear, photo © Robert Sabin