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# FINAL WORK PLAN, SEDIMENT INVESTIGATION AND BATHYMETRIC SURVEY FOR PARCEL F

11/20/2017

ECC - INSIGHT, LLC

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# FINAL WORK PLAN
# Sediment Investigation and Bathymetric Survey for Parcel F

## Hunters Point Naval Shipyard
## San Francisco, California

Contract No.: N62473-12-D-2004
Contract Task Order No.: 0014
Document Control No.: INEC-2004-0014-0003

November 2017

Prepared for:

Department of the Navy
Base Realignment and Closure
Program Management Office West

Prepared by:

ECC-Insight LLC
2749 Saturn Street
Brea, CA 92821

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Appendix C Waste Management Plan **(on Compact Disk)**

Appendix D Response to Comments **(on Compact Disk)**

## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| AWA | area-weighted average |
| bml | below mudline |
| BRAC | Base Realignment and Closure |
| BCT | BRAC Cleanup Team |
| CAD | computer-aided design |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| COC | contaminant of concern |
| CSM | Conceptual Site Model |
| ECC-Insight | ECC-Insight LLC |
| EPA | Environmental Protection Agency |
| ER-M | Effects Range-Median |
| FS | Feasibility Study |
| FSDG | Feasibility Study Data Gap |
| GPS | Global Positioning System |
| HPNS | Hunters Point Naval Shipyard |
| IDW | investigation-derived waste |
| mg/kg | milligrams per kilogram |
| MLLW | mean lower low water |
| NOAA | National Oceanic and Atmospheric Administration |
| PCB | polychlorinated biphenyl |
| PRG | preliminary remediation goal |
| RTK | Real Time Kinematic |
| RAO | remedial action objective |
| ROD | Record of Decision |
| SAP | Sampling and Analysis Plan |
| sf | square feet |
| USACE | United States Army Corps of Engineers |

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# 1.0 INTRODUCTION

This Work Plan was prepared by ECC-Insight LLC (ECC-Insight) and CDM Smith to describe field activities necessary to perform sediment sampling and bathymetric survey activities of offshore sediments at Hunters Point Naval Shipyard (HPNS) Parcel F, San Francisco, California, hereafter, referred to as the Site. This work is being conducted for Naval Facilities Engineering Command Southwest under Contract No. N62473-12-D-2004, Task Order No. 0014.

Field activities include collection of grab sediment samples from the seafloor surface within six separate site areas where Former Parcel B and C pier and wharf structures have been removed (ERS, 2012). The sediment sampling effort is in response to regulatory comments made by the Base Realignment and Closure (BRAC) Cleanup Team (BCT) on the Removal Action Completion Summary Report, Pier Radiological Surveys and Removal (ERS, 2012) (Attachment 1). In addition, a bathymetric survey will be conducted within several subareas of Parcel F. The data will be used to fill identified data gaps in the Conceptual Site Model (CSM), address regulatory comments on the removal action at the former Parcel B and C piers and wharf structures, and refine the remedial alternatives for Parcel F in support of the Proposed Plan, Record of Decision (ROD) and the Remedial Design.

## 1.1. Regulatory Framework and History

HPNS is a former Naval shipyard located on a peninsula in southeast San Francisco that extends east into San Francisco Bay (Figures 1-1 and 1-2). The land side portion of HPNS, which is approximately 420 acres, was purchased by the Department of the Navy in 1939 and operated as a shipbuilding, repair and maintenance facility by the Navy and several tenant companies until 1989. Past shipyard operations left hazardous materials on site and as a result, HPNS was listed on the National Priorities List in 1989. HPNS was designated for closure in 1991 and its mission as a Navy shipyard ended in April 1994. Ongoing closure activities at HPNS involve conducting environmental remediation and making the property available for nondefense use such as residential and commercial development. Environmental restoration activities at HPNS are being conducted in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) as amended by the Superfund Amendments and Reauthorization Act of 1986.

Parcel F comprises the offshore portion of HPNS and is approximately 446 acres of underwater property. This proposed investigation focuses on Areas III (Point Avisadero Area), IX (Oil Reclamation Area), and X (South Basin Area), as well as the footprint of six former Parcel B and C piers and wharf structures located within Parcel F (Figures 1-2 and 2-1). Area III is an open water area within San Francisco Bay located south of the Islais Creek channel entrance with water depths of up to 70 feet. Area IX/X is a shallow embayment located to the south of HPNS between HPNS and Candlestick Point with water depths ranging from less than 2 to 6 feet (Battelle, BBL, and Neptune & Company, 2005).

The six former Parcel B and C pier and wharf structures include Submarine Piers B and C, portions of the submarine quay wall, Berths 61 and 64, and Wharf No.2. The structures are located adjacent to the shoreline along Parcel B, and within and adjacent to portions of offshore Areas I and III (Figure 2-1).

### 1.2. Work Plan Overview

Additional characterization of sediment and the bathymetric survey are necessary to address BCT comments regarding a potential data gap for sediment sampling of non-radiological contaminants of concern (COCs) and change in bathymetry/erosional patterns following removal of the six former Parcel B and C pier and wharf structures (ERS, 2012; Attachment 1). In addition, updated bathymetric surveys are necessary to accurately estimate the areas and volume of sediments requiring remediation in Areas III and IX/X in support of the Parcel F Remedial Design, and to estimate change in sediment bed bathymetry through comparison to previous bathymetric surveys. The survey data will also be used to determine whether the installation of cofferdams is a viable option in Area IX/X.

### 1.3. Work Plan Objectives

The primary objective of this Work Plan is to present the proposed technical approach to collect additional data to address previously identified data gaps associated with offshore sediments at Parcel F. Numerous investigations have been completed in support of Parcel F (Section 2). The Feasibility Study (FS) (Barajas, 2008) and FS Addendum (KCH, 2017) document the latest stage in the CERCLA process for HPNS Parcel F. This document will be used to address BCT comments on the type, quality, and location of data that will be collected to refine the remedial alternatives for Parcel F in support of the Proposed Plan, ROD and Remedial Design.

### 1.4. Work Plan Organization

Following this introduction, the major sections of this Work Plan are organized as follows:

Section 2.0 – Background and Site Conditions: discusses previous investigations, the CSM, and nature and extent of contamination.

Section 3.0 – Technical Approach: discusses the technical approach that will be implemented for the Site.

Section 4.0 – General Site Activities and Requirements: provides a description of the activities to be performed, including notifications in advance of the proposed work.

Section 5.0 – Scheduling and Deliverables: presents the proposed deliverable and field work schedule.

Section 6.0 – References.

Attachment 1– Environmental Protection Agency (EPA) Comments 8 and 9 on the Removal Action Completion Report, Pier Radiological Surveys and Removal (ERS, 2012), pertinent to this investigation.

The following appendices support this Work Plan:

Appendix A Sampling and Analysis Plan (SAP),

Appendix B Environmental Protection Plan,

Appendix C Waste Management Plan, and

Appendix D Response to Comments.

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# 2.0 BACKGROUND AND SITE CONDITIONS

This section includes site-specific description and history, a summary of relevant previous investigations, remedial action objectives, remedial goals, and the current CSM for Parcel F.

## 2.1. Site Description and History

During historical operations at HPNS, Berths 61 and 64, Submarine Piers B and C, and Wharf No. 2 were used for shipbuilding, maintenance and repair. Operations included metal work and sandblasting. COC sources to offshore sediments identified in Area III include stormwater discharge, a drainage tunnel that was used to rapidly drain water from Dry Docks 2 and 3, and surface runoff and groundwater discharge from Site IR-26 in Parcel B (Battelle, BBL, and Neptune & Company, 2005). COCs in Areas IX/X are not discussed in this work plan because sediment sampling is not proposed in these Areas.

## 2.2. Previous Investigations

Since 1991, numerous investigations have taken place to evaluate shoreline and offshore contamination at Parcel F. Detailed summaries of previous investigations are provided in the FS Report (Barajas, 2008) and FS Addendum (KCH, 2017). Key investigations include:

- Ecological risks assessments (PRC, 1994; PRC, 1996),
- 1998 FS (Tetra Tech and LFR, 1998),
- 2000 Validation Study (Battelle, BBL and Neptune & Company, 2005),
- 2002 Shoreline Investigation (SulTech, 2005),
- FS Data Gaps (FSDGs) Investigation (Barajas and Associates, 2007),
- Phase 1 and Phase 2a Radiological Screening Survey (Battelle and Sea Engineering, 2013),
- Phase 2b Radiological Data Gap Investigation (ITSI Gilbane & SAIC, 2013), and
- Final Addendum to the FS (KCH, 2017).

## 2.3. Remedial Action Objectives and Remediation Goals

The FS Report for Parcel F (Barajas, 2008) documents the remedial action objectives (RAOs) and defines the areas of Parcel F that require remediation based on the RAOs. The following RAOs were identified for Parcel F:

> **RAO 1:** Reduce the risk of benthic feeding and piscivorous birds, including surf scoters, to acceptable levels from exposure to copper, lead, mercury, and polychlorinated biphenyls (PCBs) through consumption of contaminated prey and incidental ingestion of sediment.
>
> **RAO 2:** Limit or reduce the potential risk to human health from consumption of shellfish from Parcel F.

**RAO 3:** Limit or reduce the potential biomagnifications of total PCBs at higher trophic levels in the food chain to reduce the potential risk to human health from consumption of sport fish.

The COCs (copper, lead, mercury, and total PCBs) in sediment were identified based on potential risks to ecological receptors (Table 2-1). In addition, numerical remediation goals were developed for the COCs found in Parcel F sediments with the exception of lead. A numerical remediation goal was not calculated for lead because of the uncertainty associated with both the bioavailability and toxicity of lead (Barajas, 2008). However, the National Oceanic and Atmospheric Administration (NOAA) Effects Range-Median (ER-M) value for the protection of the benthic community will be used as a numeric screening value for lead (218 milligrams per kilogram [mg/kg]). An area-weighted average (AWA) of each COC was calculated for each area to evaluate which areas in Parcel F should be carried forward for remedial evaluation. A conservative approach was taken by using the highest chemical concentrations detected within the top 2 feet to calculate the surface-weighted average concentrations. Only Areas III and X exceeded the preliminary remediation goals (PRGs) on an AWA basis. Although Area IX did not exceed the PRGs, a risk management decision was made to include a portion of the northern shoreline with the evaluation of Area X, which is referred to as Area IX/X (Barajas, 2008).

An Addendum to the FS for Parcel F was finalized in January 2017, which included results from a three-phased data gap investigation for radionuclides in sediment. The FS Addendum investigation included collection and laboratory analysis of more than 800 sediment samples for radionuclides of concern and concluded that no radioactivity in excess of naturally occurring background levels were present within sediment at Parcel F. No additional radiological investigation, cleanup, or controls for radiological contamination in sediment were recommended. However, institutional controls were recommended to address the potential for radiological objects in dredged sediments (KCH, 2017).

## 2.4. Previous Remedial Activities

A detailed summary of previous remedial activities associated with Parcel F is provided in the FS Report (Barajas, 2008) and FS Addendum (KCH, 2017). Several source control measures have been implemented along the HPNS shoreline to protect against releases to each subarea of Parcel F. Source removal activities conducted in the vicinity of the former pier structures include excavation of over 40,000 cubic yards of waste from Parcel B between 1998 and 2002.

In 2011, wooden pier structures at designated piers and wharfs deemed potentially radiologically impacted were demolished and surveyed as documented in the Removal Action Completion Summary Report (ERS, 2012). The associated structures included pilings and pier structures at Submarine Piers B and C, the wooden portion of the submarine quay wall, the wooden remnants of Berths 61 and 64, and the wooden decking and pilings at Wharf No.2. The footprints of demolition activities are shown on Figure 2-1. The chemical portion of this investigation is focused on the seafloor sediment areas below the footprints of the previously removed structures.

## 2.5. Current Conceptual Site Model

A current CSM for Parcel F sediments is provided in detail in the FS (Barajas, 2008) and the FSDGs Investigation Technical Memorandum (Figures 2-2 and 2-3; Barajas and Associates, 2007). The flow charts provide a summary of the relationships between potential chemical sources, release mechanisms, potentially complete transport pathways, exposure media, potential current and future exposure pathways and receptors. A brief discussion of the CSM elements that relate to this investigation are provided in the following subsections.

### 2.5.1. Hydrodynamic Setting

Area III (Point Avisadero Area) is a 3.5-acre peninsula located in the northeastern portion of HPNS. It is bordered on the north and east by San Francisco Bay, on the south by Dry Dock 3, and on the west by the remainder of the HPNS property (Figure 1-2). Point Avisadero is flat with a steep armored riprap bank. The riprap banks extend well below low tide elevation. A high-resolution bathymetric survey conducted during the FSDGs investigation shows a shelf of sediment approximately 5 feet below mean lower low water (MLLW) located northwest of the drainage tunnel outfall. This shelf and the eastern bank of Point Avisadero both slope steeply to the northeast to a depth of about minus 35 feet MLLW, after which the bottom continues to deepen to minus 80 feet MLLW in the southeast direction. A sediment dynamics study conducted in Area III, indicated that surface sediment was re-suspended 16 percent of the time during the winter and 4 percent of the time during the summer due to strong tidal currents (Barajas, 2008). The distribution of COCs previously reported within Area III suggests that any sediments transported to depths greater than minus 65 feet MLLW were transported away from the Site. The shelf to the north and west of Point Avisadero is a net depositional environment. Evaluation of vertical COC profiles also suggest that deposition has reduced the concentration of surface sediments (Barajas and Associates, 2007).

Area IX/X is a shallow basin located south of HPNS (Figure 1-2). Circulation in South Basin is restricted and tidal currents are weak. The most significant sediment resuspension occurs as a result of storm waves that are generated from the southeast winds during the winter. Yosemite Creek enters the South Basin from the west and is characterized as a shallow, tidally influenced channel with no permanent flow. Sediment stability was evaluated in the South Basin to assess the likelihood that sediment would erode under typical and extreme hydrodynamic conditions and to predict the maximum depth of erosion. The analysis determined that up to 4.2 centimeters of erosion may be expected in a typical year during a winter storm event, whereas the maximum probable erosion during a 25-year event was estimated to be approximately 6 centimeters assuming a maximum event duration of 18.6 hours. Sediment deposition rates within the South Basin were evaluated using radioisotope data from sediment cores collected within the South Basin. The results of this evaluation determined that the net sediment accumulation rate is approximately 1 centimeter per year (Barajas and Associates, 2007).

In 2011, bathymetric surveys (single- and multi-beam) were conducted within the former Parcel B and C Piers investigation study area to verify that the pier structures, particularly pier pilings, were properly removed within the demolition footprints (Figure 2-1). The survey identified that all of the pilings had been removed with the exception of submerged steel pier pilings around Wharf No. 2. The steel pilings at Wharf No. 2 were subsequently removed (ERS, 2012).

### 2.5.2 Contaminant Extent and Distribution in Area III

The Final Validation Study Report (Battelle, BBL, and Neptune & Company, 2005) and the FSDG Technical Memorandum (Barajas and Associates, 2007) describe in detail the chemical distribution in sediments of Parcel F. Only Areas III, a portion of Area IX, and Area X require remedial action per the FS (Barajas, 2008). This investigation is focused on the areas beneath the former piers and designed to address the COCs, in support of the Proposed Plan, ROD, and Remedial Design for Parcel F. Hence, only the contaminant extent and distribution of Area III, in close proximity to the former piers removal area, is discussed.

The primary COCs for ecological receptors at Point Avisadero (Area III) are copper and mercury as documented the FS (Barajas, 2008). Target treatment zones for Area III were identified in the FS (Barajas, 2008) based on the "do not exceed" PRGs. Shoreline areas exceeding the PRGs are expected to be remediated in conjunction with activities in the adjacent upland Parcel B.

Copper was detected in Area III surface sediments during previous investigations above the San Francisco Bay ambient threshold and ER-M values in surface sediment samples in Area III. The highest concentration of copper (6,550 mg/kg) was reported in a sample collected immediately offshore of the northeast point of Point Avisadero. Concentrations of lead (1 sample location) and mercury were also reported in surface sediments above the San Francisco Bay ambient threshold and ER-M values (Barajas, 2008).

The highest concentrations of copper in subsurface sediments (above 500 mg/kg) were reported within approximately 200 feet from the shoreline and extended to depths of approximately 2.0 to 3.0 feet below mudline (bml). Reported concentrations of mercury in subsurface sediments were highest (above 2 mg/kg) within 200 feet of the shoreline from locations north and northeast of Point Avisadero. The highest concentration of mercury (252 mg/kg) was reported in a sample collected from 1.0 to 1.5 feet bml northeast of Point Avisadero (Barajas, 2008).

Concentrations of total PCBs exceeding the ER-M were reported during numerous investigation activities at Area III. The highest total PCB concentrations (between 2,000 and 6,000 µg/kg) were detected in subsurface sediment samples collected between 1.5 to 3.0 feet bml at two locations east to northeast of Point Avisadero (Barajas, 2008).

The horizontal and vertical distribution of COCs in Area III sediments are localized and discontinuous rather than exhibiting a gradient away from a well-defined source. COCs did not tend to co-occur, suggesting impacts are from episodic discharges (Barajas, 2008).

### 2.5.3. Data Gaps in Conceptual Site Model

In 2011, demolition and removal of several wooden pier structures within the boundary of Parcel F were conducted subsequent to approval of the FS Report. The associated structures were adjacent to Parcel B and included pilings and pier structures at Submarine Piers B and C, the wooden portion of the submarine quay wall, the wooden remnants of Berths 61 and 64, and the wooden decking and pilings at Wharf No.2 (ERS, 2012). Per BCT comments on the ERS (2012) report (Attachment 1), additional sampling is being proposed to assess if sediments in close proximity to and beneath the former piers were impacted by previously identified COCs at Parcel F as well as identify any change in erosional patterns as a result of removal of these piers.

In addition, bathymetric surveys are necessary to accurately estimate the volume of sediments proposed for removal and/or remediation, to identify the placement of fill and capping materials during implementation of the selected remedial alternative, and to estimate sediment erosion/deposition in Areas III and IX/X. The survey data will also be used to determine whether the installation of cofferdams is a viable option in Area IX/X.

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# 3.0 TECHNICAL APPROACH

The following sections describe the proposed data collection effort to address previously identified data gaps to refine the remedial alternatives for Parcel F in support of the Proposed Plan, ROD, and Remedial Design (Figures 3-1 and 3-2). Sediment sampling will be conducted at six locations for Parcel F COCs, where the Parcel B and C pier and wharf structures have been removed, as summarized in the Removal Action Completion Report (ERS, 2012; Figure 3-2). Surveying and sediment sampling activities will be conducted concurrently to streamline the field effort.

All field work will be conducted in accordance with the approved SAP, Environmental Protection Plan, and Waste Management Plan, Appendices A through C, respectively.

## 3.1. Bathymetric Surveys

Bathymetric surveys will be conducted within three areas: 1) area in footprint adjacent to sections of the former Parcel B and C shoreline piers and wharf structures removed in 2011 (ERS, 2012); 2) Area III; and 3) Area IX/X (Figures 2-1 and 3-1). Due to the differences in water depths between these areas, different survey technologies are proposed. For the deeper areas (former Parcel B and C piers, wharfs and Area III), a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder is proposed. For the shallow areas (Area IX/X), a survey skiff equipped with a single-beam echo sounder is proposed. Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet apart for the deeper areas and 50-feet apart in the shallower areas (Area IX/X). Since the depth to water in Area IX/X is less than 6 feet (Battelle, BBL, and Neptune & Company, 2005), the 50-foot spacing in this shallow area will provide sufficient resolution with the single beam survey. A narrow single-beam (3°) transducer will be utilized for data acquisition within Area IX/X. Due to the fan shape of the emitted sound pulses, the width of the transducer footprint is variable based on the depth to seafloor along each survey line. The width of coverage is equal to approximately 5% of the distance between the water surface and the seafloor (e.g., At 10’ above the seafloor the transducer will achieve approximately 0.5’-wide coverage along the survey track, while at 5’ above the seafloor it will achieve approximately 0.25’-wide coverage along the survey track). For the deeper waters beneath the former pier and wharf structures and Area III, the multi-beam survey provides greater resolution (see Section 3.1.1) that will identify change in bathymetry between the 100-foot lines. The resulting bathymetric survey data will be processed to provide 1-foot contours. Proposed survey areas and approximate line paths are provided on Figure 3-1.

The surveys will be conducted in accordance with the procedures defined in the United States Army Corps of Engineers (USACE) Design Hydrographic Survey Manual (USACE, 2013). Horizontal and vertical positions will be acquired with a Real Time Kinematic (RTK) Global Positioning System (GPS), or equivalent. A base station will be set up on the GPS survey control points used in previous bathymetric surveys of the areas (Figure 1-2). A Trimble dual frequency GPS receiver will be used in RTK mode on this control point. The unit transmits Radio Technical Commission for Maritime Services corrections to the system on board the survey vessel to improve

horizontal and vertical positioning to better than 0.5-meters (1.6 feet) and provide accurate inertial navigation through GPS outages for up to 30 seconds. Position data will be used in real-time to provide navigation information to the vessel operator. A preliminary coverage plot with survey information is generated in real-time to show survey coverage. Water surface measurements will be obtained by RTK GPS with on-the-fly ambiguity resolution. Differential corrections for the vessel position will be conducted against the GPS survey control points identified in Figure 1-2. A quality control point will be established using RTK techniques on a nearby dock, if possible, to confirm real time tide levels collected with the Hypack navigation software aboard the vessel. If a viable dock QC control point cannot be established, direct water line measurements will be made from the RTK Rover to confirm Hypack RTK tide readings as collected.

### 3.1.1. Multi-Beam Survey System

The bathymetric survey within the deeper area (former Parcel B and C piers, wharfs and Area III) will be conducted with a high resolution multi-beam system, such as a R2 Sonic 2024, or equivalent. The R2 Sonic 2024 works on user selectable frequency ranges so it is adaptable to a wide range of survey depths and conditions. Frequencies can be adjusted during the survey without having to shut down the sonar system, change hardware or halt recording data. This system produces a user selectable swath width of 10° to 160° using all 256 beams. The selected swath angle can also be rotated port or starboard, while recording, to direct the highly concentrated beams towards the desired target. The multi-beam swath width is approximately 4 times the water depth below the multi-beam sensor, providing detailed resolution in the deeper areas. The R2 Sonic 2024 will output digital depth data to the Hypack/Hysweep acquisition system where it will be stored with navigation data, motion sensor data, and heading data that will be applied in post processing.

### 3.1.2. Single-Beam Survey System

The bathymetric survey within the shallower Area IX/X, will be conducted with a survey grade digital single-beam echo sounder, such as Teledyne Odom Echotrac CV100, or equivalent. This system provides highly accurate single-beam profiling depth data below the vessel. The Odom Echotrac CV100 echo sounder collects digital profile records as well as digitized depth information for output to the navigational computer. Digital depth data is logged directly to the navigation computer along with date, time, and position for post processing and mapping. The Global Navigation Satellite System antenna is mounted directly above the single beam transducer resolving any offset issues.

### 3.1.3. Sound Velocimeter

A sound velocimeter, such as an AML Smart Probe®, will be used during all surveys to record high resolution sound velocity profiles of a water column to depths of 5,000 meters. Sound velocity will be measured directly using an acoustic time of flight sensor rather than calculated from conductivity, temperature and depth measured parameters, which is critical for obtaining accurate range corrections for bathymetric surveys.

### 3.1.4. Horizontal and Vertical Controls

The horizontal datum for this survey will be North American Datum 1983 State Plane Coordinates California Zone III, Epoch 2007.00. Both horizontal and vertical datum will be measured in U.S. survey feet.

A Meridian Attitude and Heading Reference System, or equivalent, will be used to provide vessel roll, pitch, heave, and heading. This data will be used by the data collection and processing software to compensate for the dynamic motion of the vessel when converting the sonar relative bearing and ranges of the soundings to XYZ positions on the earth, relative to the survey grid and vertical datum.

### 3.1.5. Survey Accuracies

The bathymetric survey will generally conform to the accuracy standards provided in the USACE Hydrographic Survey Manual (USACE, 2013). Conforming to the bottom coverage requirement will be contingent upon unlimited access to the survey area and survey system limitations.

### 3.1.6. Survey Data Processing and Reporting

Following the initial field data collection, data processing is required to obtain accurate position information from the GPS. Before exporting data to a computer-aided design (CAD) system, the data will be reviewed to remove any data anomalies and adjusted for GPS positional accuracy.

Bathymetric data will be collected in a straight path, perpendicular to flow. However; due to the often difficult navigational conditions caused by wind, barge traffic and floating obstructions the resulting cross section sometimes deviates from a straight line. CAD software will be used as needed to interpolate data to a line that approximates the straight-line path the boat was attempting to navigate.

Hypack/Hysweep software will be used to process and convert multi-beam sounding data into elevations. During post-processing, the multi-beam data will be corrected for tide, heave, pitch, roll, and speed of sound, and anomalous data will be removed. Multi-beam calibration offsets from patch test results will also be applied during the data editing process. During editing and processing, each survey line will be individually reviewed. This review consists of visual and automated inspection of speed of sound data, RTK tides, RTK GPS position data, motion sensor data, and sounding data. Anomalous data that are obvious system errors or “noise” within the water column, such as air bubbles, suspended particles and fish, and bottom multiples, will be filtered from the final data set. Manual editing will be based on a comparison of data outliers with surrounding data points, backscatter imagery, and file notes.

Resulting data sets from this bathymetric survey will be compared against historical bathymetric survey data and a comparison map will be provided to assist in showing areas of sediment deposition and erosion.

### 3.2. Sediment Sampling

Surface sediment sampling will be conducted for analysis of Parcel F COCs in the area beneath the six former Parcel B and C pier and wharf structures that have been removed (ERS, 2012; Figure 3-2). Sediment samples will be collected from each of the following six (6) former structures: Berth 64, Berth 61, Pier B location, Pier C location, wooden Quay Wall, and Wharf #2 area (Figure 3-2). The proposed sampling density and approach follows the sediment screening and testing guidelines documented in Table 7 of the Water Board (2000), 'Draft Staff Report, Beneficial Reuse of Dredged Materials Sediment Screening and Testing Guidelines'. Although the purpose of the sampling described in this work plan is not specifically for sediment beneficial reuse, the referenced guidance does provide a basis for the determination of total number of samples to be collected and use of composite sampling of sediment in San Francisco Bay. These procedures recommend the collection of one composite sediment sample, consisting of four individual samples, for sediment volumes ranging between 5,000 cubic yards (135,000 cubic feet) and 20,000 cubic yards (540,000 cubic feet) (Water Board, 2000). Because we are collecting surface sediment samples from the upper five centimeters of sediment surface, it is proposed that one composite sample be collected for Former Berth 64 and 61 and Quay Wall Areas, with a minimum of four individual sub-samples collected per composite sample. To ensure a denser coverage in the larger areas, 8 individual sub-samples will be composited into two composite samples (four sub-samples per composite sample) for Former Pier B, Pier C, and Wharf #2 Areas. Individual sub-samples will be collected in the field. The sub-samples will be composited for each former pier area by the laboratory. Proposed individual surface sediment sample point locations for each pier area are depicted on Figure 3-2. A total of 36 individual sub-samples will be collected in the field. The individual surface sediment sub-samples will be composited by the laboratory to create 9 surface composite sediment samples from each of the former pier areas (SAP Worksheet #14) as follows:

- **Former Berth 64 Area** (approximately 16,000 square feet [sf]): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Berth 61 Area** (approximately 6,000 sf): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Pier B Area** (approximately 40,000 sf): Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,

- **Former Pier C Area** (approximately 40,000 sf): Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Quay Wall Area** (approximately 15,000 sf): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, and
- **Former Wharf #2 Area** (approximately 24,000 sf): Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for copper, mercury, lead, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory.

Based on the COCs identified at Parcel F, the sediment samples will be composited by the laboratory and analyzed for the following:

- PCBs by EPA Method 8082,
- Copper by EPA Method 6020,
- Mercury by EPA Method 7471A, and
- Lead by EPA Method 6020.

Composite sampling is expected to provide the best overall analytical representation for each of the former six pier areas and in support of the Proposed Plan and ROD. Composite sediment sample results will be compared to the RAO 2 "Area Weighted Average" PRG for PCBs and the RAO 1 "Do-Not-Exceed" PRGs for metals, with the exception of lead. The NOAA ER-M value for the protection of the benthic community will be used as a numeric screening value for lead (218 mg/kg) (NOAA, 1999). If composite sediment sample COC concentrations from any former pier area exceed half (½) the Project Action Levels, then all individual sub-samples placed on hold in the laboratory for that area, will be analyzed for the Area III COCs (PCBs, copper, mercury, and lead) to further refine any area of contamination below the former piers per the screening criteria identified in Table 3-1. The decision logic for the analytical approach is summarized in SAP Worksheet #11, 5 – Develop the Analytical Approach (Appendix A). Any subsequent characterization will be conducted during the remedial design.

As concluded in the FS Addendum, based on the results of numerous investigation activities, no radioactivity in excess of naturally occurring background levels are present within sediment at Parcel F (KCH, 2017). In addition, during demolition of the pier structures conducted in 2011, all

of the removed pier materials were screened for elevated gamma radiation prior to disposal. No radiologically contaminated material was reported (ERS, 2012). However, per the Navy Accident Prevention Plan/Site Safety and Health Plan, radiological screening will be conducted on all sediment samples by the Navy's Basewide radiological safety contractor.

# 4.0 GENERAL SITE ACTIVITIES AND REQUIREMENTS

The following work tasks will be performed to complete this investigation.

## 4.1. Permits and Notifications

Prior to initiation of field activities, ECC-Insight will notify the HPNS Caretaker Site Officer, Resident Officer In Charge of Construction, and appropriate HPNS security personnel regarding the schedule and nature of the anticipated work. In addition, the United States Coast Guard will be notified prior to conducting any offshore activities.

ECC-Insight will attend contractor integration meetings at HPNS as needed to coordinate field activities with other Navy contractors.

## 4.2. Mobilization

Mobilization activities will include site preparation, movement of equipment and materials to the site, as well as training and site orientation of field personnel. Following proper notifications and receipt of authorization, the field personnel and survey vessels will be mobilized to the site. All field equipment and vehicles will be properly inspected and logged prior to being brought on site to insure they are in good working order.

## 4.3. Bathymetric Survey Data Collection

At Area III and the area in footprint and adjacent to former Parcel B and C piers and wharf structure areas, a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder will be utilized. At Area IX/X, a survey skiff equipped with a single-beam echo sounder will be utilized. Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet (multi-beam bathymetric survey) and 50 feet apart (Area IX/X). The resulting bathymetric survey data will be processed to provide 1-foot contours. Proposed survey areas and line paths are provided on Figure 3-1.

Horizontal and vertical positions data acquired with an RTK GPS, or equivalent, will be used in real-time to provide navigation information to the vessel operator. A preliminary coverage plot with survey information is generated in real-time to show survey coverage.

The surveys will be conducted in general accordance with the procedures defined in the USACE Design Hydrographic Survey Manual (USACE, 2013).

## 4.4. Sediment Sample Collection

Surface sediment samples will be collected from the survey vessel with a Van Veen grab sampler or equivalent. Surface sediment samples will be collected from approximately the upper five centimeters of the sediment surface. Depth (vertical elevation) of samples will be calculated from the survey data. If the results of the surface sediment sampling indicate that subsurface sediment samples are required, additional sampling will be conducted as part of the pre-design investigation

in support of the remedial design. If debris or rocks prevent sampling from the proposed sample location, then the vessel will be relocated as close to the original location as possible so that an acceptable grab sample can be collected. Upon retrieval of the sampler, the sediment sample will be inspected for disturbance (e.g., excessive washing or improper sampling device closure), and if deemed acceptable, the sample will be logged and placed in a clean sample container. If the sample retrieval is unacceptable, the sample will be properly discarded in a sealable designated onboard investigation-derived waste (IDW) container and a new sample will be collected. If needed, sediment samples will be removed from the grab sampler with a dedicated disposable sampling spoon, or equivalent. Samples will be properly labeled, documented on a chain-of-custody and immediately placed in a cooler. All sampling will be conducted in accordance with the SAP (Appendix A).

### 4.5. Equipment Decontamination

Decontamination of all non-disposable sampling equipment will be performed to prevent the introduction of extraneous materials into samples and to prevent potential cross-contamination between samples. Decontamination of non-disposable sampling equipment, including the Van Veen sampler, will be conducted as follows:

1) The equipment will be cleaned between each sample location with a brush, using laboratory grade detergent (e.g., Liquinox) and a potable water solution; rinsed with potable water and rinsed again with deionized water. The equipment will be scrubbed and rinsed in three separate five-gallon buckets,
2) The equipment will be reassembled and placed in a clean area on plastic, and
3) Equipment rinsates will be collected and analyzed for target compounds to provide a QC check on the decontamination procedure. The frequency of equipment rinsate sample collection is provided in the SAP (Appendix A).

### 4.6. Waste Management

Waste management activities during the proposed investigation will involve waste characterization and profiling, manifesting, handling, transportation and disposal. Anticipated waste streams include solid and liquid wastes. Solid wastes will consist of excess sediment from grab samples, disposable sampling equipment and used personal protection equipment. Liquid wastes will include water from decontamination of field equipment. IDW generated on the boat will be placed into a sealable waste container and later transferred to a 55-gallon drum stored on site. Efforts will be taken by the field team, whenever possible, to minimize the generation of wastes while maintaining full integrity of the project and Data Quality Objectives.

Samples will be collected from the solid and liquid waste material for non-hazardous or hazardous waste characterization in accordance with applicable State of California and federal regulations. Analytical parameters will depend on the waste hauler and waste handling facility.

Further details regarding waste management are provided in the Waste Management Plan (Appendix C).

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# 5.0 SCHEDULING AND DELIVERABLES

The proposed schedule for completing the planned deliverables and field activities is presented below and may change pending stakeholder review schedules:

**Project Schedule**

| Description | Date |
|---|---|
| Draft Sediment Investigation and Bathymetric Survey Work Plan | July 2017 |
| Approval of Final Sediment Investigation and Bathymetric Survey Work Plan | November 2017 |
| Conduct Sediment Investigation, Bathymetric Survey Field Work, and Laboratory Results:<br><br>• Mobilize field crew and equipment,<br>• Collect composite samples of former piers and wharf structure and conduct bathymetry (10 day turnaround time),<br>• Request additional sampling of individual sub samples pending results of composite sampling above (as needed),<br>• Complete Bathymetric Survey, and<br>• Laboratory Validation Complete. | January 15 – March 31, 2018<br><br>January 15, 2018<br>January 15- 31, 2018<br><br>January 25-31, 2018<br><br>January 31, 2018<br>March 31, 2018 |
| Draft Sediment Investigation and Bathymetric Survey Summary Report | July 2018 |
| Final Sediment Investigation and Bathymetric Survey Summary Report | November 2018 |

Note: Schedule and duration may change pending subcontractor availability, weather conditions, and field logistics as well as regulatory and Navy approvals.

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# 6.0 REFERENCES


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# ATTACHMENTS

Attachment 1 EPA Comments 8 and 9 on the Remedial Action Completion Summary Report (ERS, 2012) **(on Compact Disk)**

## Paulding, Reginald F CIV, BRAC

**From:** LEE, LILY <LEE.LILY@EPA.GOV>
**Sent:** Monday, August 01, 2016 15:57
**To:** Paulding, Reginald F CIV, BRAC
**Cc:** Tina Low (TLow@waterboards.ca.gov); juanita.bacey@dtsc.ca.gov; Janda, Danielle L CIV
**Subject:** [Non-DoD Source] FW: Parcel F - sampling under the removed piers off Parcel B-2 for non-rad contaminants

Dear Reggie,

Welcome again to Parcel F. I just remembered -- in case you hadn't already heard about this, here is another Parcel F action item that is pending from a while ago.

Lily Lee
Cleanup Project Manager
Superfund Division
U.S. Environmental Protection Agency, Region 9
75 Hawthorne St. (SFD-8-3)
San Francisco, CA 94105
Tel: 415-947-4187, Fax: 415-947-3518
www.epa.gov/region9/superfund

-----Original Message-----
From: Janda, Danielle L CIV NAVFAC SW [mailto:danielle.janda@navy.mil]
Sent: Wednesday, October 07, 2015 6:44 PM
To: LEE, LILY <LEE.LILY@EPA.GOV>
Cc: tlow@waterboards.ca.gov; Bacey, Juanita@DTSC <Juanita.Bacey@dtsc.ca.gov>; Robinson, Derek J CIV NAVFACHQ, BRAC PMO <derek.j.robinson1@navy.mil>; kbrasaemle@techlawinc.com
Subject: RE: Parcel F - sampling under the removed piers off Parcel B-2 for non-rad contaminants

Hi Lilly,

I had discussed this potential data gap at Parcel F with KCH, the contractor we will work with to help us do an extensive review of all relevant information to Parcel F in order to identify a remedy. Rather than doing ad hoc data collection efforts at Parcel F, we will incorporate potential data gaps into our review, including the potential need for further sampling and for additional erosional/depositional pattern studies. I anticipate that we will have several discussions about this topic, and other issues related to Parcel F, that will help us determine the best course of action for future field efforts.

V/r,
Danielle Janda
Environmental Engineer
NAVFAC Southwest
Navy BRAC PMO West
33000 Nixie Way
Bldg 50, 2nd Floor
San Diego, CA 92147

Phone: 619-524-6041

-----Original Message-----
From: LEE, LILY [mailto:LEE.LILY@EPA.GOV]
Sent: Wednesday, October 07, 2015 11:23 AM
To: Janda, Danielle L CIV NAVFAC SW
Cc: tlow@waterboards.ca.gov; Bacey, Juanita@DTSC; Robinson, Derek J CIV NAVFACHQ, BRAC PMO; kbrasaemle@techlawinc.com
Subject: Parcel F - sampling under the removed piers off Parcel B-2 for non-rad contaminants

Dear Danielle,

Thank you for your presentation about Parcel F plans at last week's BCT meeting. Below are excerpts of the RTC's for the 2012 RACR regarding sampling under the removed piers at Parcel F. I talked with you by phone about this data gap in January 2015. My recollection is that you said the sampling wouldn't be part of the scope of the initial field work for the activated carbon pilot, but future quarterly field work could be other opportunities for incorporating the sampling under the piers this year. Could you please give an update on Navy's plans for performing this sampling? The results will be important to inform plans for future remedial action. Thanks!

- Lily

FINAL

REMOVAL ACTION COMPLETION

SUMMARY REPORT

Pier Radiological Surveys and Removal

Hunters Point Naval Shipyard

San Francisco, California

August 2, 2012

Appendix K RTC's:

EPA Comment # 8. Removal of the piers may have resulted in changes in the

erosional and depositional patterns in the vicinity of the former piers, so the study to evaluate erosional/depositional patterns should be redone in these areas. It is understood that this was not included in the scope of this removal action, but updated information is needed for these areas in Parcel F. Please discuss when this study can be done.

Navy Response: The Navy is in the process of contracting for a data gap investigation (DGI) study in the South Basin at Parcel F and unfortunately investigations of erosional/depositional patterns are not part of the current scope of work. However, the Navy will endeavor to modify the current scope of work to include an erosional/depositional patterns study where the piers used to be located at as part of the upcoming Parcel F DGI.

EPA Comment # 9. Although not part of the removal action scope, samples should be collected to address the data gap concerning whether sediment in close proximity to and beneath the former piers was impacted by chemicals from the piers (e.g., wood preservatives) or by sediment that migrated beneath the piers. Sediment beneath piers has been found to be contaminated at Navy sites like Alameda Point and Long Beach Naval Shipyard and at other sites like United Heckathorn. These samples should be analyzed for metals, polynuclear aromatic hydrocarbons (PAHs) and constituents of creosote, pentachlorophenol, polychlorinated biphenyls (PCBs),

dioxins, and radionuclides. Ideally, these samples would be collected before the winter of 2012-2013, when storms could result in eroding and re-depositing these sediments. Please state when sampling to address potential contamination in close proximity to and beneath the piers can be conducted.

Navy response:

The analyses requested for specific analytes listed in the comment will be considered as part of the upcoming Parcel F DGI in the South Basin as the contracting efforts that are already underway permit.

Source: pp. 130-131 of Appendix J&K pdf at this link:

http://www.envirostor.dtsc.ca.gov/regulators/deliverable_documents/4973310708/Final%20RACSR%20HPS%20Pier%20Removal%20_APP%20JK-%20Copy.pdf

Lily Lee

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www.epa.gov/region9/superfund

# TABLES

**Table 2-1**
**Remedial Action Objectives and Preliminary Remediation Goals for Chemicals of Concern**

| RAO | COC | PRG/PAL (µg/kg) | Basis |
|---|---|---|---|
| **RAO 1** | Copper | 271,000 | Not to exceed threshold |
| **RAO 1** | Lead | NE; 218,000 [1] | |
| **RAO 1** | Mercury | 1,870 | |
| **RAO 1** | PCBs (total) | 1,240 | |
| **RAO 2** | PCBs (total) | 1,350 | Area-weighted average |
| **RAO 3** | PCBs (total) | 200[2] | |

[1]A PRG for lead was not established, due to uncertainty associated with bioavailability and toxicity of lead. Lead is collocated with PCBs in sediment, so achieving the cleanup goals for PCBs is expected to address any risks associated with lead. However, the National Oceanic and Atmospheric Administration Effects Range-Median (ER-M) value for the protection of the benthic community will be used as a numeric screening value for lead (218,000 µg/kg).

[2] 200 µg/kg total PCBs is based on background total PCB estimates for nearshore sediments in San Francisco Bay.

µg/kg – micrograms per kilogram
COC – chemical of concern
NE – not established
PAL – project action level
PCB – polychlorinated biphenyl
PRG – preliminary remediation goal
RAO – remedial action objective

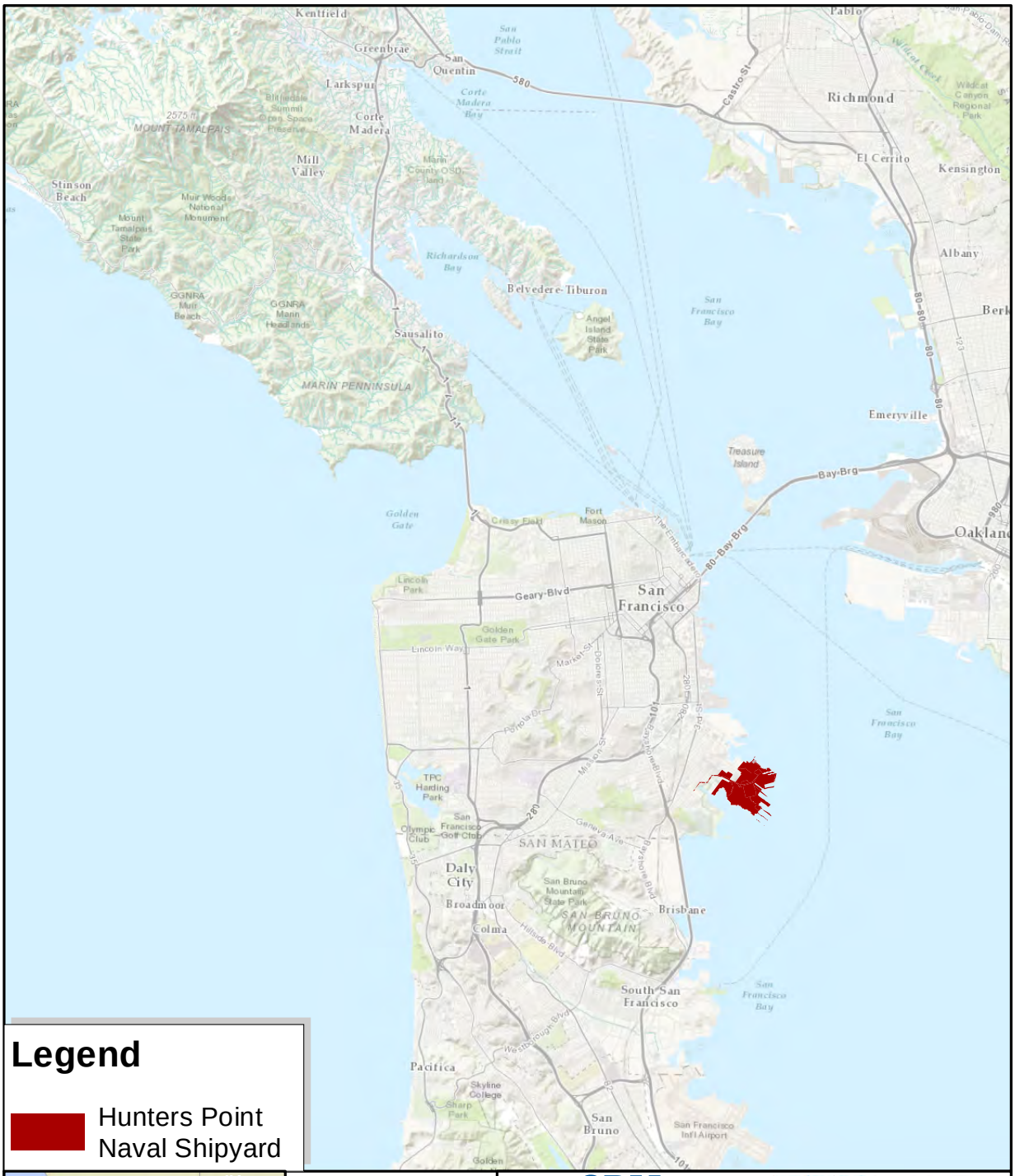
**Table 3-1**
**Sediment Sample Screening Levels**

| COC | PAL (µg/kg) | Composite Sample Screening Level (µg/kg) [5] | Individual Sub-Sample Screening Level (µg/kg) [6] |
|---|---|---|---|
| Copper (Cu) | 271,000[1] | 135,500 | 271,000[1] |
| Lead (Pb) | 218,000[4] | 109,000 | 218,000[4] |
| Mercury (Hg) | 1,870[1] | 935 | 1,870[1] |
| PCBs (total) | 1,240[1]/1,350[2]/200[3] | 675 | 1,240[1] |

[1] RAO 1 – Not to exceed threshold.
[2] RAO 2 – Area-weighted average.
[3] RAO 3 – long-term goal based on an area-weighted average background near-shore PCB concentration.
[4] No PRG has been established for lead. The PAL for lead is based on the NOAA ER-M value for the protection of the benthic community (NOAA, 1999).
[5] Composite sample screening levels are ½ of the PALs (RAO 1 – Not-To-Exceed PRGs for Cu and Hg, ER-M value for Pb, and RAO 2 – Area Weighted Average PRG for PCBs).
[6] Individual sub-sample screening levels are the RAO 1 – Not-To-Exceed PRGs for Cu, Hg, and PCBs, and the ER-M value for Pb.

µg/kg – micrograms per kilogram
COC – chemical of concern
ER-M – Effects Range-Median
NOAA – National Oceanic and Atmospheric Administration
PAL – project action level
PCB – polychlorinated biphenyls
PRG – preliminary remediation goal
RAO – remedial action objective

# FIGURES



1.5 0 1.5
Miles

*1 inch = 3 miles*

CDM Smith
ECC INSIGHT LLC

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 1-1**
**Site Location Map**

Sediment Investigation and Bathymetric Survey for Parcel F

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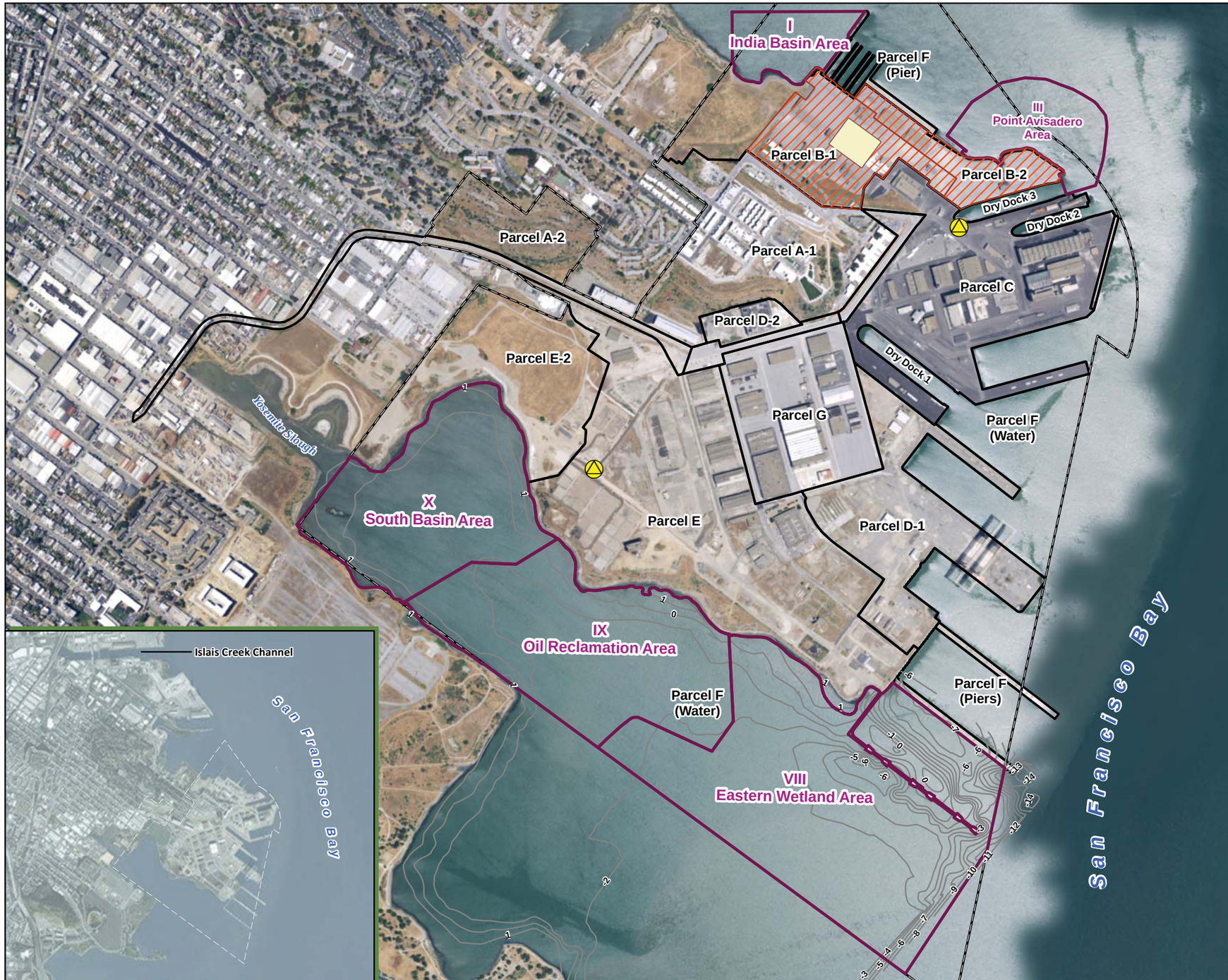


## Legend

- Navy Property Boundary
- Parcel F Subareas - Hunters Point Naval Shipyard
- Parcel Boundary
- IR-10 Carve Out
- Parcel B-1/B-2
- Bathymetry Contour (Depth in meters Referenced to MLLW)
- Survey Control Point

Notes:
MLLW - mean lower low water

1,000 0 1,000
Feet
*1 inch = 1,000 feet*

CDM Smith
ECC INSIGHT LLC

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 1-2**
**Parcel F Subareas**

Sediment Investigation and
Bathymetric Survey for Parcel F

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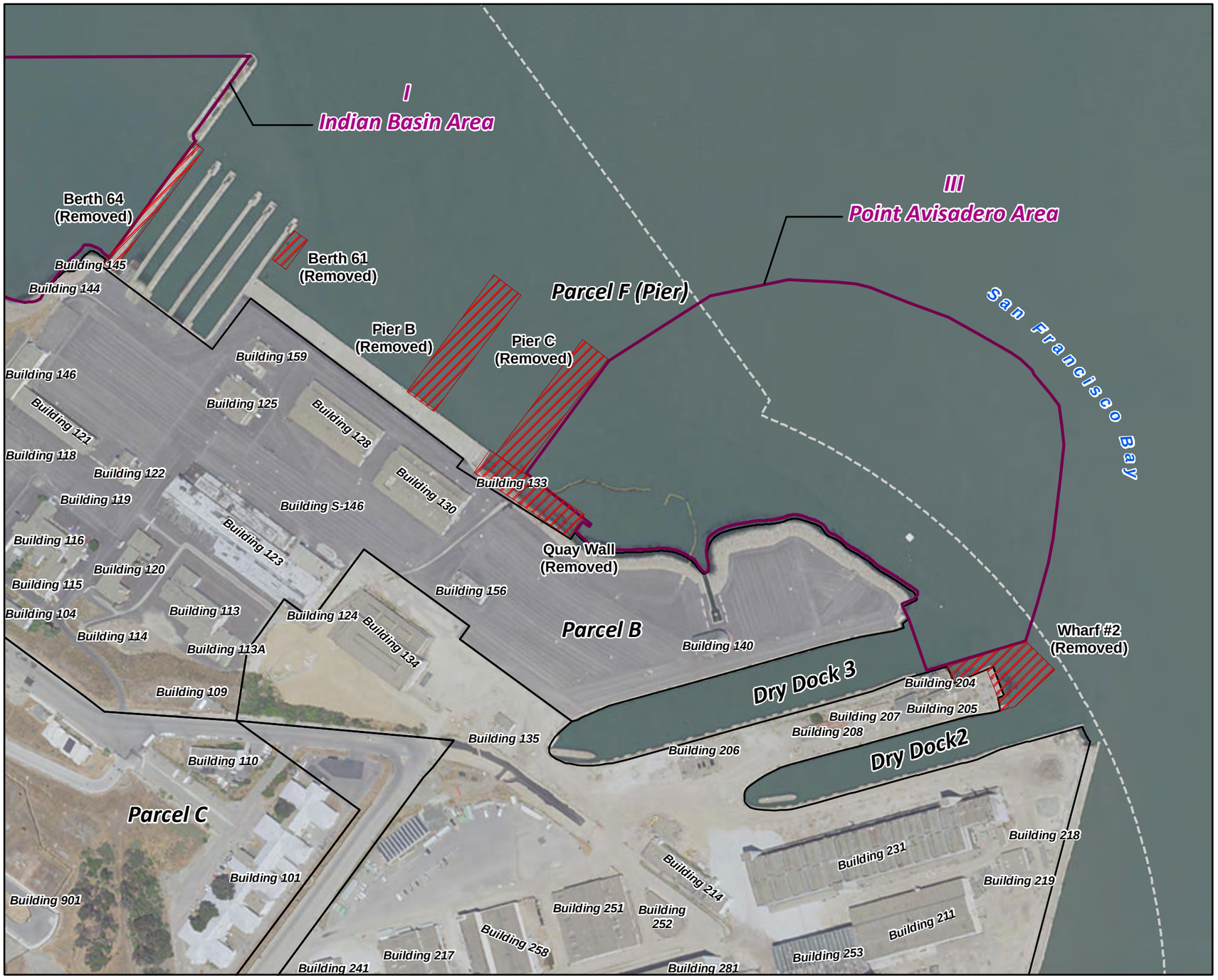


## Legend

- Navy Property Boundary
- Parcel Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Former Parcel B Pier Structure (Removed in 2011)

Note:
Utilities that existed on the piers were capped at the point of demolition on Berth 64 and Piers B & C at the points indicated by the hash marking (Demolition Area).

N

300 0 300
Feet
*1 inch = 300 feet*

CDM Smith

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 2-1**
**Former Parcel B Pier Structure Locations**

Sediment Investigation and
Bathymetric Survey for Parcel F

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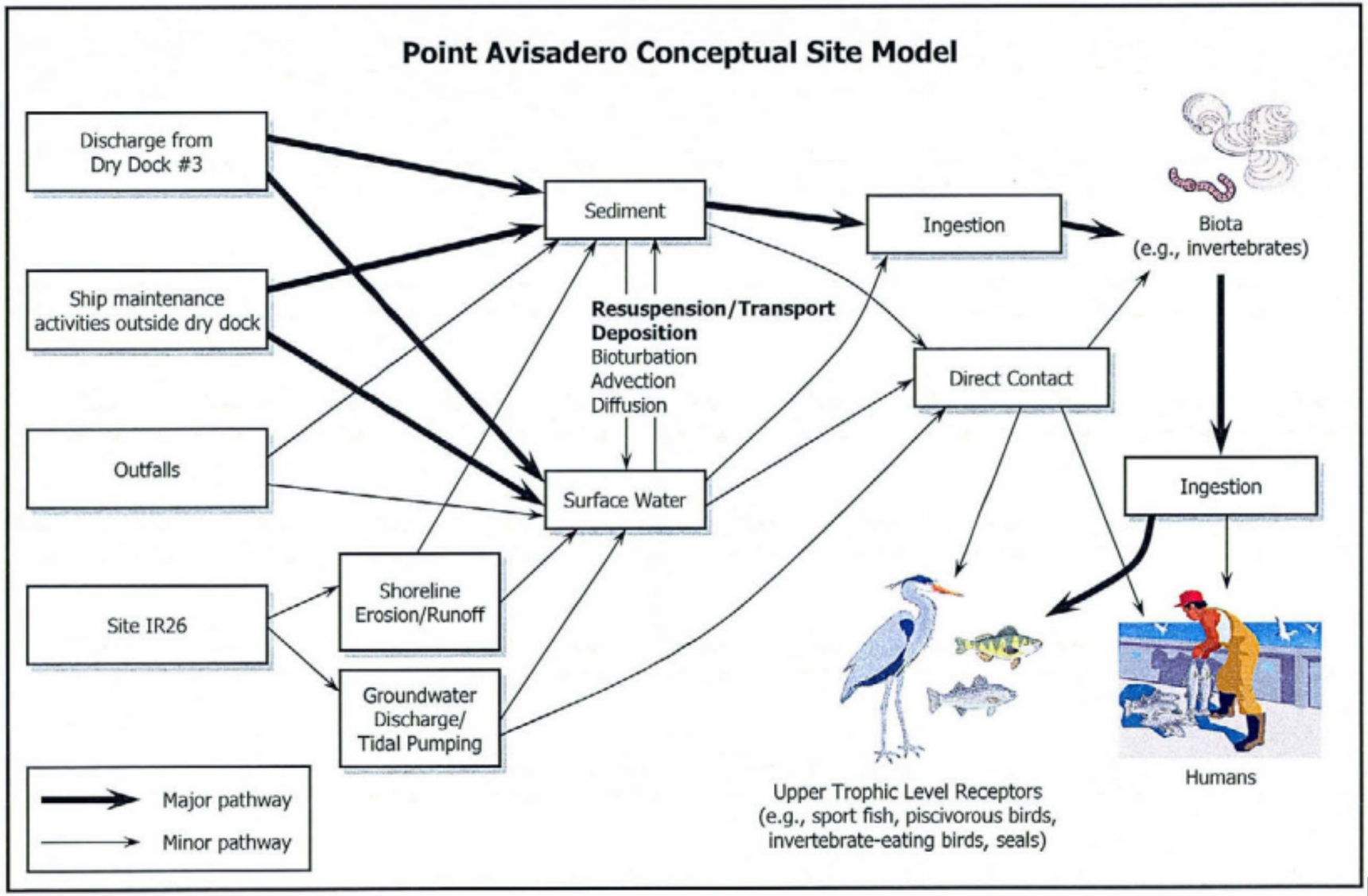


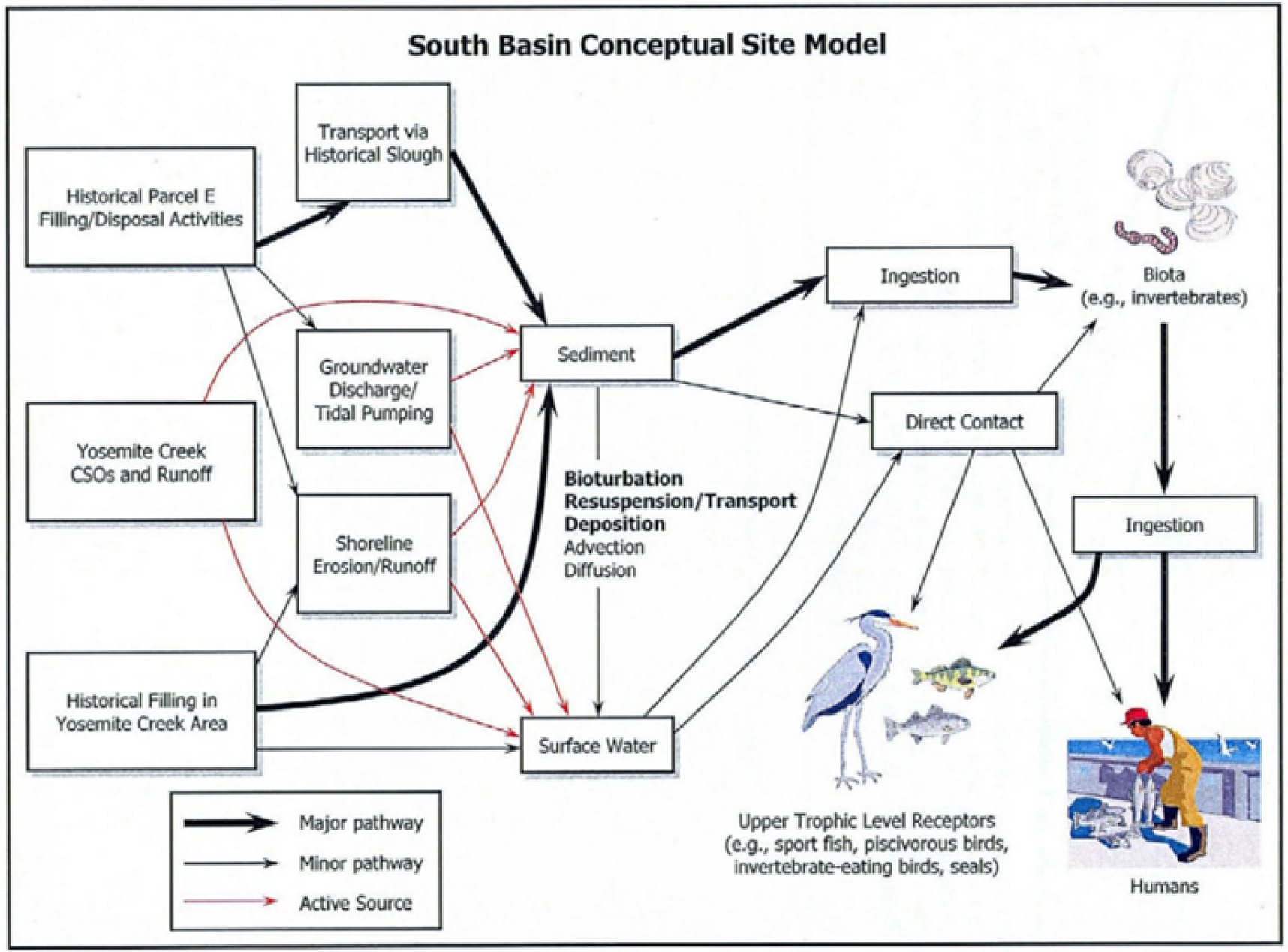

Source: Figure 3-3, Hunters Point Shipyard Parcel F Data Gaps Investigation (Barajas and Associates, 2007)

CDM Smith ECC INSIGHT LLC

Hunters Point Naval Shipyard, San Francisco, California
U.S. Department of the Navy, BRAC PMO West

**Figure 2-2**
**Point Avisadero**
**Conceptual Site Model**
**Sediment Investigation and Bathymetric Survey for Parcel F**

schellerka G:\GIS\212737_HP_ParcelF\RAA\Figure2-3_SouthBasinConceptualSiteModel 6/7/2017



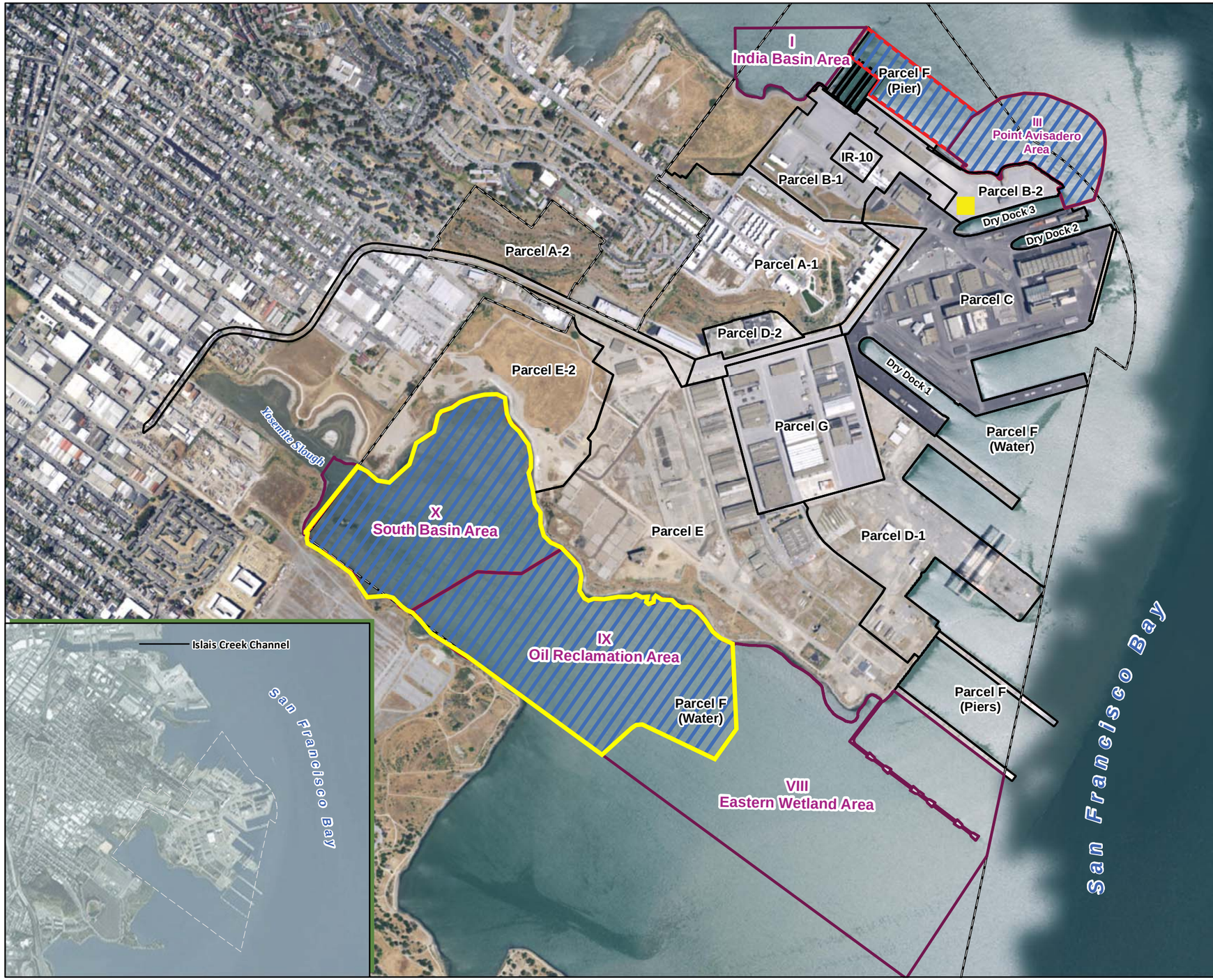

Source: Figure 3-3, Hunters Point Shipyard Parcel F Data Gaps Investigation (Barajas and Associates, 2007)

CDM Smith ECC INSIGHT LLC

Hunters Point Naval Shipyard, San Francisco, California
U.S. Department of the Navy, BRAC PMO West

**Figure 2-3**
**South Basin**
**Conceptual Site Model**
**Sediment Investigation and Bathymetric Survey for Parcel F**

schellerka G:\GIS\212737_HP_ParcelF\RAA\Figure2-3_SouthBasinConceptualSiteModel 6/7/2017




## Legend

- Navy Property Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Parcel Boundary
- Approximate Bathymetric Survey Lines (100-foot spacing) and Areas
- Approximate Bathymetric Survey Lines (50-foot spacing) and Areas
- Staging Area

N

1,000 0 1,000
Feet

*1 inch = 1,000 feet*

ECC INSIGHT LLC

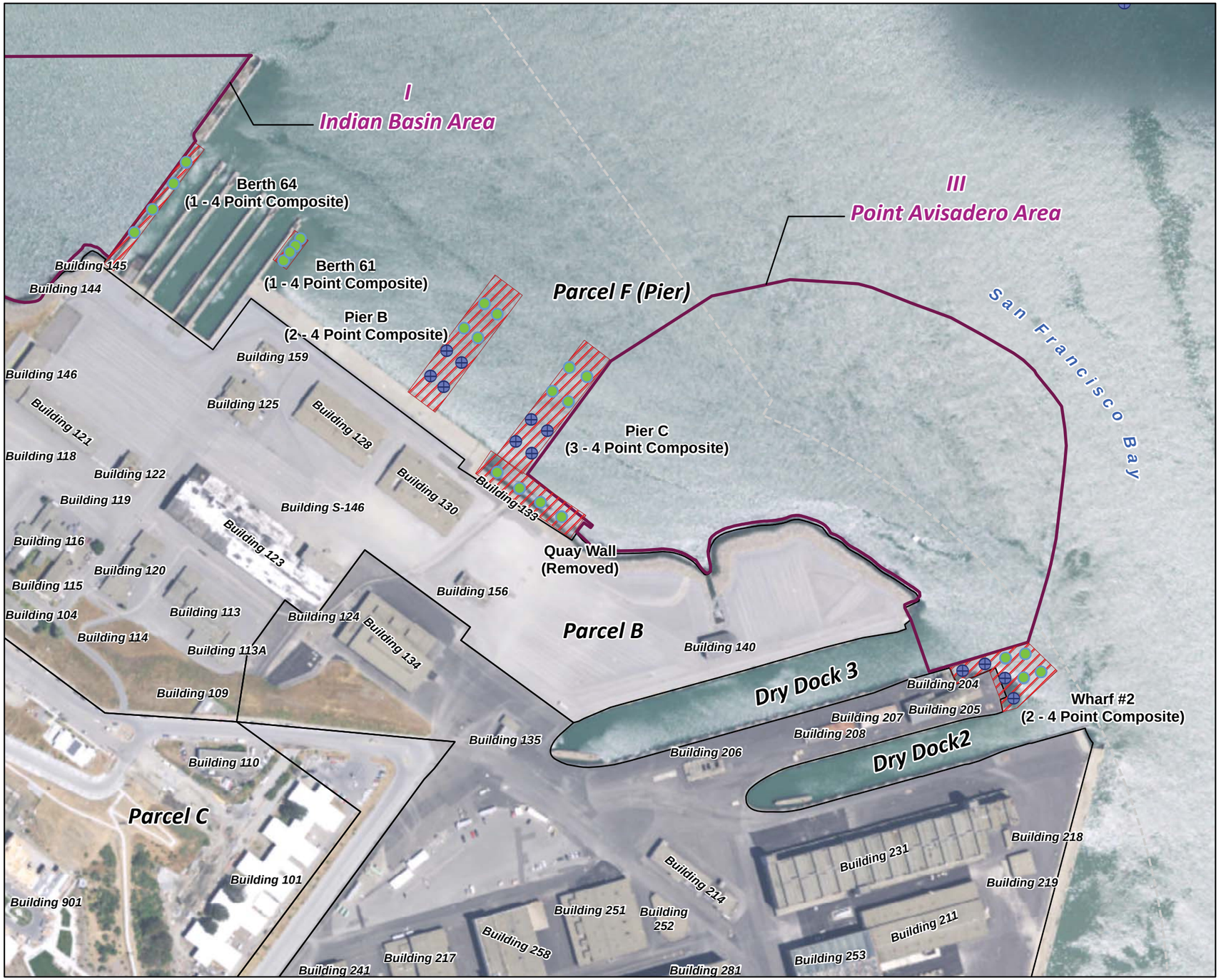
CDM Smith

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 3-1**
**Proposed Bathymetric Survey Areas**

Sediment Investigation and
Bathymetric Survey for Parcel F

schellerka G:\GIS\212737_HP_ParcelF\Task8_SAP\DraftFinal\Figure3-1_ProposedBathymetricSurveyAreas.mxd 9/22/2017




## Legend

- Navy Property Boundary
- Parcel Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Former Parcel B Pier Structure (Removed in 2011)
- Proposed Surface Sediment Sample Location (for Composite)
- Second Set of Four Sub-samples for Second Composite Sample for Pier B, Pier C, and Wharf #2

N

300 0 300
Feet
*1 inch = 300 feet*

CDM Smith
ECC INSIGHT LLC

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 3-2**
**Proposed Sediment Sampling Locations**

Sediment Investigation and
Bathymetric Survey for Parcel F

Schellerka G:\GIS\212737_HP_ParcelF\WorkPlan\Figure3-2_ProposedSedimentSamplingLocations.mxd 9/12/2017

# FINAL
# Sampling and Analysis Plan

# Sediment Investigation and Bathymetric Survey for Parcel F Hunters Point Naval Shipyard, San Francisco, California

**November 2017**

Prepared for:
**DEPARTMENT OF THE NAVY**
**Naval Facilities Engineering Command Southwest**
33000 Nixie Way, Building 50
San Diego, California 92147-5101

Prepared by:
ECC-Insight LLC
2749 Saturn Street
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In Association with:
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100 Pringle Avenue, Suite 300
Walnut Creek, California 94596

Prepared under:
Naval Facilities Engineering Command Southwest
Contract Number: N62473-12-D-2004
Delivery Order: 0014
DCN: INEC-2004-0014-0003

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## SAP Worksheet #1: Title and Approval Page

**FINAL**

**SAMPLING AND ANALYSIS PLAN**

**November 2017**

# Sediment Investigation and Bathymetric Survey for Parcel F
# Hunters Point Naval Shipyard
# San Francisco, California

**Prepared for:**
**Department of the Navy**
**Naval Facilities and Engineering Command Southwest Division**
**33000 Nixie Way, Building 50**
**San Diego, California 92147-5101**

**Prepared by:**
**ECC-Insight, LLC**
**2749 Saturn Street**
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**In Association with:**
**CDM Smith**
**100 Pringle Avenue, Suite 300**
**Walnut Creek, California 94596**

**Prepared under:**
**N62473-12-D-2004**
**Task Order #0014**
**DCN#: INEC-2004-0014-0003**

Review Signature: 11/16/2017
Nick Weinberger/QAM Date

Approval Signature: ARLAUSKAS.JOSEPH.1229257192 Digitally signed by ARLAUSKAS.JOSEPH.1229257192 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=ARLAUSKAS.JOSEPH.1229257192 Date: 2017.11.16 10:36:54 -08'00'
Joe Arlauskas NAVFAC SW QAO Date

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## ATTACHMENTS (ON COMPACT DISK)

**ATTACHMENT A-1 FIELD FORMS**

**ATTACHMENT A-2 LABORATORY SOPS**

# ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| °C | degrees Celsius |
| µg/kg | micrograms per kilogram |
| APP | Accident Prevention Plan |
| BCT | BRAC Cleanup Team |
| bml | below mudline |
| BRAC | Base Realignment and Closure |
| C&T | Curtis and Tompkins |
| CAD | computer aided design |
| CAS | Chemical Abstract Service |
| CCV | continuing calibration verification |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| cm | centimeter |
| Cu | copper |
| COC | constituent of concern |
| CVAA | cold-vapor atomic absorption |
| CY | cubic yard |
| DL | detection limit |
| DoD | Department of Defense |
| DQO | Data Quality Objective |
| DTSC | Department of Toxic Substances Control |
| ECC-Insight | ECC-Insight LLC |
| ECD | electron capture detector |
| EDD | electronic data deliverable |
| ELAP | Environmental Laboratory Accreditation Program |
| EPA | Environmental Protection Agency |
| EWI | Environmental Work Instruction |
| FS | Feasibility Study |
| FTL | Field Team Leader |
| GC | gas chromatograph |
| GPS | Global Positioning System |
| Hg | mercury |
| HNO3 | nitric acid |
| HPNS | Hunters Point Naval Shipyard |
| ICAL | initial calibration |
| ICP-MS | Inductively coupled plasma mass spectrometry |
| ICS | interference check standard |
| ICV | initial calibration verification |
| IDW | investigation-derived waste |
| LCS/LCSD | laboratory control sample/laboratory control sample duplicate |
| LDC | Laboratory Data Consultants |
| LOD | limit of detection |
| LOQ | limit of quantitation |
| m | meter |
| mg/kilogram | milligrams per kilogram |
| MS/MSD | matrix spike/matrix spike duplicate |
| NA | not applicable |

| | |
|---|---|
| NAD | North American Datum |
| NAVFAC SW | Naval Facilities Engineering Command Southwest |
| NEDD | Naval electronic data deliverable |
| NIRIS | Naval Installation Restoration Information System |
| NOAA | National Oceanic and Atmospheric Administration |
| PARCC | precision, accuracy, representativeness, completeness, and comparability |
| PAL | project action level |
| PCB | polychlorinated biphenyl |
| PEM | performance evaluation material |
| PM | Project Manager |
| PMO | Program Management Office |
| PRG | preliminary remediation goal |
| PQCM | Project Quality Control Manager |
| PQL | practical quantitation limit |
| PQO | project quality objective |
| QA | quality assurance |
| QAM | Quality Assurance Manager |
| QAO | Quality Assurance Officer |
| QC | quality control |
| QL | quantitation limit |
| QSM | Quality Systems Manual |
| RG | Remediation goal |
| ROICC | Resident Officer-in-Charge of Construction |
| RPD | relative percent difference |
| RPM | Remedial Project Manager |
| RTCM | Radio Technical Commission for Maritime |
| RTK | Real Time Kinematic |
| RWQCB | Regional Water Quality Control Board |
| SAP | Sampling and Analysis Plan |
| SOP | standard operating procedure |
| SSHO | Site Safety and Health Officer |
| SVOC | semi-volatile organic compound |
| TBD | to be determined |
| USACE | United States Army Corp of Engineers |
| US EPA | United States Environmental Protection Agency |

## SAP Worksheet #2: Sampling and Analysis Plan Identifying Information

**Site Name/Number: Hunters Point Naval Shipyard (HPNS) Parcel F**

**Contractor Name:** ECC-Insight LLC (ECC-Insight)

**Contract Number:** N62473-12-D-2004

**Contract Title:** Unrestricted Environmental Multiple Award Contracts

**Work Assignment Number (optional):** Task Order 0014

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plan* (EPA, 2005) and United States Environmental Protection Agency (US EPA) *Guidance for Quality Assurance (QA) Project Plans, EPA QA/G-5* (EPA 2002),

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA),

3. This SAP is a project-specific SAP,

4. List organizational partners (stakeholders) and identify the connection with lead organization:

| Organization Partners/Stakeholders | Connection |
|---|---|
| San Francisco Regional Water Quality Control Board (RWQCB) | Base Realignment and Closure (BRAC) Cleanup Team (BCT) |
| Department of Toxic Substances Control (DTSC) | BCT |
| US EPA | BCT |
| San Francisco Department of Public Health | BCT |

5. Lead organization: BRAC Program Management Office (PMO), and
6. If any required SAP elements and required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   None.

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## SAP Worksheet #3: Distribution List

| Name of SAP Recipients | Title/Role | Organization | Telephone Number | E-mail Address or Mailing Address |
|---|---|---|---|---|
| Danielle Janda | Lead Remedial Project Manager (RPM) | BRAC PMO | (619) 524-6041 | danielle.janda@navy.mil |
| Sharon Ohannessian | RPM | BRAC PMO | (202) 685-1627 | sharon.ohannessian.ctr@navy.mil |
| Joe Arlauskas | Quality Assurance Officer (QAO) | Naval Facilities Engineering Command Southwest (NAVFAC SW) | (619) 532-4125 | joseph.arlauskas@navy.mil |
| Diane Silva | Administrative Record | NAVFAC SW | (619) 556-1280 | diane.silva@navy.mil |
| Shirley Ng | Resident Officer-in-Charge of Construction (ROICC) | NAVFAC SW | (510) 521-8713 (Office)<br>(510) 502-5051 (Cell) | shirley.ng@navy.mil |
| Derek Robinson | Base Environmental Coordinator | BRAC PMO | (619) 524-6026 | derek.robinson1@navy.mil |
| Doug DeLong | BRAC Compliance, Safety & Security Manager | BRAC PMO | (415) 743-4713 (office)<br>(510) 220-1894 (cell) | douglas.delong.ctr@navy.mil |
| Patricia McFadden | BRAC Caretaker Site Office | BRAC PMO | (415) 743-4720 | patricia.a.macfadden@navy.mil |
| Mitra Fattahipour, PG | Project Manager (PM) | ECC-Insight | (858) 342-5585 (Office/Cell) | mfattahipour@ieeci.com |
| Nick Weinberger | QA Manager | ECC-Insight | (714) 678-6700 | nweinberger@ieeci.com |
| Maureen Sassoon, CIH | Health and Safety Manager | ECC-Insight | [redacted] | [redacted] |
| Dave Marks, PG | Field Operations Manager/Senior Geologist | ECC-Insight | (916) 201-7187 | dmarks@ieeci.com |
| Mehdi Siavoshani | Field Team Lead/Site Safety and Health Officer (SSHO) | ECC-Insight | (619) 888-6977 | msiavoshani@ieeci.com |
| Aaron Heidt | Project Quality Control Manager (PQCM) | ECC-Insight | (208) 598-2998 | aheidt@ieeci.com |

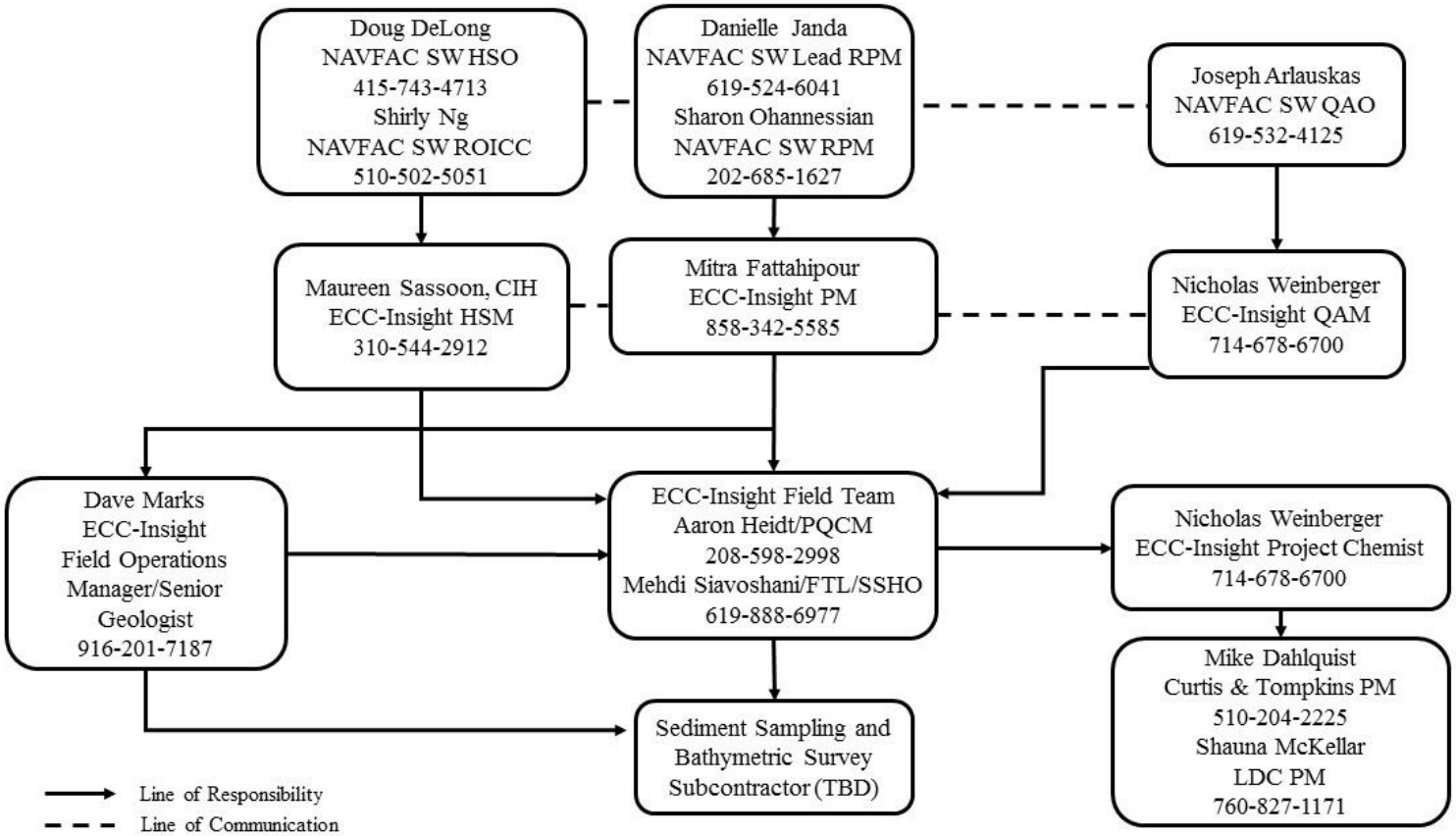
| Name of SAP Recipients | Title/Role | Organization | Telephone Number | E-mail Address or Mailing Address |
|---|---|---|---|---|
| Marco Mendoza, PG | Alternate PQCM/Alternate Field SSHO | ECC-Insight | (619) 843-9968 | mmendoza@ieeci.com |
| Eddie Stutts | Bathymetric Survey and Sediment Sampling Subcontractor | Fugro Pelagos, Inc. (Fugro) | (805) 289-3891 | estutts@fugro.com |
| Eric Blischke | Remediation Technical Lead | CDM Smith | (208) 904-0238 | blischkee@cdmsmith.com |
| Tina Ures | PM | RWQCB, San Francisco Bay Region | (510) 622-2064 | tina.ures@waterboards.ca.gov |
| Nina Bacey | PM | DTSC | (510) 540-2480 | Juanita.Bacey@dtsc.ca.gov |
| Judy Huang | PM | US EPA | (415) 972-3681 | huang.judy@epa.gov |
| Charlie Huang | PM | US Department of Fish and Wildlife | (916) 324-9805 | chuang@ospr.dfg.cca.gov |
| Amy Brownell | PM | San Francisco Department of Public Health | (415) 252-3967 | amy.brownell@sfdph.org |
| Mike Dahlquist | PM | Curtis and Tompkins (C&T) | (510) 486-0900 | mike.dahlquist@ctberk.com |
| Shauna McKellar | PM | Laboratory Data Consultants (LDC) | (760) 827-1171 | smckellar@lab-data.com |

## SAP Worksheet #4: Project Personnel Sign-Off Sheet

| Name | Organization/Role | Signature/E-mail Receipt | SAP Section Reviewed | Date SAP Reviewed |
|---|---|---|---|---|
| Sharon Ohannessian | BRAC PM | | Entire SAP | |
| Mitra Fattahipour, PG | ECC-Insight PM | | Entire SAP | |
| Eric Blischke | Principal Technical Lead | | Entire SAP | |
| Maureen Sassoon | Health and Safety Manager | | Entire SAP | |
| Mike Dahlquist | C&T PM | | Entire SAP | |
| Shauna McKellar | LDC PM | | Entire SAP | |
| Dave Marks, PG | Field Operations Manager | | Entire SAP | |
| to be determined (TBD) | Fugro/Field Team | | Entire SAP | |

[1] Final selection of field personnel to be made at least two weeks prior to field activities. Blank spots on Worksheet 4 are for field staff names and signature
Final sign off sheet will be kept in the project folder.

## SAP Worksheet #5: Project Organizational Chart



## SAP Worksheet #6: Communication Pathways

The communication pathways for the SAP are shown below:

| Communication Drivers | Responsible Entity | Name | Phone Number | Procedure (Timing, Pathway To/From, etc.) |
|---|---|---|---|---|
| Regulatory Agency Interface | BRAC RPM | Sharon Ohannessian | (202)685-1627 | RPM will inform Regulatory Agencies during monthly meetings. |
| Field Progress Reports | Site Superintendent/Field Operations Manager | Dave Marks, PG | (916)201-7187 | Superintendent will upload daily reports on a daily basis. |
| Stop Work due to Safety Issues | (SSHO)/Field Team Leader (FTL)/PQCM/Field Staff | Aaron Heidt/Mehdi Siavoshani / Dave Marks/Maureen Sassoon | See Worksheet #3 | Immediately stop work of all persons onsite if potential safety problem is observed. |
| SAP/Work Plan Changes prior to Field/ Laboratory work | ECC-Insight PM/ECC-Insight Chemist/FTL | Mitra Fattahipour / Nick Weinberger | See Worksheet #3 | The ECC-Insight PM will notify NAVFAC SW RPM as soon as possible by phone or email. Also, complete and submit field change requests to NAVFAC SW RPM. Regulators will be notified of any required changes within 24 hours. |
| SAP/Work Plan Changes in the Field | ECC-Insight PM/ECC-Insight Chemist/FTL | Mitra Fattahipour / Nick Weinberger | See Worksheet #3 | The ECC-Insight PM will notify NAVFAC SW RPM as soon as possible by phone or email. Also, complete and submit field change requests to NAVFAC SW RPM. Regulators will be notified of any required changes within 24 hours. |
| Field Corrective Actions | ECC-Insight PM/ECC-Insight Chemist/FTL | Mitra Fattahipour / Nick Weinberger | See Worksheet #3 | The ECC-Insight PM will notify NAVFAC SW RPM by email. |
| Sample Receipt Variances | Laboratory PMs | Mike Dahlquist | (510)486-0900 | Laboratory PM will call or email the FTL and project chemist after sample login. |
| Reporting Lab Quality Variances | Laboratory PMs | Mike Dahlquist | (510)486-0900 | Laboratory PM will call or email the ECC-Insight PM and Project Chemist. |
| Analytical Corrective Actions | Laboratory PMs and ECC-Insight QA Manager | Mike Dahlquist / Nick Weinberger | See Worksheet #3 | The laboratory PM or ECC-Insight QA Manager will inform the ECC-Insight PM. |
| Reporting Data Validation Issues | LDC PM | Shauna McKellar | (760)827-1171 | Validator PM will contact the ECC-Insight Chemist. |
| Severe Issues with Sample Analyses or Data Quality | ECC-Insight Chemist | Nick Weinberger | (714)678-6700 | ECC-Insight Chemist will inform the ECC-Insight PM who will inform the Navy RPM of severe issues with sample analyses or data quality. The Navy RPM will contact the Navy Chemist at their discretion. |

## SAP Worksheet #7: Personnel Responsibilities Table

| Title/Role | Organizational Affiliation | Responsibilities |
|---|---|---|
| Sharon Ohannessian/ RPM | BRAC | • Verify that work is accomplished as required by the project scope of work,<br>• Oversees project cost and schedule, and<br>• Serves as the lead interface between agencies. |
| Joe Arlauskas / QAO | NAVFAC SW | • Reviews and approves of the SAP, and<br>• Has authority to suspend the project if the QA requirements are not met. |
| Mitra Fattahipour / PM | ECC-Insight | • Management of the overall project,<br>• Coordinates team members and subcontractors including ensuring all personnel adhere to the administrative and technical requirements of the project scope of work,<br>• Monitors and reports the progress of work and ensures that the project deliverables are completed on time and within project budget,<br>• Monitors the budget and schedule, and notifies the RPM of any changes that may require administration actions,<br>• Ensures all work meets the requirements of the technical specifications and complies with applicable codes and regulations,<br>• Ensures Accident Prevention Plan (APP) is implemented,<br>• Primary point of contact for NAVFAC SW and the Contractor team members,<br>• Coordinates satisfactory resolution and completion of evaluation and acceptance reporting for corrective action reports,<br>• Ensures all technical work meets the requirements of the technical specifications and complies with applicable codes and regulations, and<br>• Ensures all work is conducted in accordance with Work Plan(s). |
| Dave Marks / Field Operations Manager/Project Geologist | ECC-Insight | • Coordinates on site team members and subcontractors including ensuring all personnel adhere to the administrative and technical requirements of the project scope of work,<br>• Supervises and reports the progress of onsite work and ensuring that the project deliverables are completed on time and within project budget,<br>• Ensures that all work meets the requirements of the technical specifications and complies with applicable codes and regulations,<br>• Ensure APP is implemented on site, and<br>• Address the need to modify field activities in order to meet the goals of the NTPS. Ensures that all work is conducted in accordance with Work Plan(s). |

| | | |
|---|---|---|
| Nick Weinberger / Quality Assurance Manager (QAM) | ECC-Insight | • Reviews the SAP and SAP Amendments prior to submittal to the NAVFAC SW,<br>• Oversees compliance with QA requirements, and<br>• Monitors corrective actions. |
| Mehdi Siavoshani / PQCM | ECC-Insight | • Perform onsite quality control (QC) inspection of field activities and materials,<br>• Stops work if QC criteria are not being met during field activities,<br>• Prepares daily construction quality control reports and submits them to the ROICC, and<br>• Ensures all daily forms, logs, etc. are completed and filed. |
| TBD / Field Staff | ECC-Insight | • Performs oversight to ensure samples are collected in accordance with the SAP,<br>• Data coordinator for incoming field and laboratory data, and<br>• Performs oversight of bathymetric survey activities. |
| Mehdi Siavoshani / Field Team Lead/SSHO | ECC-Insight | • Oversees health and safety of onsite activities,<br>• Leads sediment sampling and bathymetric survey activities, and<br>• Ensure APP is implemented on site. |
| Nick Weinberger / Database Manager | ECC-Insight | • Incorporates analytical data into the project database, and<br>• Uploads Naval electronic data deliverables (NEDD) to Naval Installation Restoration Information System (NIRIS). |
| Mike Dahlquist / PM | C & T | • Performs laboratory analysis of site and QC samples by the methods listed in Worksheet #15 within holding times,<br>• Coordinates field supplies necessary for sample collection and shipment, and<br>• Reviews laboratory reports for accuracy and grammar. |
| Shauna McKellar / PM | LDC | • Performs data validation according to National Function Guidelines and Environmental Work Instruction (EWI) #1. |

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## SAP Worksheet #8: Special Personnel Training Requirements Table

All personnel who will be working on a boat will have or undergo boat safety training in accordance with the APP.

Health and safety training is not considered specialized training and training requirements are covered in the APP. Experienced field personnel who have been trained during previous onsite training will be used to conduct field activities. Experienced field personnel will perform onsite training for junior field team members. Field personnel will have been or will be trained in the following:

- Sediment sampling,
- Boating safety,
- Decontamination procedures,
- Investigation-derived waste (IDW) management,
- Sample custody,
- Field screening equipment, and
- Sample packaging and shipment.

Sampling personnel will be required to read and understand the SAP prior to any sample collection activities. The Project Personnel Sign-off Sheet (Worksheet #4) will be signed by any on-site personnel conducting sampling to indicate that they have read the SAP and will perform the task as described. The sign-off sheet will be maintained in the project file.

At least one field person onsite will have completed the United States Army Corps of Engineers Construction Quality Management for Contractors training, while construction activities are occurring. This field person will be responsible for ensuring construction activities and materials meet the QC criteria.

## SAP Worksheet #9: Project Scoping Session Participants Sheet

A project kick off meeting was conducted on September 15, 2016 to discuss the Parcel F scope of work.

**Objective:** Kick Off Meeting, Parcel F, Hunters Point Naval Shipyard, San Francisco, CA

**Meeting Date:** Thursday, September 15, 2016

**Meeting Location:** BRAC PMO office and via teleconference

**Participants:**

| Name | E-mail Address or Mailing Address |
|---|---|
| Derek Robinson | derek.robinson1@navy.mil |
| Danielle Janda | danielle.janda@navy.mil |
| Reginald Paulding | reginald.paulding@navy.mil |
| Mitra Fattahipour, PG | mfattahipour@ieeci.com |
| Ryan Wymore, P.E.* | wymorera@cdmsmith.com |
| Tamzen Macbeth, Ph.D., PE* | macbethtw@cdmsmith.com |
| Eric Blischke* | blischkee@cdmsmith.com |
| John Nyznyk, P.E.* | nyznykjp@cdmsmith.com |
| Nick Weinberger, CQA* | nweinberger@ieeci.com |
| Dave Marks, PG* | dmarks@ieeci.com |
| Mehdi Siavoshani* | msiavoshani@ieeci.com |

* Via teleconference

The following topics were discussed:

1. Introductions and Points of Contact,
2. Review of Scope of Work including US EPA Comments on the former Parcel B Piers Investigation,
3. Bathymetric Surveys,
4. Final Feasibility Study (FS) Addendum in progress, and
5. Deliverables/Tasks and Schedule.

## SAP Worksheet #10: Conceptual Site Model

### SITE LOCATION

HPNS is located in the southeastern part of San Francisco on a long promontory that extends east into San Francisco Bay (**Figure 1-1**). HPNS comprises 866 acres: 420 acres on land and 446 acres under water in the San Francisco Bay (Parcel F). In 1939, the Navy obtained ownership of HPNS for ship-building, repair, and maintenance. After World War II, activities at HPNS shifted to submarine maintenance and repair. HPNS was also the site of the Naval Radiological Defense Laboratory. HPNS was deactivated in 1974 and remained relatively unused until 1976. Between 1976 and 1986, the Navy leased most of HPNS to Triple A Machine Shop, Inc., a private ship-repair company. In 1987, the Navy resumed occupancy of HPNS.

HPNS property was placed on the National Priorities List in 1989 pursuant to CERCLA because past shipyard operations had left hazardous substances onsite. In 1991, HPNS was designated for closure pursuant to the Defense Base Closure and Realignment Act of 1990. Closure at HPNS involved conducting environmental remediation and making the property available for non-defense use. To facilitate site cleanup and property transfer, HPNS was originally divided into six parcels, Parcels A through F (**Figure 1-2**). Parcel A has been transferred to the City of San Francisco and is no longer federal government property.

### NATURE AND EXTENT OF CONTAMINATION

During a series of investigations at Parcel F from 1991 to 2016, the Navy collected samples from shoreline and offshore locations to determine whether hazardous substances have been released at Parcel F. Potential contamination at Parcel F is associated with metals (copper, mercury, and lead) and polychlorinated biphenyls (PCBs) in sediment. A current conceptual site model for Parcel F sediments is provided in detail in the FS (Barajas, 2008) and the FSDGs Investigation Technical Memorandum (**Figures 2-2** and **2-3**; Barajas and Associates, 2007).

Detailed evaluations of the nature and extent of contamination are presented in the 2005 Validation Study (Battelle, BBL, and Neptune & Company, 2005), the 2007 FS Data Gaps Investigation Report (Barajas and Associates, 2007), and the FS Report (Barajas, 2008), and FS Addendum (KCH, 2017). The results of the investigations relevant to sediments adjacent to the former pier sites are below.

Elevated concentrations of copper were detected in Area III surface sediments during previous investigations. The highest concentration of copper (6,550 milligrams per kilogram [mg/kg]) was reported in a sample collected immediately offshore of the northeast of Point Avisadero (Area III, **Figures 1-2** and **2-1**). Concentrations of lead and mercury were also reported in surface sediments above the regulatory threshold values.

Elevated concentrations of copper and mercury were also reported in shallow subsurface sediments, although the distribution of exceedances were localized and discontinuous. The highest concentrations of copper in subsurface sediments (above 500 mg/kg) were reported within approximately 200 feet from the shoreline and extended to depths of approximately 2.0 to 3.0 feet below mudline (bml). Reported concentrations of mercury in subsurface sediments were highest (above 2 mg/kg) within 200 feet of the shoreline from locations north and northeast of Point Avisadero. The highest concentration of mercury (252 mg/kg) was reported in a sample collected from 1.0 to 1.5 feet bml northeast of Point Avisadero (Barajas, 2008).

Concentrations of total PCBs exceeding the regulatory threshold were reported during numerous investigation activities at Area III. The highest total PCB concentrations (between 2,000 and 6,000 micrograms per kilogram [µg/kg]) were detected in subsurface sediment samples collected between 1.5 to 3.0 feet bml at two locations east to northeast of Point Avisadero.

The horizontal and vertical distribution of constituents of concern (COCs) in Area III sediments are localized and discontinuous rather than exhibiting a gradient away from a well-defined source. COCs did not tend to co-occur, suggesting impacts are from episodic discharges (Barajas, 2008). With the exception of mercury, no COCs have been detected above the regulatory thresholds in sediment samples collected from Area I. Mercury was detected slightly above the regulatory threshold of 0.71 mg/kg in a single subsurface sediment sample collected between 2 to 4 feet bml (Barajas, 2008).

The primary chemicals of concern for ecological receptors at Point Avisadero are copper and mercury. Additionally, debris, metal slag, and/or riprap along the shoreline in Area III (Point Avisadero) are of concern because they may act as future sources of contamination to offshore sediments (KCH, 2017). These shoreline sites are being remediated in conjunction with activities in the adjacent upland Parcel B.

Target treatment zones for Area III were identified in the FS based on the "do not exceed" preliminary remediation goals (PRGs). Based on this analysis, copper and mercury exceeded the PRGs on an area-weighted average in Area III.

The final "do-not-exceed" remediation goals (RGs) for sediment at Parcel F presented in the FS are as follows:

- Copper: 271 mg/kg,
- Mercury: 1.87 mg/kg, and
- Total PCBs: 1,240 µg/kg.

A numerical remediation goal was not calculated for lead because of the uncertainty associated with both the bioavailability and toxicity of lead.

### Data Gaps in Conceptual Site Model

In 2011, demolition and removal of several wooden pier and wharf structures associated with Parcels B and C, within the boundary of Parcel F, was conducted (ERS, 2012). The associated structures included pilings and pier structures at Submarine Piers B and C, the wooden portion of the submarine quay wall, the wooden remnants of Berths 61 and 64, and the wooden decking and pilings at Wharf No.2 (**Figure 2-1**). Sampling is necessary to assess if sediments in close proximity to and beneath the former piers are impacted by previously identified COCs at Parcel F and whether erosional and bathymetric changes have occurred since removal in 2011.

## SAP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements

Project quality objectives (PQOs) define the type, quantity, and quality of data that are needed to answer specific environmental questions and support environmental decisions. PQOs are developed using a systematic planning process described in the Guidance for the Data Quality Objective (DQO) Process (EPA, 2006). The DQOs consist of the following seven iterative steps:

1. State the problem,
2. Identify the goals of the study,
3. Identify information inputs,
4. Define the boundaries of the study,
5. Develop the analytic approach,
6. Specify performance or acceptance criteria, and
7. Develop the plan for obtaining data.

**The project specific DQOs are described below:**

### 1. State the Problem

Sediment sampling will be performed at the locations of several former piers and a wharf structure at Parcel F in order to evaluate if COCs are present in the footprint of the former piers or former wharf. A bathymetric survey will be conducted in the area of the former piers and wharf structure in order to determine if bathymetric changes have occurred since removal of the piers and wharf structure. Current bathymetry surveys of Areas III, IX, and X are also required to accurately estimate the areas and volumes of sediments requiring remediation, to identify the placement of fill and capping materials during implementation of the selected alternative, and to estimate sediment erosion/deposition compared to historic data. The bathymetric survey will also be used to determine whether the installation of cofferdams is a viable option in Areas IX/X.

### 2. Identify the Goals of the Study

The principal study questions that must be addressed are:

- Are the levels of COCs (copper [Cu], mercury [Hg], lead [Pb], and PCBs) at the former pier sites below the project action limits?
- Have erosion or bathymetric changes occurred in and around the former piers, wharf, Area III, Area IX, or Area X?
- Are cofferdams a viable option in Areas IX/X during remediation?

### 3. Identify the Information Inputs

**Table 11-1** identifies the information inputs.

**Table 11-1 – Information Inputs**

| Input | Data Quality Level (Screening or Definitive)[1] |
|---|---|
| Bathymetric Survey | Screening Data -<br>• Survey Area III with a multi-beam bathymetric echo sounder, Survey Area IX/X with a single-beam echo sounder,<br>• Survey area adjacent to former piers, and former wharf, and<br>• Survey areas and line paths are provided in **Figure 3-1.** |
| Sediment Sampling | Definitive Data – a total of 36 individual surface sediment sub-samples will be collected in the field. The individual surface sediment sub-samples will be collected from within Parcel F at the following former pier and wharf structure removal locations and composited by the laboratory to create 9 surface composite sediment samples :<br>• **Former Berth 64 –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,<br>• **Former Berth 61 –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,<br>• **Former Pier B –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,<br>• **Former Pier C –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, |

| | |
|---|---|
| | • **Former Quay Wall** –Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory ,<br>• **Former Wharf #2** – Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, and<br>• **Figure 3-2** shows the proposed sampling locations for all the individual composite sediment sub-samples that will be collected.<br>• If laboratory composited sediment samples exceed ½ Project Action Levels (PALs) [RAO 2 – Area Weighted average for PCBs], all individual sub-samples that make up the composite will be analyzed for all the same site COCs as the composite as well. |
| [1] Data which will be compared to PAL (Worksheet #15) and will be used to make decisions for Parcel F will be considered definitive data. Data which is not quantifiable, is not compared to a PAL, or used to estimate subsurface conditions will be considered screening level data. | |

**4. Define the Boundaries of the Study**

The proposed lateral boundaries of the study include the survey areas and sampling locations shown on **Figures 3-1 and 3-2**. The proposed lateral boundaries for Areas III, IX, and X were defined in the Final FS Report for Parcel F (Barajas, 2008). Temporal boundaries are indicated by the timeframe shown on the project schedule in Worksheet #16. Results of the activities will be presented in the Sediment Investigation Summary Report.

Samples will be analyzed for Site COCs. Site COCs with RGs can be found in Worksheet #15 and are presented as PALs. PALs are equivalent to the PRGs established in the FS (Barajas, 2008), with the exception of lead. The established PAL for lead is the National Oceanic and Atmospheric Administration (NOAA) Effects Range Median (ER-M) value for the protection of the benthic community (NOAA, 1999). Sample analysis by location can be found in Worksheet #18.

**5. Develop the Analytic Approach**

- If composite sediment sample COCs are above ½ the PALs (RAO 2 – Area Weighted average PRG for PCBs, RAO1 – Do-Not-Exceed PRGs for Cu and Hg, and ER-M value for Pb), then individual sub-samples retained by the laboratory remaining after preparation of the original composite sample, will be analyzed for site COCs. The individual sub-samples will be screened against the RAO1 Do-Not-Exceed PRGs or ER-M value for Pb. Please note that the laboratory will be requested to retain the remaining individual sub-samples after they have prepared composite samples, and

- If composite sediment sample COCs are below the ½ the PALs (RAO2- Area Weighted Average PRg for PCBs, RAO1 – Do-Not-Exceed PRGs for Cu and Hg, and ER-M value for Pb), then no further action will be necessary for the associated pier area, and
- If individual sediment sub-sample COC results are above the Do-Not-Exceed PALs, then there will be a discussion with the Navy and the BCT to determine the next course of action.
- If individual sediment sub-sample COC results are below the Do-Not-Exceed PALs, then no further action will be necessary for the associated pier area.
- If bathymetric survey results are conducive to cofferdams for Areas IX/X, then cofferdams may be utilized during remediation in these areas per specifications identified during the remedial design.
- If the bathymetric survey shows no erosion or bathymetric changes, then no further action will be necessary, and
- If the bathymetric survey shows significant bathymetric changes, then the results will be reviewed and reported to the Navy with input from the BCT to determine if any further action is needed.

**6. Specify Performance or Acceptance Criteria**

Site COCs, proposed sampling locations, and PALs are based on results from previous investigations at the site and the *Addendum to the Feasibility Study Report for Parcel F, Feasibility Study Data Gaps Investigation* (KCH, 2017). To minimize sampling error, samples will be collected by ECC-Insight subcontract personnel trained in the collection of sediment samples using procedures described in this Uniform Federal Policy- Quality Assurance Project Plan.

Samples for chemical analysis will be analyzed by C&T, a Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP)-certified laboratory. Laboratories will perform the analysis in accordance with the methods presented in Worksheet #15 and the standard operating procedures (SOPs) listed in Worksheet #23.

Analytical data reported by C&T will be validated by a third party, LDC, and loaded into NIRIS. Analytical data collected only for IDW or waste-characterization for off-site disposal will not be validated or loaded into NIRIS.

All samples submitted to C&T will be analyzed by an industry-standard EPA method. Project quantitation limits (PQLs) for chemical analytical methods are listed on Worksheet #15. The PALs for this project are also shown in Worksheet #15. If the PAL for an analyte is below the PQL, then the limit of detection (LOD) will be used (Worksheet #15) and non-detect results at the LOD will be considered to be an acceptable result for determining that PALs have been met.

Data collected with field instruments will be reviewed with the manufacturer's guidance on precision and accuracy.

Data validation will be conducted in accordance with EWI No. 1 (NAVFAC Southwest, 2001), this SAP, *Department of Defense Quality Systems Manual (version 5.1)*, and *EPA National Functional Guidelines for Superfund Organic Methods Data Review* (EPA, 2014), *EPA National Functional Guidelines for Inorganic Superfund Data Review* (EPA, 2014), *General Data Validation Guidelines* (DoD EMDQW, 2017), and *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA, 2009). Validated data will be assessed for usability as described in Worksheet #37. Data assessment and validation will determine if collected data can be used for comparison. Data validation will be performed to Level III (Stage 2b in EMDQW guidance) for 80% of the COC data and Level IV (Stage 3 in EMDQW guidance) for 20% of the COC data. Data used from secondary sources, listed on Worksheet

#13, was reviewed for usability by the organization which generated and compiled the data, as described within the report narratives.

Evaluation of the site analytical results will include a review of the final, validated, definitive data. This collective body of knowledge will be used to identify any possible problems with the data, and minimize decision errors.

## 7. Develop the Plan for Obtaining Data

Bathymetric survey and sediment sampling will be conducted in accordance with the criteria discussed above in Worksheet #11, Worksheet #14, and Worksheet #17, including any referenced SOPs, and at the locations shown in **Figures 3-1 and 3-2**. Should field conditions require adjustment of sampling locations, frequency, and/or depths, modifications to such must be made in consideration of achieving the project goals described above.

Bathymetric survey and sediment sampling will be conducted by onsite field staff and subcontractors as scheduled in Worksheet #16. Samples will be sent to C&T for analysis. The laboratories will produce hard copy reports and a NEDD file for upload to NIRIS. Validation will be performed by LDC using the data verification and validation procedures described in Worksheets #34-36. Hard copies of the raw data reports and validation reports will be submitted to NAVFAC Southwest to be included in the NAVFAC Southwest Administrative Record. Worksheet #14 describes the data management process in detail.

## SAP Worksheet #12: Field Quality Control Samples for Sediment Samples

| QC Sample | Analytical Group | Frequency | Data Quality Indicators | Measurement Performance Criteria |
|---|---|---|---|---|
| Equipment Blank | PCBs and metals | 1 per sampling event per non-disposable piece of sampling equipment. | Contamination – accuracy/bias | ≤ Project LOQs |
| MS/MSD | PCBs and metals | 5% | Precision Accuracy/Bias | Varies. See WS#28 |
| Field Duplicates | PCBs and metals | 10% | Homogeneity/Precision | <30% |
| Field Blank | PCBs and metals | 1 per event | Contamination – accuracy/bias | ≤ Project LOQs |
| Temperature Indicator | PCBs and metals | 1 per cooler | Accuracy/Bias | 4 °C ± 2°C |

QC samples will only be analyzed for the parameters analyzed in the parent samples.
Analytical methods are listed on Worksheet #15.
Due to the proximity of the laboratory to the site, samples may not have cooled completely upon arrival. Elevated sample temperatures for samples received the same day as sampled will not be considered QC failures.

## SAP Worksheet #13: Secondary Data Criteria and Limitations Table

| Secondary Data | Data Source (Originating organization, report title, and date) | Data Generator (Originating organization, data types, data generation / collection date) | How Data Will Be Used | Limitations on Data Use |
|---|---|---|---|---|
| Analytical Data and Site Specific COCs | 2017 – Final Addendum to the Feasibility Study Report for Parcel F, Hunters Point Shipyard, San Francisco, CA (CH2M Hill Kleinfelder Joint Venture [KCH]) | KCH, sediment data, radiological survey data | To provide background data on existing conditions at the Site, and to guide sediment sampling activities. | No known factors affecting the reliability of the data and no known limitations on data use. |
| Analytical Data, Proposed Sample Locations, and Site Specific COCs | 2008 – Final Feasibility Study for Parcel F, Hunters Point Shipyard, San Francisco, CA (Barajas and Associates [Barajas]) | Barajas, soil and sediment data | To provide background data on existing conditions at the Site, and to guide sediment sampling activities. | No known factors affecting the reliability of the data and no known limitations on data use. |
| Analytical Data | 2007 - Technical Memorandum, Hunters Point Shipyard Parcel F, Feasibility Study Data Gaps Investigation. Hunters Point Shipyard, San Francisco, CA (Barajas and Associates) | Barajas and Associates, soil and sediment data | To provide background data on existing conditions at the Site. | No known factors affecting the reliability of the data and no known limitations on data use. |
| Analytical Data | 2012 - Final Removal Action Completion Summary Report, Pier Radiological Surveys and Removal, Hunters Point Naval Shipyard, San Francisco, California. (ERS Joint Venture [ERS]) | ERS, soil and sediment data | To provide background data on existing conditions at the Site. | No known factors affecting the reliability of the data and no known limitations on data use. |

## SAP Worksheet #14: Summary of Project Tasks

This worksheet summarizes the major tasks for this project.

### Preparation

*Site Access*

Prior to site mobilization all necessary authorization will be obtained. Due to the nature of the survey and sampling, the Coast Guard will be notified prior to any activity involving boats.

*Permitting*

- In accordance with Section 121(e) of CERCLA 1980 (CERCLA, 42 United States Code, Section 9621[e]), as amended, no federal, state, or local permits shall be required for the portion of any removal or remedial action conducted entirely onsite,
- Substantive compliance with applicable permit requirements will be met,
- ECC-Insight team responsible will coordinate with the Navy to obtain any permits determined to be necessary, and
- Navy RPM, ROICC, Coast Guard, and appropriate HPNS security and fire department personnel will be notified of work.

*Site Survey*

- Survey will include locations and estimated depths of sediment samples collected, and
- Locations of each sediment sample will be surveyed using a boat-mounted Global Positioning System (GPS) with coordinate off-set for the relative positions of the GPS and sampler on the boat, and taken at the time of collection. All data will conform to the North American Datum (NAD) 83 Zone III (feet) for horizontal datum.

*Equipment Staging / Storage*

- Majority of equipment staged/stored in existing building space,
- Acceptable location for storage will be coordinated with the Navy prior to mobilization,
- Work is anticipated during daylight hours; generally 7 AM and 7 PM, and
- Weekend work will be coordinated with HPNS personnel as needed.

*Investigation-Derived Waste Management*

- A Waste Management Plan has been prepared and describes the management of investigation-derived waste. The Waste Management Plan is included as Appendix C of the Work Plan.

### Data Gap Investigation

*Bathymetric Survey*

Bathymetric surveys will be conducted at Area III and adjacent former pier structure areas with a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder, and at Area IX/X with a survey skiff equipped with a single-beam echo sounder.

*Sediment Sampling*

Sediment samples will be collected from 6 sites within Parcel F. Each sediment sample will be a composite of up to 4 sub-samples collected from within each site. The number of sub-samples will be based on the overall sediment area at each site. A minimum of 1 sub-sample will be collected per 4,000 square feet, and a minimum of 4 sub-samples will be collected from each site. Samples will be collected from a boat using a Van Veen grab sampler or equivalent. A total of 36 individual sub-samples will be collected in the field. The individual surface sediment sub-samples will be composited by the laboratory to create 9 surface composite sediment samples from each of the former pier areas. Individual sub-samples will be composited

by the laboratory, with a portion of the individual sub-samples retained by the laboratory, if further COC analyses are required.

- **Former Berth 64 (~16,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Berth 61 (~6,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Pier B (~40,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Pier C (~40,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Quay Wall (~15,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, and
- **Former Wharf #2 (~24,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory.

Proposed sample locations for each site can be found on **Figure 3-2**.

**Procedures**

*Bathymetric Survey*

Bathymetric surveys will be conducted within three areas: 1) area in the footprint and adjacent to sections of the former Parcel B and C shoreline piers and wharfs structures (ERS, 2012); 2) Area III; and 3) Area IX/X (**Figure 3-1**). Due to the differences in water depths between the north and south areas, different survey technologies are proposed. For the deeper north areas (former Parcel B and C piers, wharfs and Area III) a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder will be utilized. For the shallow south areas (Area IX/X) a survey skiff equipped with a single-beam echo sounder will be utilized. Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet apart. Since the depth to water in Area IX/X is less than 6 feet (Battelle, BBL, and Neptune & Company, 2005), the 100-foot spacing for the deeper areas and 50-feet apart in the shallower areas (Area IX/X). A narrow single-beam (3°) transducer will be utilized for data acquisition within Area IX/X. Due to the fan shape of the emitted sound pulses, the width of the transducer footprint is variable based on the depth to seafloor along each survey line. The width of coverage is equal to approximately 5% of the distance between the water surface and the seafloor (e.g., At 10’ above the seafloor the transducer will achieve approximately 0.5’-wide coverage along the survey track, while at 5’ above the seafloor it will achieve approximately 0.25’-wide coverage along the survey track). For the deeper waters beneath the

former pier and wharf structures and Area III, the multi-beam survey provides greater resolution (see Section 3.1.1) that will identify change in bathymetry between the 100-foot lines. The resulting bathymetric survey data will be processed to provide 1-foot contours. Proposed survey areas and approximate line paths are provided on **Figure 3-1**.

The surveys will be conducted in general accordance with the procedures defined in the United States Army Corp of Engineers (USACE) Design Hydrographic Survey Manual (USACE, 2013). Horizontal and vertical positions will be acquired with a Real Time Kinematic (RTK) Global Positioning System (GPS), or equivalent. A base station will be set up on the GPS survey control marker used in previous bathymetric surveys of the areas. A Trimble dual frequency GPS receiver will be used in RTK mode on this control point. The unit transmits Radio Technical Commission for Maritime (RTCM) Services corrections to the system on board the survey vessel to improve horizontal and vertical positioning to better than 0.5-meters (1.6 feet) and provide accurate inertial navigation through GPS outages for up to 30 seconds. Position data will be used in real-time to provide navigation information to the vessel operator. A preliminary coverage plot with survey information is generated in real-time to show survey coverage. Water surface measurements will be obtained by RTK GPS with on-the-fly ambiguity resolution. A quality control point will be established using RTK techniques on a nearby dock, if possible, to confirm real time tide levels collected with the Hypack navigation software aboard the vessel. If a viable dock QC control point cannot be established, direct water line measurements will be made from the RTK Rover to confirm Hypack RTK tide readings as collected.

#### MULTI-BEAM SURVEY SYSTEM

The bathymetric survey within the deeper north area (former Parcel B and C piers, wharfs and Area III) will be conducted with a high resolution multi-beam system, such as a R2 Sonic 2024, or equivalent (**Figure 3-1**). The R2 Sonic 2024 works on user selectable frequency ranges so it is adaptable to a wide range of survey depths and conditions. Frequencies can be adjusted during the survey without having to shut down the sonar system, change hardware or halt recording data. This system produces a user selectable swath width of 10° to 160° using all 256 beams. The selected swath angle can also be rotated port or starboard, while recording, to direct the highly concentrated beams towards the desired target. The multi-beam swath width is approximately 4 times the water depth below the multi-beam sensor, providing detailed resolution in the deeper areas. The R2 Sonic 2024 will output digital depth data to the Hypack/Hysweep acquisition system where it will be stored with navigation data, motion sensor data, and heading data that will be applied in post processing.

#### SINGLE-BEAM SURVEY SYSTEM

The bathymetric survey at Area IX/X will be conducted with a survey grade digital single-beam echo sounder, such as Teledyne Odom Echotrac CV100, or equivalent (**Figure 3-1**). This system provides highly accurate single-beam profiling depth data below the vessel. The Odom Echotrac CV100 echo sounder collects digital profile records as well as digitized depth information for output to the navigational computer. Digital depth data is logged directly to the navigation computer along with date, time, and position for post processing and mapping. The GNSS antenna is mounted directly above the single beam transducer resolving any offset issues.

### SOUND VELOCIMETER

A sound velocimeter, such as an AML Smart Probe, will be used during all surveys to record high resolution sound velocity profiles of a water column to depths of 5000 meters. Sound velocity will be measured directly using an acoustic time of flight sensor rather than calculated from conductivity, temperature and depth measured parameters, which is critical for obtaining accurate range corrections for bathymetric surveys.

### HORIZONTAL AND VERTICAL CONTROLS

The horizontal datum for this survey will be NAD 83) State Plane Coordinates California Zone III, Epoch 2007.00. Both horizontal and vertical datum will be measured in U.S. survey feet.

A Meridian Attitude and Heading Reference System, or equivalent, will be used to provide vessel roll, pitch, heave, and heading. This data will be used by the data collection and processing software to compensate for the dynamic motion of the vessel when converting the sonar relative bearing and ranges of the soundings to XYZ positions on the earth, relative to the survey grid and vertical datum.

### SURVEY ACCURACIES

The bathymetric survey will generally conform to the accuracy standards provided in the USACE Hydrographic Survey Manual (USACE, 2013). Conforming to the bottom coverage requirement will be contingent upon unlimited access to the survey area and survey system limitations.

### SURVEY DATA PROCESSING AND REPORTING

Following the initial field data collection, data processing is required to obtain accurate position information from the GPS. Before exporting data to a computer aided design (CAD) system, the data will be reviewed to remove any data anomalies and adjust for GPS positional accuracy.

Bathymetric data will be collected in a straight path, perpendicular to flow. However, due to the often difficult navigational conditions caused by wind, barge traffic and floating obstructions the resulting cross section sometimes deviates from a straight line. CAD software will be used as needed to interpolate data to a line that approximates the straight-line path the boat was attempting to navigate.

Hypack/Hysweep software will be used to process and convert multi-beam sounding data into elevations. During post-processing, the multi-beam data will be corrected for tide, heave, pitch, roll, and speed of sound, and anomalous data will be removed. Multi-beam calibration offsets from patch test results will also be applied during the data editing process. During editing and processing, each survey line will be individually reviewed. This review consists of visual and automated inspection of speed of sound data, RTK tides, RTK GPS position data, motion sensor data, and sounding data. Anomalous data that are obvious system errors or “noise” within the water column, such as air bubbles, suspended particles and fish, and bottom multiples, will be filtered from the final data set. Manual editing will be based on a comparison of data outliers with surrounding data points, backscatter imagery, and file notes.

Resulting data sets from this bathymetric survey will be compared against 2003 bathymetric survey data and a comparison map will be provided to assist in showing areas of sediment deposition and erosion.

*Sediment Sampling*

Surface sediment samples will be collected from the survey vessel with a Van Veen grab sampler or equivalent. A stainless steel Van Veen grab sampler will be used to collect surface (within the top 5 centimeters) sediment samples. The grab scoop has a sampling area of 14" (35.6 centimeters) by 11" (27.9 centimeters), and has a maximum penetration depth of 10" (25.4 centimeters) but is dependent on lithology and hardness of sediment. The sample volume (mass) collected with the grab sampler is maximum of approximately 24 Liters per deployment. If debris or rocks prevent sampling from the proposed sample location, then the vessel will be relocated as close to the original location as possible so that an acceptable grab sample can be collected. Upon retrieval of the sampler, the sediment sample will be inspected for disturbance (e.g., excessive washing or improper sampling device closure), and if deemed acceptable, the sample will be logged and placed in a clean sample container. If the sample retrieval is unacceptable, the sample will be containerized for disposal as IDW and a new sample will be collected. If needed, sediment samples will be removed from the grab sampler with a dedicated disposable sampling spoon, or equivalent. Samples will be properly labeled and immediately placed in a cooler.

*Equipment Decontamination*

Decontamination of all non-disposable sampling equipment will be performed to prevent the introduction of extraneous materials into samples and to prevent potential cross-contamination between samples. Decontamination of non-disposable sampling equipment, including the Van Veen sampler, will be conducted as follows:

1) The equipment will be cleaned after each sample location with a brush, using laboratory grade detergent (e.g., Liquinox) and a potable water solution; rinsed with potable water and rinsed again with deionized water. The equipment will be scrubbed and rinsed in three separate five-gallon buckets, and
2) The equipment will be reassembled and placed in a clean area on plastic.

Decontamination will be performed after each sample location on non-disposable sampling equipment.

Equipment rinsates will be collected and analyzed for target compounds to provide a QC check on the decontamination procedure.

*Investigation Derived Waste Management*

Site activities will generate IDW including personal protective equipment, decontamination fluids, wastewater, and sediment from sampling activities. IDW generated on the boat will be placed into a sealable waste container and later transferred to a 55-gallon drum stored on site. IDW will be sampled and submitted for laboratory analyses in accordance with the waste facility requirements and will be handled and disposed of in accordance with procedures described in the Waste Management Plan, Appendix C of the Work Plan.

*Survey Data*

Survey data will be recorded by a field surveyor and also entered into the database by the ECC-Insight Data Manager as follows:

- All sediment sample locations will be surveyed in accordance with EWI EVR.6, Environmental Data Management and Required Electronic Delivery Standards (NAVFAC SW 2005), and
- For NEDD deliverables, horizontal survey will be captured (to the nearest 0.01-foot) in NAD 83 Zone III (feet) for horizontal datum.

All survey data which is manually entered will be QC checked by another team member.

*Compositing*

Sample compositing will be performed by the laboratory. For each analysis a portion of each subsample equal to approximately its portion of the required sample mass will be removed for compositing with the portions from other sub-samples. A 4 sub-sample composite requiring 30 g of sample would have ~7.5 g collected from each sub-sample. Full lab procedure can be found in Attachment A-2.

*Data Management*

Data will be generated from the activities described in Worksheets #11 and #17. The ECC-Insight FTL will be responsible for sample tracking from the planning phase to sample delivery to the laboratories. The intent of the tracking process is to ensure that all proposed samples are collected, named properly, located properly, recorded properly on chain-of-custody forms, analyzed for the proposed analytes, validated correctly, and recorded accurately in the data deliverables.

The ECC-Insight FTL will describe sampling activities and deviations to the ECC-Insight Chemist/Data Manager and provide the field data, which will be recorded in the field logbook, on field forms included as Attachment A1-1, and on chains-of-custody as described in Worksheet #27. Field data from the chains-of-custody (date and time collected, sample identification, etc.) will be entered into the database by the ECC-Insight Chemist and will be QC checked by another team member.

Laboratories will verify the sample receipt and document it in a sample receipt form. Samples will be assigned a unique number and recorded in the laboratory internal sample management software. Labs will send a confirmation email to the ECC-Insight Chemist describing the samples which were received, the planned analysis, and any problems identified during sample login. The ECC-Insight Chemist will review sample logins against the field logbook, field forms, and chain-of-custody to verify the laboratory login.

All data reported by the analyst must be reviewed by a peer analyst qualified to perform the method, and a supervisor, prior to reporting the data. The laboratory QA manager must annually review 10 percent of the data reported for each section (may be completed after the data have been reported). All data will be reported on or before the designated turnaround time by email.

On or before 21 calendar days from sample receipt, Labs will submit hard-copy data with associated QC information, along with an electronic data deliverables (EDD) including the NEDD files. Geotechnical data and physical properties data will be delivered in hardcopy and electronic reports on or before 21 calendar days from sample receipt.

The ECC-Insight Data Manager will upload the EDDs to an ECC-Insight Microsoft Access database. Ten percent of the data will be checked by the ECC-Insight Chemist against the hard-copy data package. Any discrepancies in the database or EDD will either be corrected by ECC-Insight Data Manager, or C&T will be notified to make corrections. The ECC-Insight Chemist will review the data upon receipt prior to releasing it to the ECC-Insight Data Manager to verify that the sampling procedures and analytical results were obtained following the protocols in this SAP and are of sufficient quality to satisfy DQOs. The ECC-Insight Data Manager will QC data before it is distributed to the end users and all manually entered data will be QC checked by another team member.

The EDD from Labs will be compatible with NEDD requirements and will be uploaded into NIRIS as specified in EWI #6. IDW sample results will not be uploaded NIRIS.

PDF's of laboratory SDGs, data validation packages, and EDDs will be stored on an ECC-Insight server for the duration of the project +7 years. Physical copies of laboratory SDGs and data validation packages will be stored onsite at ECC-Insight for the duration of the project +7 years. In addition, all reports and data packages are archived by NAVFAC SW Administrative Records in accordance with Navy policy and guidance.

*Data Validation*

The laboratory will report data by submitting data packages. For this project, 80 percent of the data will be submitted in an US EPA Level III-equivalent data package (Stage 2b), and 20 percent submitted in an US EPA Level IV-equivalent data package (Stage 3). Final data will be validated by LDC, an independent third-party data validation company. Upon finishing their review, LDC will issue a validation report. Worksheet #18 identifies the samples that will undergo level IV (Stage 3) data validation.

The ECC-Insight chemist will review the validation report provided by LDC for accuracy and completeness. Data validation qualifiers will be uploaded into the Microsoft Access database and checked by another team member prior to distribution and upload to NIRIS.

## SAP Worksheet #15.1: Reference Limits and Evaluation Tables for PCBs by EPA 8082

Soil

| Analyte | Chemical Abstract Service (CAS) # | PAL (µg/kg) | PAL Reference | PQL Goal (µg/kg) | LOQ (µg/kg) | LOD (µg/kg) | DL (µg/kg) |
|---|---|---|---|---|---|---|---|
| PCB-1016 | 12674-11-2 | NE | NE | 12 | 12 | 6 | 3.0 |
| PCB-1221 | 11104-28-2 | NE | NE | 24 | 24 | 16 | 8.0 |
| PCB-1232 | 11141-16-5 | NE | NE | 12 | 12 | 8 | 3.9 |
| PCB-1242 | 53469-21-9 | NE | NE | 12 | 12 | 8 | 3.6 |
| PCB-1248 | 12672-29-6 | NE | NE | 12 | 12 | 8 | 3.8 |
| PCB-1254 | 11097-69-1 | NE | NE | 12 | 12 | 6 | 3.1 |
| PCB-1260 | 11096-82-5 | NE | NE | 12 | 12 | 6 | 1.9 |
| PCBs (total) | NA | 1240[1]/1350[2]/200[3] | PRGs | NA | NA | NA | NA |

1. RAO 1 – Not to exceed threshold
2. RAO 2 – Area weighted average
3. RAO 3 – long-term goal based on an Area weighted average background near-shore PCB concentration

PALs are based on the PRGs established in the FS (Barajas, 2008)
Sub-Sample Analysis: If the composite sample exceeds ½ the RAO 2 Area-weighted average PRG for PCBs, all sub-samples associated with the parent sample will be analyzed for all tests.

Note: Limits are verified on a quarterly basis per DoD Quality Systems Manual (QSM) and may be subject to change. Posted limits were established April 2017. Any changes to these Limits which impact the project SAP objectives, must be approved by the NAVFAC SW RPM and QAO in advance of sample testing. Results will be reported on a dry-weight basis for comparison to the PAL/PSL.

µg/kg: micrograms per kilogram
PQL: Practical Quantitation Limit
NE: No PAL has been established for this compound
LOQ: Limit of quantitation
LOD: Limit of detection
DL: Method detection limit
CAS#: Chemical Abstract Service number

# SAP Worksheet #15.2: Reference Limits and Evaluation Tables for metals by EPA 6020/7471A

Soil

| Analyte | CAS # | Project action level [2] (µg/kg) | Project action level Reference | Project Quantitation Limit Goal (µg/kg) | LOQ (µg/kg) | LOD (µg/kg) | DL (µg/kg) |
|---|---|---|---|---|---|---|---|
| Copper | 7440-50-8 | 271,000[1] | PRGs | 259 | 259 | 200 | 86.4 |
| Lead | 7439-92-1 | 218,000 | ER-M | 250 | 250 | 200 | 73 |
| Mercury | 7439-97-6 | 1,870[1] | PRGs | 33 | 33 | 10 | 6 |

1. RAO 1 – Not to exceed threshold.

2. PALs are based on the PRGs established in the FS (Barajas, 2008), with the exception of lead. The PAL for lead is based on the NOAA ER-M value for the protection of the benthic community (NOAA, 1999).

Sub-Sample Analysis: If the composite sample exceeds ½ the PAL for metals, all sub-samples associated with the parent sample will be analyzed for all tests.

Note: Limits are verified on a quarterly basis per DoD QSM and may be subject to change. Posted limits were established May 2017. Any changes to these Limits which impact the project SAP objectives, must be approved by the NAVFAC SW RPM and QAO in advance of sample testing. Results will be reported on a dry-weight basis for comparison to the PAL/PSL.

µg/kg: micrograms per kilogram
PQL: Practical Quantitation Limit
LOQ: Limit of quantitation
LOD: Limit of detection
DL: Method detection limit
CAS#: Chemical Abstract Service number

# SAP Worksheet #16: Project Schedule/Timeline Table

## Table 16-1: Project Schedule

| Description | Date |
|---|---|
| Draft Sediment Investigation and Bathymetric Survey Work Plan/SAP | July 2017 |
| Approval of Final Sediment Investigation and Bathymetric Survey Work Plan/SAP | November 2017 |
| Conduct Sediment Investigation, Bathymetric Survey Field Work, and Laboratory Results:<br><br>• Mobilize field crew and equipment<br>• Collect composite samples of former piers and wharf structure and conduct bathymetry (10 day TAT)<br>• Request additional sampling of individual sub samples pending results of composite sampling above (as needed)<br>• Complete Bathymetric Survey<br>• Laboratory Validation Complete | January 15 – March 31, 2018<br><br>January 15, 2018<br>January 15- 31, 2018<br><br>January 25-31, 2018<br><br>January 31, 2018<br>March 31, 2018 |
| Draft Sediment Investigation and Bathymetric Survey Summary Report | July 2018 |
| Final Sediment Investigation and Bathymetric Survey Summary Report | November 2018 |

Note: Schedule and duration may change pending subcontractor availability, weather conditions, and field logistics as well as regulatory and Navy approvals.

## SAP Worksheet #17: Sampling Design and Rationale

Data gathered during this sampling program will:

1. Provide bathymetric survey data to provide updated bathymetric information in support of the remedial design for Parcel F and provide an estimate of change in sediment bed bathymetry in comparison to previous 2003 investigation, and
2. Fill in the data gap by providing data on sediment COCs at 6 former Parcel B and C pier and wharf structure locations within Parcel F that were removed in 2011 (ERS, 2012).

Worksheet #14 summarizes the activities that will be performed during the field investigation. A description of activities is provided below.

*Bathymetric Survey*

Bathymetric surveys will be conducted within Parcel F Subareas IX/X and III, including the former Parcel B and C pier and wharf structure areas removed in 2011 (ERS, 2012) (**Figure 3-1**). Due to the differences in exposed water between the areas, as well as the different survey technologies suitable for differing water depths, it is proposed that Area III is surveyed with a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder, and Area IX/X is surveyed with a survey skiff equipped with a single-beam echo sounder. Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet apart for the deeper areas and 50-feet apart in the shallower areas (Area IX/X). Since the depth to water in Area IX/X is less than 6 feet (Battelle, BBL, and Neptune & Company, 2005), the 50-foot spacing in this shallow area will provide sufficient resolution with the single beam survey. A narrow single-beam (3°) transducer will be utilized for data acquisition within Area IX/X. Due to the fan shape of the emitted sound pulses, the width of the transducer footprint is variable based on the depth to seafloor along each survey line. The width of coverage is equal to approximately 5% of the distance between the water surface and the seafloor (e.g., At 10’ above the seafloor the transducer will achieve approximately 0.5’-wide coverage along the survey track, while at 5’ above the seafloor it will achieve approximately 0.25’-wide coverage along the survey track). For the deeper waters beneath the former pier and wharf structures and Area III, the multi-beam survey provides greater resolution that will identify change in bathymetry between the 100-foot lines. The resulting bathymetric survey data will be processed to provide 1-foot contours. Proposed survey areas and line paths are provided on **Figure 3-1**.

Horizontal and vertical positions will be acquired with an RTK GPS, or equivalent. A base station will be set up on the GPS survey control marker used in previous bathymetric surveys of the areas. A Trimble dual frequency GPS receiver will be used in RTK mode on this control point. The unit transmits RTCM corrections to the system on board the survey vessel to improve horizontal and vertical positioning to better than 0.5-meters (1.6 feet) and provide accurate inertial navigation through GPS outages for up to 30 seconds. Position data will be used in real-time to provide navigation information to the vessel operator. A preliminary coverage plot with survey information is generated in real-time to show survey coverage. Water surface measurements will be obtained by RTK GPS with on-the-fly ambiguity resolution. Differential corrections for the vessel position will be conducted against the GPS survey control points identified in Figure 1-2. A quality control point will be established using RTK techniques on a nearby dock, if possible, to confirm real time tide levels collected with the Hypack navigation software aboard the vessel. If a viable dock QC control point cannot be established, direct water line measurements will be made from the RTK Rover to confirm Hypack RTK tide readings as collected.

The surveys will be conducted in general accordance with the procedures defined in the USACE Design Hydrographic Survey Manual (USACE, 2013).

Results from the Bathymetric survey will provide a 1-foot contour of the sediment surface, which will be used to estimate any future sediment removal should it be required, as well as identify any debris on the surface of the sediment.

*Sediment Sampling*

A total of 36 individual sediment sub-samples will be collected in the field at six sites within Parcel F (**Figure 3-2**). The individual sub-samples will be collected in the field. The sub-samples will be composited for each former pier area by the laboratory. At each former pier area, individual sub-samples will be collected for the composite at a rate of one sub-sample per 4,000 sf, with a minimum of 4 sub-samples collected for compositing at each site. The proposed individual sub-samples to be collected for each former pier area are described below:

- **Former Berth 64 (~16,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Berth 61 (~6,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Pier B (~40,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Pier C (~40,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,
- **Former Quay Wall (~15,000 sf) –** Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, and
- **Former Wharf #2 (~24,000 sf) –** Eight (8)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment samples and analyze the composite samples for Cu, Hg, Pb, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory.

Labeled samples will be sent to the lab for compositing. Sediment samples will be analyzed for PCBs by EPA 8082, Cu and Pb by EPA 6020, and Hg by EPA 7471A. Proposed sample locations can be found on **Figure 3-2**. Sample locations with analyses can be found in WS#18. In the event that composite sample analytical results exceed ½ the PALs (e.g., RAO 2 – Area Weighted average for PCBs) established, all the individual sub-samples that make up that composite will be analyzed for all the same COCs (Cu, Hg, Pb, and PCBs). Laboratory analysis will be performed on a 10 business day turnaround time (TAT) for preliminary data, which will allow for ample time to review the data for PAL exceedances that would require subsample analysis, per the hold times listed in WS#19.

## SAP Worksheet #18-1: Location-Specific Sampling Methods/SOP Requirements Table for Sediment Sampling

| Sample Event | ID Number / Sampling Location[1,4] | Matrix | Depth (below water surface)[2] | Analytical Group | Number of Samples[3] | Sampling SOP Reference |
|---|---|---|---|---|---|---|
| Sediment Sampling | HPP-FSS-001 / Berth 64 Area | Sediment | Varies | Metals, PCBs | 1 composite (4-sub-samples in composite) | WS#14 |
| Sediment Sampling | HPP-FSS-002 / Berth 61 Area | Sediment | Varies | Metals, PCBs | 1 composite (4-sub-samples in composite) | WS#14 |
| Sediment Sampling | HPP-FSS-003 / Former Pier B Area | Sediment | Varies | Metals, PCBs | 2 composites (8-sub-samples in two composites) | WS#14 |
| Sediment Sampling | HPP-FSS-004 / Former Pier C Area | Sediment | Varies | Metals, PCBs | 2 composites (8-sub-samples in two composites) | WS#14 |
| Sediment Sampling | HPP-FSS-005 / Former Quay Wall Area | Sediment | Varies | Metals, PCBs | 1 composite (4-sub-samples in composite) | WS#14 |
| Sediment Sampling | HPP-FSS-006 / Former Wharf #2 Area | Sediment | Varies | Metals, PCBs | 2 composites (8-sub-samples in two composites) | WS#14 |

Notes:
1. A minimum of 20% of samples (2 composite, 8 sub-samples) will be validated to level IV (Stage 3) and marked on the chain-of-custody,
2. Samples will be collected from approximately the upper five centimeters of the sediment surface using a Van Veen grab sampler or equivalent. Depth below surface of water will vary, and be noted in the field during sampling. Depth (vertical elevation) of samples will be calculated from the survey data, , and
3. One composite samples will be collected from each site. Number of sub-samples to be composited are listed in parentheses. Sub-sample locations are shown on **Figure 3-2**.
4. Sub-samples at each location will have the sample ID number appended with a letter from A-I. For example, HPP-FSS-001A, -001B, etc.

## SAP Worksheet #19: Field Sampling Requirements

| **Matrix** | **Analytical Group** | **Analytical Method** | **Containers** (number, size & type) | **Sample Volume** (units) | **Preservation Requirements** (chemical, temperature) | **Maximum Holding Time** (preparation / analysis) |
|---|---|---|---|---|---|---|
| Soil | PCBs | EPA 8082 | One 4 ounce jar | 30-g | Cool, ≤ 6ºC | 1 year (for samples kept at ≤ 6ºC but not frozen) / 40 days |
| Soil | Metals | EPA 6020/7471A | One 4 ounce jar | 5-g | Cool, ≤ 6ºC | 180 days/28 days for Hg |

Due to the proximity of the laboratory to the site, samples may not have cooled completely upon arrival. Elevated sample temperatures for samples received the same day as sampled will not be considered QC failures.

## SAP Worksheet #20: Field Quality Control Sample Summary Table

| Matrix | Analytical Group | No. of Sub-Sampling Locations / Composite Samples | No. of Field Duplicates | No. of MS/MSDs | No. of Field Blanks | No. of Equip. Blanks | No. of VOA Vial Trip Blanks | Total No. of Sub-Samples / Samples to Lab |
|---|---|---|---|---|---|---|---|---|
| Soil | PCBs | 36 / 9 | 4 / 1 | 1 per 20 | 1 | 1 | 0 | 48 / 14 |
| Soil | Metals | 36 / 9 | 4 / 1 | 1 per 20 | 1 | 1 | 0 | 48 / 14 |

Notes:
MS matrix spike
MSD matrix spike duplicate
PCB polychlorinated biphenyl
VOA volatile organic analysis

## SAP Worksheet #21: Project Sampling SOP References Table

Project sampling will be performed per Worksheet #14.

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

| Field Equipment | Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference | Comments |
|---|---|---|---|---|---|---|---|
| Multi-Beam Bathymetric Echo Sounder | Gyro Heading Calibration | One time prior to the start of the survey | 0.01 degree or less difference between the calculated heading values | Repeat Gyro Heading Calibration | Aaron Heidt/Mehdi Siavoshani | USACE (2013) | Not Applicable |
| | Patch Test Calibration | One time prior to the start of the survey | Zero latency between the position, heading, and altitude strings | Perform error adjustments and repeat Patch Test Calibration | Aaron Heidt/Mehdi Siavoshani | USACE (2013) | Not Applicable |
| Single-Beam Echo Sounder | Bar Check Calibration | Once a day, after any single-beam echo sounder pole deployment, or if the survey conditions varied considerably throughout the day | No variations between the true bar depth and observed depth | Adjust the index value and repeat Bar Check Calibration | Aaron Heidt/Mehdi Siavoshani | USACE (2013) | Not Applicable |
| GPS | Position Verification | One time prior to the start of the survey | ± 3 feet. | Repeat verification | Aaron Heidt/Mehdi Siavoshani | Manufacturers manual | Not Applicable |

## SAP Worksheet #23: Analytical SOP References Table

| Lab SOP Number | Title, Revision Date, and / or Number | Definitive or Screening Data | Matrix and Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? (Y/N) |
|---|---|---|---|---|---|---|
| semi-volatile organic compound (SVOC)-3.2 | PCBs by gas chromatograph (GC)-electron capture detector (ECD) Analysis Methods EPA 8082, EPA 8082A, EPA 608, & TO-10A; Rev 11, 2/20/15 | Definitive | Organic –GC-ECD in Water/Soil | GC-ECD | C&T | N |
| MET-4.6 | Metals By Inductively coupled plasma mass spectrometry (ICP-MS) EPA 6020, EPA 6020A, EPA 200.8; Rev. 10, 8/30/13 | Definitive | Inorganics - ICP-MS in Water/Soil | ICP-MS | C&T | N |
| MET-5.2 | Digestion & Analysis of Solid Samples for Mercury cold vapor atomic absorption (CVAA) Methods EPA 7471A / 7471B; Rev. 19, 1/22/16 | Definitive | Inorganics – CVAA in Soil | CVAA | C&T | N |
| XLAB 3.2.3 | Sonication (EPA 3550) Extraction of Soil Samples for PCB For GC-ECD Method EPA 8082; Rev. 13, 8/4/15 | Preparatory | Organic –GC-ECD in Soil | GC-ECD | C&T | N |
| MET 2.4 | Acid Digestion of Soil & Solid Samples For Total Metals Analysis by ICP-AES and ICP-MS | Preparatory | Inorganics – ICP-AES/ICP-MS in Soil | ICP-MS | C&T | N |

| Lab SOP Number | Title, Revision Date, and / or Number | Definitive or Screening Data | Matrix and Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? (Y/N) |
|---|---|---|---|---|---|---|
| MET 10.1 | Moisture (% Solids) In Soil & Sediment Samples US-EPA Contract Laboratory Program Method ILM05.3; Rev. 7, 6/30/15 | Definitive | Inorganic – Soil | Mass Balance | C&T | N |
| CS 2.3 | Subsampling & Compositing | Preparatory | Inorganic/Organic – Soil | Mortar & Pestle<br>Ring & Puck Mill<br>Jaw Crusher<br>Sieves<br>Sample Pans and Drying Racks | C&T | No |

## SAP Worksheet #24: Analytical Instrument Calibration Table

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person responsible for Corrective Action | SOP Reference |
|---|---|---|---|---|---|---|
| GC/ECD | Initial Calibration (ICAL) | When new instrument is installed, after major maintenance, and whenever Continuing Calibration Verification (CCVs) fail and minor maintenance does not correct the problem. | Minimum of 5 points (or 6 points for quadratic)<br><br>Avg RF, RSD < 20%, Linear r >0.995; Quadratic r2 >0.99 | If a single point is the outlier, that point may be rerun so long as no samples have been analyzed. If ICAL still fails, perform maintenance & recalibrate. | Lab analyst | SVOC 3.2 |
| GC/ECD | Initial Calibration Verification (ICV) | Immediately following ICAL curve, prior to sample analysis. | %D ≤ 20% for all target analytes | Correct the problem and, so long as no samples have been analyzed, rerun the ICV. If samples were run or the rerun fails, perform maintenance & recalibrate. If samples were analyzed, non-detects associated with a high response may be reported. | Lab analyst | SVOC 3.2 |
| GC/ECD | CCV | At beginning of each analytical sequence, after every 10 field samples and at the end of the sequence. | %D ≤ 20 for all target analytes | Perform maintenance & recalibrate if necessary. Non-detects associated with a high response may be reported; rerun all others. | Lab analyst | SVOC 3.2 |
| ICP-MS | Tune Check | Beginning of analytical sequence, prior to ICAL, and every 12 hours. | Mass calibration ≤ 0.1 amu from the true value; resolution < 0.9 amu full width at 10% peak height. | Retune instrument and rerun check; if still out, perform instrument maintenance and retune instrument, then rerun tune check. | Lab analyst | MET 4.6 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person responsible for Corrective Action | SOP Reference |
|---|---|---|---|---|---|---|
| ICP-MS | Initial Calibration (ICAL) | Daily, prior to sample analysis or whenever CCVs fail and maintenance does not correct the problem.<br><br>Minimum 3 points + a blank | Linear coefficient: $r \geq 0.995$ | Perform maintenance & recalibrate. | Lab analyst | MET 4.6 |
| ICP-MS | Initial Calibration Verification (ICV) | Immediately following ICAL curve, prior to sample analysis, using a second source standard. | $\%D \leq 10$ | Perform maintenance & rerun. If still out, recalibrate. | Lab analyst | MET 4.6 |
| ICP-MS | Low Level ICV (LLICV, CRI) | Concentration $\leq$LOQ; run after mid-level ICV and before samples. | $\%D \leq 20\%$ | Perform maintenance & rerun. If still out, recalibrate. | Lab analyst | MET 4.6 |
| ICP-MS | Initial Calibration Blank | Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence. | < LOD | Flush system and rerun. If contamination persists, perform instrument maintenance and recalibrate. | Lab analyst | MET 4.6 |
| ICP-MS | Interference Check Standard A (ICS-AB) | Beginning of each sequence, after the ICAL and before samples. | Unspiked elements: < \| LOQ \| | Perform instrument maintenance and rerun any elements that are not verified trace impurities from one of the spiked analytes. | Lab analyst | MET 4.6 |
| ICP-MS | (ICS-A) | Beginning of each sequence, after the ICAL and before samples. | Absolute value of concentration for all nonspiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); | Perform instrument maintenance and rerun any samples requiring affected elements. | Lab analyst | MET 4.6 |
| ICP-MS | CCV | After every 10 field samples and at the end of the sequence. | $\%D \leq 10$ | Perform maintenance & recalibrate if necessary. Non-detects associated with a high response may be reported; rerun all others. | Lab analyst | MET 4.6 |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action | Person responsible for Corrective Action | SOP Reference |
|---|---|---|---|---|---|---|
| ICP-MS | Continuing Calibration Blank | Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence. | No analytes detected > LOD. | Flush system and rerun. If contamination persists, perform instrument maintenance and recalibrate. | Lab analyst | MET 4.6 |
| ICP-MS | Internal Standards | Add to every sample, batch QC, and standard. | %Recovery: 30-120 | Dilute and reanalyze for elements associated with failing IS | Lab analyst | MET 4.6 |
| CVAA | Initial Calibration (ICAL) | Daily, prior to sample analysis.<br><br>Minimum of 5 points and a blank. | Coefficient (r) $\geq$ 0.995 – or $r^2 \geq 0.99$ | Perform maintenance & recalibrate. | Lab analyst | MET 5.2 |
| CVAA | Initial Calibration Verification (ICV) | Immediately following ICAL curve, prior to sample analysis. | %D $\leq$ 10 | Perform maintenance & rerun. If still out, recalibrate. | Lab analyst | MET 5.2 |
| CVAA | Initial Calibration Blank | After ICV and before samples. | <LOD | Flush system and rerun. If contamination persists, perform instrument maintenance and recalibrate. | Lab analyst | MET 5.2 |
| CVAA | CCV | After every 10 field samples and at the end of the sequence. | %D $\leq$ 10 | Perform maintenance & recalibrate if necessary. Non-detects associated with a high response may be reported; rerun all others. | Lab analyst | MET 5.2 |
| CVAA | Continuing Calibration Blank | After each CCV. | No analytes detected > LOD. | Flush system and rerun. If contamination persists, perform instrument maintenance and recalibrate. | Lab analyst | MET 5.2 |

## SAP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, Inspection Table

| Instrument Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|
| GC/ECD | Change septa and liner, clean injection port, clip column | Analyze CCV and/or performance evaluation material (PEM) | | Daily for Preventative Maintenance | CCV and/or PEM passes criteria | Reinspect injection port, cut additional column, reanalyze CCV, recalibrate instrument | Analyst or Department Manager | SVOC-3.2 |
| GC/ECD | Clean Syringe | | No residue on plunger, plunger moves freely | Weekly for preventative maintenance | CCV passes criteria | Replace syringe | Analyst or Department Manager | SVOC-3.2 |
| GC/ECD | Bake out detectors | | Background levels decrease | Every 3 months for preventative maintenance | CCV passes, blanks clean | Rebake out detector, recalibrate, replace detector | Analyst or Department Manager | SVOC-3.2 |
| GC/ECD | Prime System with high level CCV | Analyze PEM | | Whenever maintenance performed on the system | PEM passes | Run another Primer | Analyst or Department Manager | SVOC-3.2 |
| ICP-MS Metals | Replace peristaltic pump tubing | Analyze tune check | Yellow, discolored, or cracked | Daily | Passes RSD criteria | Retune and recalibrate | Analyst or Department Manager | MET-4.6 |
| ICP-MS Metals | Clean nebulizer, spray chamber, and torch | Analyze CCV | | When CCV failing criteria | CCV passes criteria | Replace parts, recalibrate | Analyst or Department Manager | MET-4.6 |
| ICP-MS Metals | Clean cone | Analyze CCV | | CCV fails criteria, low response, distorted peak shape, vacuum pressure increases | CCV passes criteria, response increases, peak shape improves, pressure decreases | Replace, recalibrate | Analyst or Department Manager | MET-4.6 |

| Instrument Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|
| ICP-MS Metals | Clean extraction lens and Einzel lens assembly | Analyze Tune | | Tune won't pass, ion signal unstable | Instrument will tune | Replace parts | Analyst or Department Manager | MET-4.6 |
| ICP-MS Metals | Lubricate autosampler tracks | | Build-up visible on tracks | Every 6 months | Tracks clean | Replace tracks | Analyst or Department Manager | MET-4.6 |
| ICP-MS Metals | Replace vacuum pump oil | | | Every 6 months | | | Analyst or Department Manager | MET-4.6 |
| CVAA Mercury | Replace pump windings | Analyze ICAL | | Cracked or leaking | ICAL and CCVs pass criteria | Replace again | Analyst or Department Manager | MET-5.2 |
| CVAA Mercury | Clean drying tube | Analyze ICAL | | Glass has a yellow color | ICAL and CCVs pass criteria | Replace | Analyst or Department Manager | MET-5.2 |
| CVAA Mercury | Clean gas-liquid separator | Analyze ICAL | | Appears smudged or dirty | ICAL and CCVs pass criteria | Replace | Analyst or Department Manager | MET-5.2 |
| CVAA Mercury | Replace lamp | Analyze ICAL | | Voltage reaches 15mV | ICAL and CCVs pass criteria | Replace, call for service | Analyst or Department Manager | MET-5.2 |

## SAP Worksheet #26: Sample Handling System

| |
|---|
| **SAMPLE COLLECTION, PACKAGING, AND SHIPMENT** |
| Sample Collection (Personnel/Organization): Field Team / ECC-Insight |
| Sample Packaging (Personnel/Organization): Field Team / ECC-Insight |
| Coordination of Shipment (Personnel/Organization): Field Team / ECC-Insight |
| Type of Shipment/Carrier: Courier or Federal Express Priority Overnight |
| **SAMPLE RECEIPT AND ANALYSIS** |
| Sample Receipt (Personnel/Organization): Laboratory Sample Custodian, C&T |
| Sample Custody and Storage (Personnel/Organization): Laboratory Sample Custodian, C&T |
| Sample Preparation (Personnel/Organization): Analyst, C&T |
| Sample Determinative Analysis (Personnel/Organization): Analyst, C&T |
| **SAMPLE ARCHIVING** |
| Field Sample Storage (No. of days from sample collection): 30 days after delivery of the data package |
| Sample Extract/Digestate Storage (No. of days from extraction/digestion): 30 days after delivery of the data package |
| Biological Sample Storage (No. of days from sample collection): NA |
| **SAMPLE DISPOSAL** |
| Personnel/Organization: Laboratory Sample Custodian, C&T |
| Number of Days from Analysis: 30 days after delivery of the data package |

## SAP Worksheet #27: Sample Custody Requirements

The following sections describe sample handling procedures, including sample ID and labeling, documentation, chain-of-custody, and shipping.

**Sample Identification and Labeling**

Samples will be assigned unique sample numbers as specified in Worksheet #18. A sample label will be affixed to all sample containers. The label will be completed with the following information written in indelible ink:

- Project name and location,
- Sample ID number,
- Date and time of sample collection,
- Preservative used,
- Sample collector's initials, and
- Analysis required.

**Sample Documentation**

Documentation during sampling is essential to ensure proper sample identification. ECC-Insight personnel will adhere to the following general guidelines for maintaining field documentation:

- Documentation will be completed in indelible ink,
- All entries will be legible,
- Errors will be corrected by crossing out with a single line and then dating and initialing the lineout,
- Any serialized documents will be maintained at ECC-Insight and referenced in the site logbook, and
- Unused portions of pages will be crossed out, and each page will be signed and dated.

**Chain-of-Custody**

ECC-Insight will use standard sample custody procedures to maintain and document sample integrity during collection, transportation, storage, and analysis. A sample will be considered to be in custody if one of the following statements applies:

- It is in a person's physical possession or view,
- It is in a secure area with restricted access, and
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal.

Chain-of-custody procedures provide an accurate written record that traces the possession of individual samples from the time they are collected in the field to the time they are accepted at the laboratory. The chain-of-custody record will be used to document all samples collected and the analysis requested. Information that the field personnel will record on the chain-of-custody record includes the following:

- Project name and number,
- Sampling location,
- Name and signature of sampler,
- Destination of samples (laboratory name),
- Sample ID number,
- Date and time of collection,
- Number and type of containers filled,
- Analysis requested,
- Preservatives used (if applicable),
- Filtering (if applicable),
- Sample designation (grab or composite),

- Signatures of individuals involved in custody transfer, including the date and time of transfer,
- Airbill number (if applicable), and
- Project contact and phone number.

Unused lines on the chain-of-custody record will be crossed out. Field personnel will sign chain- of-custody records that are initiated in the field, and the airbill number will be recorded. The record will be inserted in a waterproof plastic bag and taped to the inside of the shipping container used to transport the samples. Signed airbills will serve as evidence of custody transfer between field personnel and the courier, and between the courier and the laboratory. Copies of the chain-of-custody record and the airbill will be retained and filed by field personnel before the containers are shipped.

Laboratory chain-of-custody begins when samples are received and continues until samples are discarded. The laboratory should designate a specific individual as the sample custodian. The custodian will receive all incoming samples, sign the accompanying custody forms, and retain copies of the forms as permanent records. The laboratory sample custodian will record all pertinent information on the samples, including the persons who delivered the samples, the date and time received, sample condition at the time of receipt (sealed, unsealed, or broken container; temperature; or other relevant remarks), the sample ID numbers, and any unique laboratory ID numbers for the samples. This information should be entered into a computerized laboratory information management system. When the sample transfer process is complete, the custodian is responsible for maintaining internal logbooks, tracking reports, and other records necessary to maintain custody throughout sample preparation and analysis. The laboratory will provide a secure storage area for all samples. Access to this area will be restricted to authorized personnel. The custodian will ensure that samples that require special handling, including samples that are heat- or light-sensitive, radioactive, or have other unusual physical characteristics, will be properly stored and maintained prior to analysis.

**Sample Shipment**

The following procedures will be implemented when samples collected during this project are shipped:

- The cooler will be filled with bubble wrap, sample bottles, and packing material,
- Sufficient packing material will be used to prevent sample containers from breaking during shipment,
- Enough ice will be added to maintain the sample temperature of 4 ± 2 °C,
- The chain-of-custody records will be placed inside a plastic bag. The bag will be sealed and taped to the inside of the cooler lid. The air bill, if required, will be filled out before the samples are handed over to the carrier. The laboratory will be notified if the sampler suspects that the sample contains any substance that would require laboratory personnel to take safety precautions,
- The cooler will be closed and taped shut with strapping tape around both ends. If the cooler has a drain, it will be taped shut both inside and outside of the cooler,
- Signed and dated custody seals will be affixed on the front and side of each cooler,
- Wide clear tape will be placed over the seals to prevent accidental breakage,
- The chain-of-custody record will be transported within the taped sealed cooler, and
- When the cooler is received at the analytical laboratory, laboratory personnel will open the cooler and sign the chain-of-custody record to document transfer of samples.

Multiple coolers may be sent in one shipment to the laboratory. The outside of the coolers will be marked to indicate the number of coolers in the shipment.

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## SAP Worksheet #28.1: Laboratory QC Samples Table for Metals by 6020

| | |
|---|---|
| **Matrix** | Soil/Water |
| **Analytical Group** | Metals |
| **Analytical Method/ SOP Reference** | SW-846 6020/ MET 4.6 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blank | 1 per batch of 20 or fewer samples | No analytes > ½ RL or > 1/10 the amount measured in any sample. For common laboratory contaminants, no analytes detected > the RL. | Report samples that are ND or >10x contamination. Identify & correct the problem then reprep and rerun all others. | Laboratory Analyst | Positive bias or false positives | No analytes > ½ RL or > 1/10 the amount measured in any sample. For common laboratory contaminants, no analytes detected > the RL. |
| laboratory control sample/laboratory control sample duplicate (LCS/LCSD) | 1 per batch of 20 or fewer samples | See control limits table for acceptance criteria | Low recovery: Reprep and rerun all samples.<br>High recovery: Reprep and rerun samples with results ≥LOQ. Report results <LOQ.<br>High relative percent difference (RPD): Report ND samples; reprep & rerun all others | Laboratory Analyst | Accuracy/ Bias | See control limits table for acceptance criteria |
| MS/MSD | 1 pair per batch of 20 or fewer samples | See control limits table for acceptance criteria | Run post-digestion spike. Flag results as possible matrix interference. | Laboratory Analyst | Accuracy/ Bias, Precision | See control limits table for acceptance criteria |

| | |
|---|---|
| **Matrix** | Soil/Water |
| **Analytical Group** | Metals |
| **Analytical Method/ SOP Reference** | SW-846 6020/ MET 4.6 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Serial Dilution | 1 for every batch of 20 or fewer samples | %D $\leq$ 10 | Flag results as possible matrix interference | Laboratory Analyst | Matrix Interferences | %D $\leq$ 10 |
| Post Digestion Spike | 1 per batch of 20 or fewer samples | Recovery: 80-120% | Dilute and rerun; if again outside limits, flag as possible matrix interference | Laboratory Analyst | Matrix Interferences | Recovery: 80-120% |
| Internal Standard | Every sample, batch QC sample, and standard | Recovery: 30-120% | Rerun sample; if repeated, narrate | Laboratory Analyst | Sensitivity, Bias | Recovery: 30-120% |

| Analyte | CAS number | Water Control Limits (%) | Water RPD Limits (%) | Soil Control Limits (%) | Soil RPD Limits (%) |
|---|---|---|---|---|---|
| Copper | 7440-50-8 | 85-118 | 20 | 84-119 | 20 |
| Lead | 7439-92-1 | 82-120 | 20 | 81-119 | 20 |

Limits in WS#28 are provided by the laboratory.

## SAP Worksheet #28.2: Laboratory QC Samples Table for Mercury by 7470

| | |
|---|---|
| **Matrix** | Soil/Water |
| **Analytical Group** | Metals |
| **Analytical Method/ SOP Reference** | SW-846 7470/7471<br>MET 5.1/5.2 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blank | 1 per batch of 20 or fewer samples | One per every analytical /preparation batch | Report samples that are ND or >10x contamination. Identify & correct the problem then reprep and rerun all others. | Laboratory Analyst | Positive bias or false positives | No analytes > ½ RL or > 1/10 the amount measured in any sample. For common laboratory contaminants, no analytes detected > the RL. |
| LCS/LCSD | 1 per batch of 20 or fewer samples | One LCS or LCS/LCSD pair per analytical /preparation batch | Low recovery: Reprep and rerun all samples.<br>High recovery:<br>Reprep & rerun samples with results ≥LOQ. Report results <LOQ.<br>High RPD: Report ND samples, reprep & rerun all others | Laboratory Analyst | Accuracy/ Bias | See control limits table for acceptance criteria |
| MS/MSD | 1 pair per batch of 20 or fewer samples | One MS/MSD per every 20 project samples per matrix | Flag results as possible matrix interference. | Laboratory Analyst | Accuracy/ Bias, Precision | See control limits table for acceptance criteria |

| Analyte | CAS number | Water Control Limits (%) | Water RPD Limits (%) | Soil Control Limits (%) | Soil RPD Limits (%) |
|---|---|---|---|---|---|
| Mercury | 7439-97-6 | 82-119 | 20 | 80-124 | 20 |

Limits in WS#28 are provided by the laboratory.

## SAP Worksheet #28.3: Laboratory QC Samples Table for PCBs by 8082

| Matrix | Soil/Water |
|---|---|
| Analytical Group | PCB |
| Analytical Method/ SOP Reference | SW-846 8082/ SVOC 3.2 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blank | 1 per batch of 20 or fewer samples | <1/2 LOQ, per (QSM) 5.0 | Report samples that are ND or >10x contamination. Identify & correct the problem then reprep and rerun all others. | Laboratory Analyst | Positive bias or false positives | <1/2 LOQ, per QSM 5.0 |
| LCS/LCSD | 1 per batch of 20 or fewer samples | See table below | Low recovery: Reprep and rerun all samples.<br>High recovery: Reprep & rerun samples with results ≥LOQ. Report results <LOQ<br>High RPD: Report ND samples, reprep & rerun all others | Laboratory Analyst | Accuracy/ Bias | See Table below |
| MS/MSD | 1 pair per batch of 20 or fewer samples | See table below | Flag results as possible matrix interference. | Laboratory Analyst | Accuracy/ Bias, Precision | See Table below |
| Surrogate Spike | Every sample and batch QC sample | See table below | Low recovery: Reprep & rerun affected sample<br>High recovery: | Laboratory Analyst | Accuracy/ Bias | See Table below |

| Matrix | Soil/Water |
|---|---|
| **Analytical Group** | PCB |
| **Analytical Method/ SOP Reference** | SW-846 8082/ SVOC 3.2 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| | | | If due to obvious coelution, flag and narrate without reanalysis. Otherwise, report results <LOQ; reprep & rerun results ≥LOQ | | | |
| Second Column Confirmation | Any results >DL | RPD ≤ 40% between primary & secondary column | If coelution is evident on one column, report from the other column, otherwise flag the result and narrate. | Laboratory Analyst | False Positives | RPD ≤ 40% between primary & secondary column |

| Analyte | CAS number | Water Control Limits (%) | Water RPD Limits (%) | Soil Control Limits (%) | Soil RPD Limits (%) |
|---|---|---|---|---|---|
| Aroclor-1016 | 12674-11-2 | 46-129 | 30 | 47-134 | 30 |
| Aroclor-1221 | 11104-28-2 | 60-140 | 30 | 50-150 | 30 |
| Aroclor-1232 | 11141-16-5 | 60-140 | 30 | 50-150 | 30 |
| Arochlor-1242 | 53469-21-9 | 80-176 | 30 | 50-150 | 30 |
| Aroclor-1248 | 12672-29-6 | 78-165 | 30 | 50-150 | 30 |
| Aroclor-1254 | 11097-69-1 | 34-127 | 30 | 67-135 | 30 |
| Aroclor-1260 | 11096-82-5 | 45-134 | 30 | 53-140 | 30 |
| **Surrogate** | | | | | |
| Decachlorobiphenyl | 2051-24-3 | 28-120 | | 25-135 | |
| TCMX | | 39-120 | | 44-130 | |

Limits in WS#28 are provided by the laboratory.

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## SAP Worksheet #29: Project Documents and Records Table

| Document | Where Maintained |
|---|---|
| Field logbook | ECC-Insight project file |
| Field screening results | ECC-Insight project file (Logbook) |
| Sample labels | Laboratories project files |
| Field change request form | ECC-Insight project file |
| Equipment calibration log/certificate of calibration | ECC-Insight project file (Logbook) |
| Photographic log | ECC-Insight project file (Logbook) |
| Chain-of-Custody | ECC-Insight project file and laboratories project file |
| Shipping records | ECC-Insight project file |
| Health and safety sign-off sheet | ECC-Insight project file |
| Daily reports | ECC-Insight project file |
| Laboratory data package | ECC-Insight project file, laboratories project files, and NAVFAC SW Administrative Record |
| Data validation reports | ECC-Insight project file, laboratories project files, and NAVFAC SW Administrative Record |

Note: copies of the field logbook, field screening results, field change request form, purge logs, photographic log, chain-of-custodies, and daily reports will be included in the final report. The final report will be maintained in the NAVFAC SW Administrative Record.
Laboratory data packages will include a mix of EPA Level III (Stage 2b) and Level IV (Stage 3), with all data being at least Level III (Stage 2b).
Level III (Stage 2b) data packages will include sample logins, COCs, analytical results (including LOQ/LOD/DL), results of QC data specified in WS#'s 12, 24, 25, and 28. (Stage 2b)
Level IV (Stage 3) data packages will include all Level III (Stage 2b) data as well as raw instrument data, sample weight forms, and chromatograms (where applicable) for specified samples to allow recalculation of final results. (Stage 3)

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## SAP Worksheet #30: Analytical Services Table

| Matrix | Analytical Group | Sample Locations/ID Numbers | Analytical SOP | Data Package Turnaround Time | Laboratory/Organization[1] (name and address, contact person and telephone number) | Backup Laboratory/Organization (name and address, contact person and telephone number) |
|---|---|---|---|---|---|---|
| Soil | Metals/mercury EPA 3050B/6020/7471 | Soil locations described on Worksheet #18 | MET-4.6<br>MET-5.2<br>MET 2.4<br>MET 10.1 | 10 day TAT preliminary / 21 day TAT final | Mike Dahlquist<br>Curtis & Thompkins Laboratories<br>2323 5th Street<br>Berkeley, CA 94710<br>510-486-0900 | Richard Beauvil<br>EMAX Laboratories, Inc.<br>1835 W. 205th Street<br>Torrance, CA 90501<br>310-618-8889 |
| Soil | PCBs EPA 3550/8082 | Soil locations described on Worksheet #18 | SVOC-3.6<br>XLAB 3.2.3<br>MET 10.1 | 10 day TAT preliminary / 21 day TAT final | Mike Dahlquist<br>Curtis & Thompkins Laboratories<br>2323 5th Street<br>Berkeley, CA 94710<br>510-486-0900 | Richard Beauvil<br>EMAX Laboratories, Inc.<br>1835 W. 205th Street<br>Torrance, CA 90501<br>310-618-8889 |

Notes:
1 Both primary and backup laboratories are a DoD ELAP and National ELAP certified laboratories.

## SAP Worksheet #31: Planned Project Assessments Table

| Assessment Type | Frequency | Internal or External | Organization Performing Assessment | Person(s) Responsible for Performing Assessment (title and organizational affiliation) | Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation) | Person(s) Responsible for Identifying and Implementing CA (title and organizational affiliation) | Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation) |
|---|---|---|---|---|---|---|---|
| Field Planning Meeting | Prior to initiating work | Internal | ECC-Insight | ECC-Insight PM | ECC-Insight PM and FTL | ECC-Insight QC Manager | ECC-Insight QC Manager |
| Field Sampling TSA | Once, on the first day of sediment sampling episodes | Internal | ECC-Insight | Field Team Lead | Site Geologist | FTL | ECC-Insight Program QC Manager |
| Laboratory Audit/Review of SOPs and representative data packages | Once, prior to start of analytical work | Internal | ECC-Insight | ECC-Insight Project Chemist | Laboratory QAM | Laboratory Technical Director/QAM | ECC-Insight Project Chemist |

## SAP Worksheet #32: Assessment Findings and Corrective Action Responses Table

| Assessment Type | Nature of Deficiencies Documentation | Individual(s) Notified of Findings (name, title, organization) | Timeframe of Notification | Nature of Corrective Action Response Documentation | Individual(s) Receiving Corrective Action Response (name, title, organization) | Timeframe for Response |
|---|---|---|---|---|---|---|
| Field Planning Meeting | Field Planning Meeting Form | ECC-Insight PM and QAM | 24 hours | Corrective Action Request Form | ECC-Insight PM and QAM | 24 hours |
| Management Review | Management Review Report | ECC-Insight PM and QAM | 24 hours | Corrective Action Request Form | ECC-Insight PM and QAM | Fifteen to 30 days from the date of the corrective action notice |

## SAP Worksheet #33: Quality Assurance Management Reports Table

| Type of Report | Frequency (daily, weekly monthly, quarterly, annually, etc.) | Projected Delivery Date(s) | Person(s) Responsible for Report Preparation (title and organizational affiliation) | Report Recipient(s) (title and organizational affiliation) |
|---|---|---|---|---|
| Status Reports | Monthly | 15th of every month | Mitra Fattahipour (ECC-Insight PM) | Sharon Ohannessian (NAVFAC RPM) |
| QC Reports | Daily | Following day | Dave Marks (ECC-Insight QC) | Sharon Ohannessian (NAVFAC RPM) |
| Management Review Report | Once during field activities | 10 days after management review | Nick Weinberger (ECC-Insight QAM) | Mitra Fattahipour (ECC-Insight PM) |

# SAP Worksheets #34-36: Data Verification and Validation (Steps I and IIa/IIb) Process Table

| Data Review Input | Description | Responsible for Verification (name, organization) | Step I / IIa / IIb [1] | Internal/ External |
|---|---|---|---|---|
| Field logbook | Field logbooks will be reviewed and placed in the project file upon project completion. | FTL (Mehdi Siavoshani)<br>QAM (Nick Weinberger) | Step I | Internal |
| Chain-of-custody forms | Chain-of-custody forms will be reviewed upon their completion and verified for completeness. A copy of the chain-of-custody will be retained in the project file, and the original and remaining copies taped inside the container for shipment. | FTL (Mehdi Siavoshani) | Step I | Internal |
| Sample logins | Sample login information will be reviewed and verified for completeness in accordance with the chain-of-custody forms. | Laboratory PM (Mike Dahlquist, C&T)<br>Project Chemist ( Nick Weinberger) | Step I | Internal<br>External |
| Laboratory data prior to release | Laboratory data will be reviewed and verified for completeness against analyses requested on the chain-of-custody forms. | Laboratory PM ( Mike Dahlquist, C&T) | Step I | External |
| Laboratory data packages | All laboratory data packages will be verified by the laboratory performing the work for completeness and technical accuracy prior to submittal. Data packages will then be reviewed by the Project Chemist for completeness. Subsequently, data packages will be evaluated externally according to the external data validation requirements specified in Worksheet #36 of this SAP, performed by a third party validator. Data packages will be validated based on the requirements of EWI No. 1, this SAP, NFGs, EPA validation labeling guidance, and DoD QSM 5.1. Data will be validated at 80% level 3 (Stage 2b), 20% level 4 (Stage 3). Geotechnical, and IDW data will not be validated, but will be verified by the laboratory. | Laboratory PM (Mike Dahlquist, C&T)<br>Project Chemist (Nick Weinberger)<br>FTL (Mehdi Siavoshani) | Step I | External<br>Internal |
| Electronic data deliverables (EDDs) | All EDDs will be verified by the chemical laboratory performing the work for completeness and technical accuracy prior to submittal. All received EDDs will be verified against the hard-copy laboratory data packages. EDDs will not be generated for geotechnical data. | Laboratory PM (Mike Dahlquist, C&T)<br>Project Chemist (Nick Weinberger) | Step I | External<br>Internal |
| Holding Times | Verify that samples were analyzed within holding times specified in method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made to the laboratory. Holding times are not applicable to geotechnical samples. | Laboratory PM and Analyst | Step I | Internal |
| Analytes | Verify that required lists of analytes were reported as specified in the SAP. | Laboratory PM and Analyst | Step I | Internal |
| Communication | Establish that required communication procedures were followed by field or laboratory personnel. | PM (Mitra Fattahipour)<br>QAM (Nick Weinberger) | Step IIa | Internal |
| Sampling Methods and Procedures | Establish that the required sampling methods were used and that any deviations were noted. Verify that the sampling | QAM (Nick Weinberger) | Step IIa | Internal |

| | | | | |
|---|---|---|---|---|
| | procedures and field measurements met performance criteria and that any deviations were documented. | | | |

## SAP Worksheets #34-36: Data Verification and Validation (Steps I and IIa/IIb) Process Table Cont.

| Data Review Input | Description | Responsible for Verification (name, organization) | Step I / IIa / IIb [1] | Internal/ External |
|---|---|---|---|---|
| Holding Times | Verify that samples were analyzed within holding times specified in method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made to the laboratory. Holding times are not applicable to geotechnical samples. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa | Internal |
| Analytes | Verify that required lists of analytes were reported as specified in the SAP. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa | Internal |
| Analytical Methods and Procedures | Verify that the required analytical methods were used and that any deviations were noted. Verify that the QC samples met performance criteria and that any deviations were documented. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa | Internal |
| Data Qualifiers | Verify that the laboratory data qualifiers were defined in the laboratory data package and applied as specified. Data qualifiers are NA for geotechnical samples. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa/IIb | Internal/External |
| Sampling Plan | Determine whether the sampling plan was executed as specified (i.e., the number, location, and type of field samples were collected and analyzed as specified in the SAP. | Project Chemist (Nick Weinberger) for chemical samples. FTL (Mehdi Siavoshani) for geotechnical samples. | Step IIb | Internal |
| Sampling Procedures | Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (e.g., techniques, equipment, decontamination, volume, and temperature, preservative). | Project Chemist (Nick Weinberger)<br>FTL (Mehdi Siavoshani) | Step IIb | Internal |
| Field Duplicates | Compare results of field duplicates with criteria established in the SAP. Field duplicates are not collected for geotechnical samples. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa/IIb | External |
| Sample Results | Verify that the required field QC samples were run and met required limits. | Project Chemist (Nick Weinberger)<br>Data validation PM(Shauna McKellar) | Step IIa/IIb | External |
| Project Quantitation Limits | Determine that quantitation limits were achieved, as outlined in the SAP. Quantitation limits are NA for geotechnical samples. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa/IIb | External |
| Performance Criteria | Evaluate QC data against performance criteria in the SAP. | Project Chemist (Nick Weinberger)<br>Data Validation PM (Shauna McKellar) | Step IIa/IIb | Internal/ External |
| Validation Packages | All validation packages will include a review and discussion of all QC Parameters indicated in WS#12, #24, and #28. | Validation PM (Shauna McKellar)<br>Project Chemist (Nick Weinberger) | Step IIa/IIb | Internal/ External |

## SAP Worksheet #37: Usability Assessment

The data usability assessment will be performed by a team of personnel at ECC-Insight. Analytical DQOs will be assessed through application of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters to ensure that the data of sufficient quality to meet the project objectives. The report will include a description of how PARCC parameters were evaluated, a discussion of any significant trends and biases in the QC results, a summary of the data validation findings, and sufficient information to support data usability conclusions.

The following subsections describe each of the PARCC parameters and how they will be assessed within this project.

### Precision

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

$$RPD = \frac{|A - B|}{\frac{(A + B)}{2}} \text{ X } 100\%$$

Where:

A = First duplicate concentration
B = Second duplicate concentration

Field sampling precision is evaluated by analyzing field duplicate samples. Field duplicates will be collected and analyzed at a frequency of 10 percent.

Laboratory analytical precision is evaluated by analyzing laboratory duplicates or MSs and MSDs. MS/MSD samples will be generated for all analytes for this project except compliance samples related to IDW samples. The results of the analysis of each MS/MSD pair will be used to calculate an RPD for evaluating precision. Acceptance criteria for precision is presented in Worksheet #28.

### Accuracy

Field accuracy will be assessed by collecting and analyzing equipment rinsate blanks, trip blanks, and source water blank QC samples as appropriate. These QC samples will be used to evaluate the potential for target analytes to enter samples as a result of sampling processes.

A program of sample spiking will be conducted to evaluate laboratory accuracy. This program includes analysis of the MS and MSD samples, LCSs or blank spikes, surrogate standards, and method blanks. MS and MSD samples will be prepared and analyzed at a frequency of 5 percent for site samples. LCS or blank spikes are also analyzed at a frequency of 5 percent. Surrogate standards, where applicable, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

$$\text{Percent Recovery} = \frac{S - C}{T} \text{ x } 100$$

Where:

S = Measured spike sample concentration

C = Sample concentration
T = True or actual concentration of the spike

Results that fall outside the accuracy goals, which are presented on Worksheet #28, will be further evaluated based on the results of other QC samples.

**Representativeness**

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. Representative data will be obtained for this project through selection of sampling locations, selecting analytical parameters based upon the sampling activity, and conducting multiple sampling events in some instances. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will also be ensured through the consistent application of established field and laboratory procedures. Field blanks (if appropriate) and laboratory blank samples will be evaluated for the presence of contaminants to aid in evaluating the representativeness of sample results. Data determined to be non-representative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

**Completeness**

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP. When all data validation is completed, the percent completeness value will be calculated by dividing the number of usable sample results by the total number of sample results planned for this investigation. The completeness goal for this project is 90%.

Completeness will also be assessed to determine if the samples which were planned for collection, were collected. The total number of samples planned will be compared to the total number of samples collected.

Completeness will also be evaluated as part of the data quality assessment process. This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected.

**Comparability**

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data.

**Sensitivity**

The DL is defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%. The LOD is defined as the smallest amount of concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. The LOQ is at the lowest concentration that produces a quantitative result with specified limits of precision and bias. Analytical methods for this project have been selected so that the LOQ for each target analyte is below the PALs listed in Worksheet #15, wherever practical.

**Specific Validation**

In addition to the evaluations noted above, all laboratory data will be evaluated during data validation for all QC Parameters listed in WS#12, #24, and #28.

**Data Quality Assessment**

After data are validated, the project chemist will review and assess field and laboratory QC. The PARCC parameters will be assessed as described above. Data validation reports will be reviewed and assessed for meeting DQOs. The Project Chemist will review and apply the data validation qualifiers to the final data. The following data qualifiers will be used:

J – Result is estimated

U – Analyte is not detected at or above the stated Quantitation Limit (QL)

R – Data are rejected

UJ – Analyte is not detected, but there is an uncertainty about the QL

Data qualifiers are used to indicate uncertainties associated with the data. The assigned qualifiers will be entered into the validation code field in the database.

The Project Chemist will prepare a data quality assessment report that will summarize the findings of the data assessment and discuss the usability of the data to be included in the report.

Data will be reported in tabular format to be included in the report. The electronic data in NEDD format will be submitted to the NIRIS database within 30 days of completion of validation, as described in EWI EVR.6, Environmental Data Management and Required Electronic Delivery Standards (NAVFAC Southwest 2005). An e-mail confirmation received by ECC-Insight will be forwarded to the project file.

## References

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_____. 2002. *EPA Guidance for Quality Assurance Project Plans, EPA/QA/G-5. EPA/240/R-02/009.* December.

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_____. 2005. *Environmental Work Instruction No.6: Environmental Data Management and Required Electronic Delivery Standards. NFECSW. San Diego, California.* April 19.

# Figures

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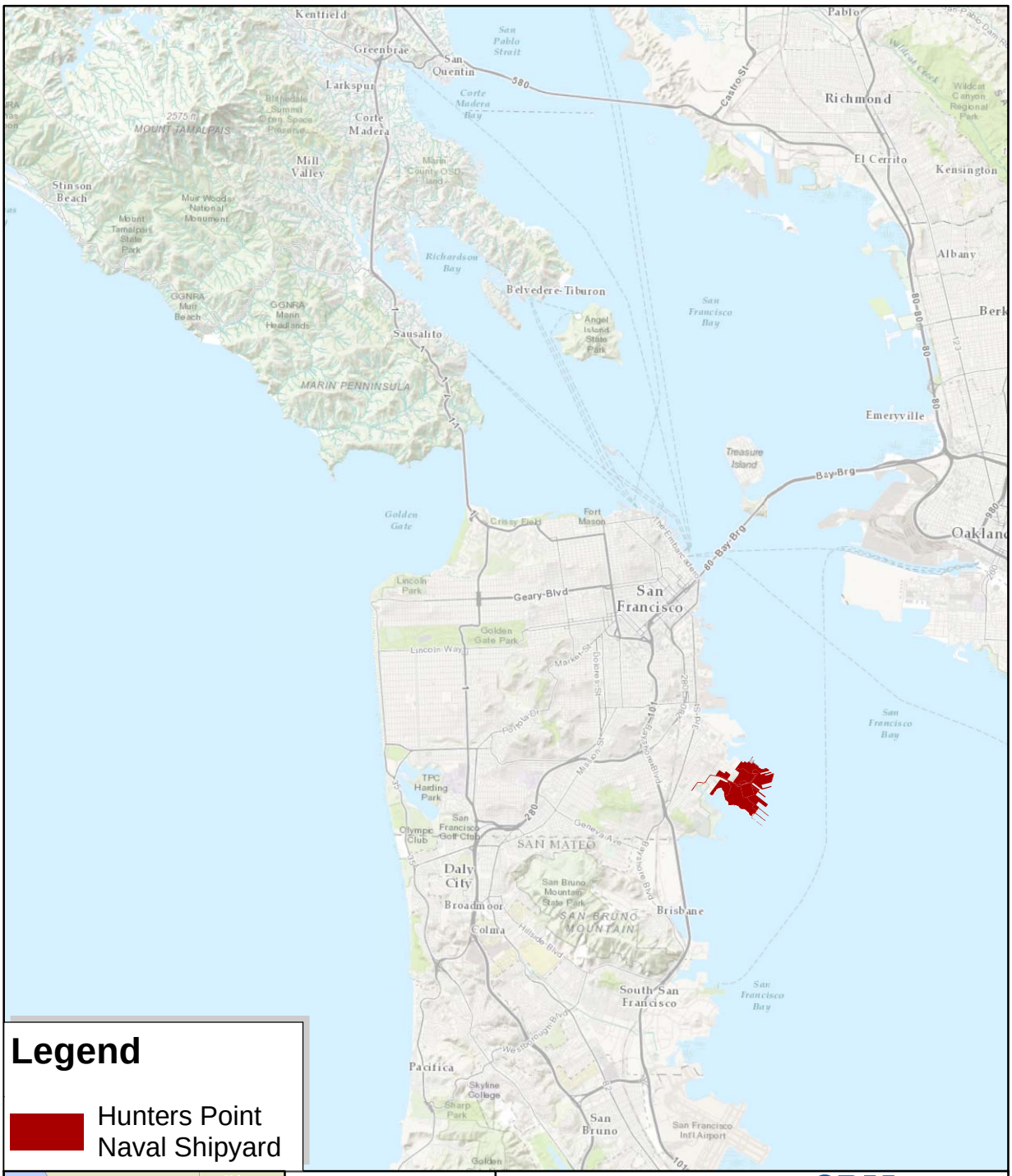

1.5 0 1.5
Miles

*1 inch = 3 miles*

ECC INSIGHT LLC

CDM Smith

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 1-1**
**Site Location Map**

Sediment Investigation and Bathymetric Survey for Parcel F

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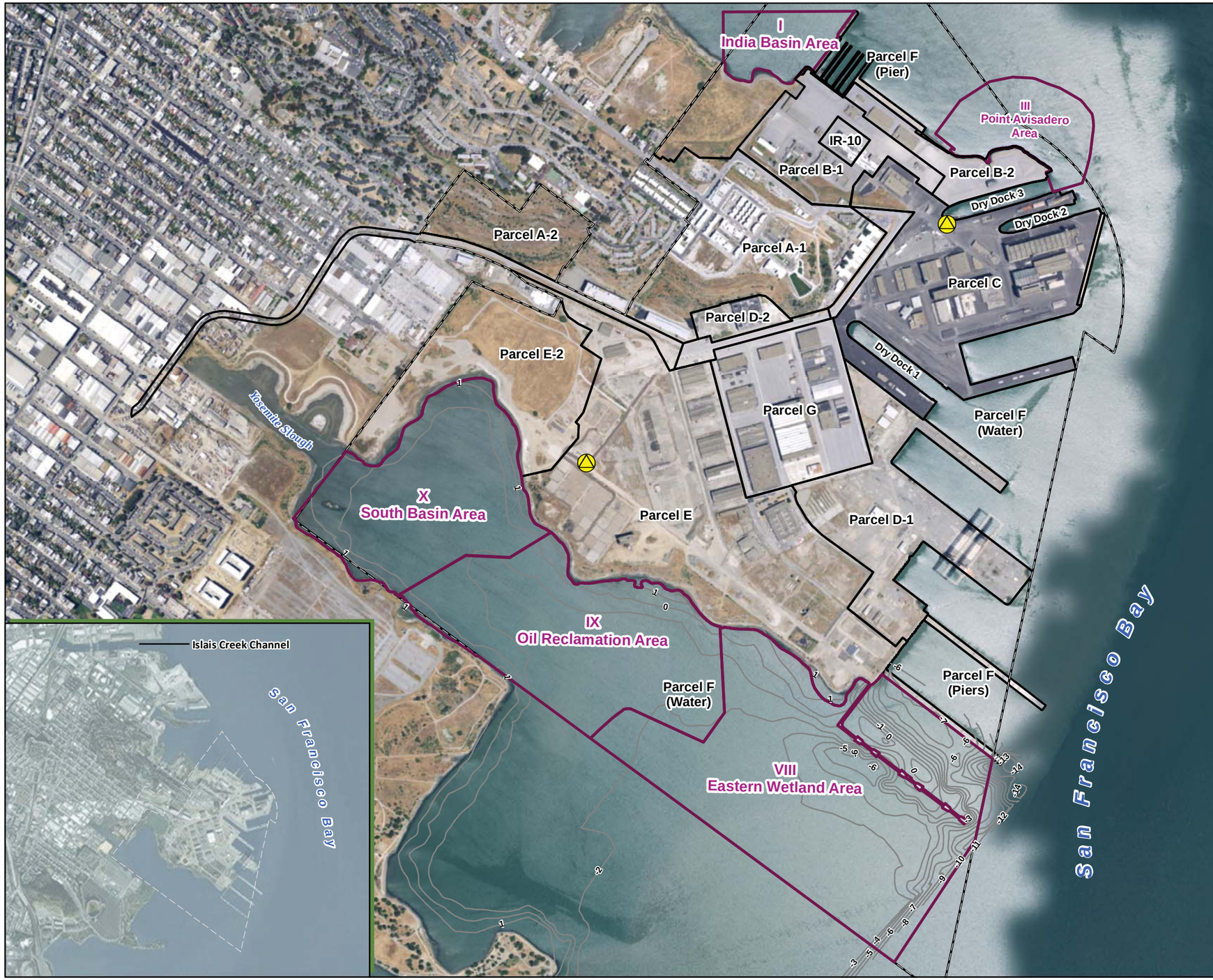


## Legend

- Navy Property Boundary
- Parcel F Subareas - Hunters Point Naval Shipyard
- Parcel Boundary
- Bathymetry Contour (Depth in meters Referenced to MLLW)
- Survey Control Point

Notes:
MLLW - mean lower low water

1,000 0 1,000
Feet
*1 inch = 1,000 feet*

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**U.S. Department of the Navy, BRAC PMO West**

**Figure 1-2**
**Parcel F Subareas**

Sediment Investigation and
Bathymetric Survey for Parcel F

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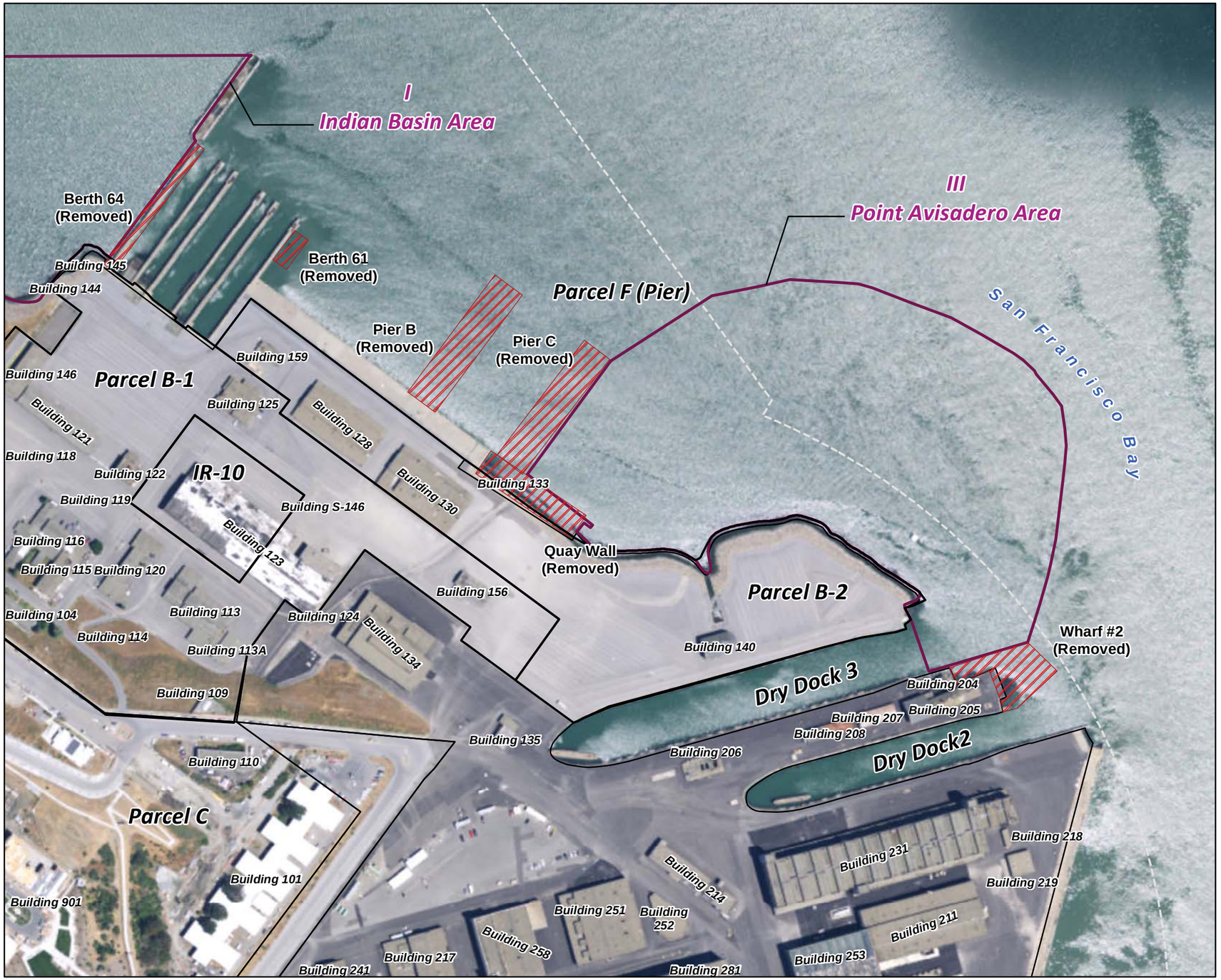


## Legend

- Navy Property Boundary
- Parcel Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Former Parcel B Pier Structure (Removed in 2011)

Note:
Utilities that existed on the piers were capped at the point of demolition on Berth 64 and Piers B & C at the points indicated by the hash marking (Demolition Area).

N

300 0 300
Feet
*1 inch = 300 feet*

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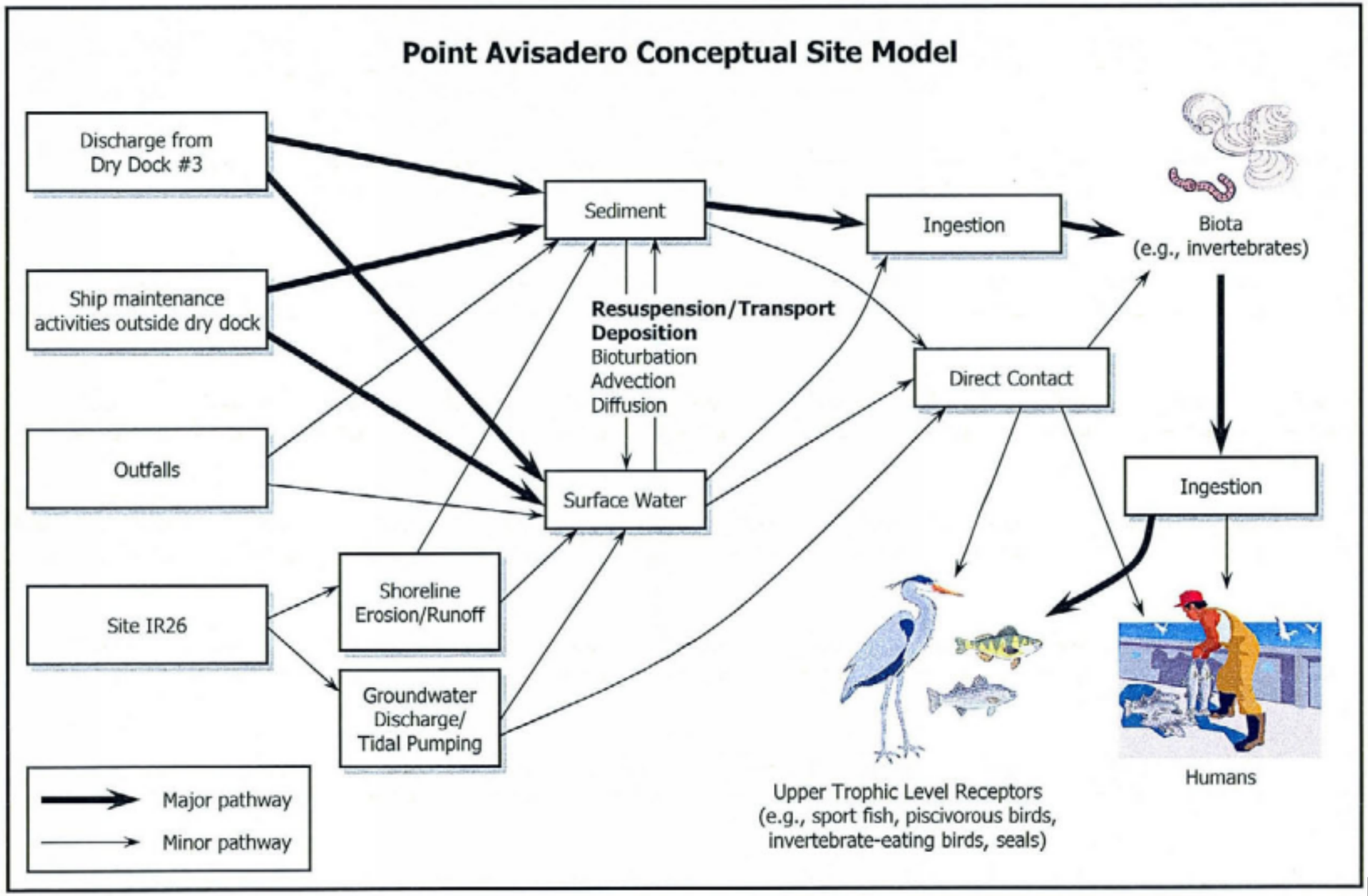
**U.S. Department of the Navy, BRAC PMO West**

**Figure 2-1**
**Former Parcel B Pier Structure Locations**

Sediment Investigation and
Bathymetric Survey for Parcel F

schellerka G:\GIS\212737_HP_ParcelF\Task8_SAP\DraftFinal\Figure2-1_FormerParcelBPierStructureLocations.mxd 9/22/2017

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U.S. Department of the Navy, BRAC PMO West

**Figure 2-2**
**Point Avisadero**
**Conceptual Site Model**

Sediment Investigation and
Bathymetric Survey for Parcel F

Source: Figure 3-3, Hunters Point Shipyard Parcel F Data Gaps Investigation (Barajas and Associates, 2007)

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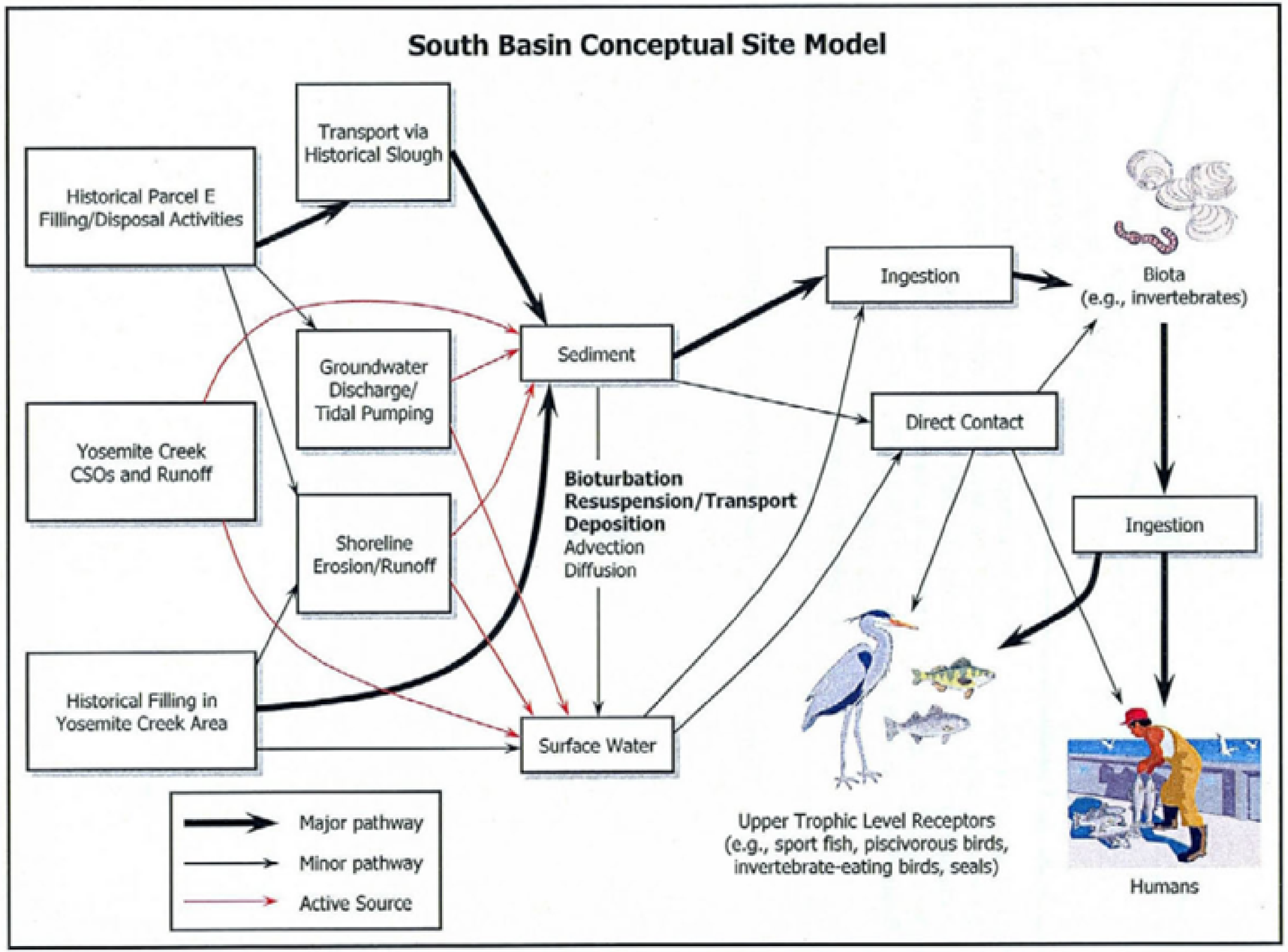


Source: Figure 3-3, Hunters Point Shipyard Parcel F Data Gaps Investigation (Barajas and Associates, 2007)

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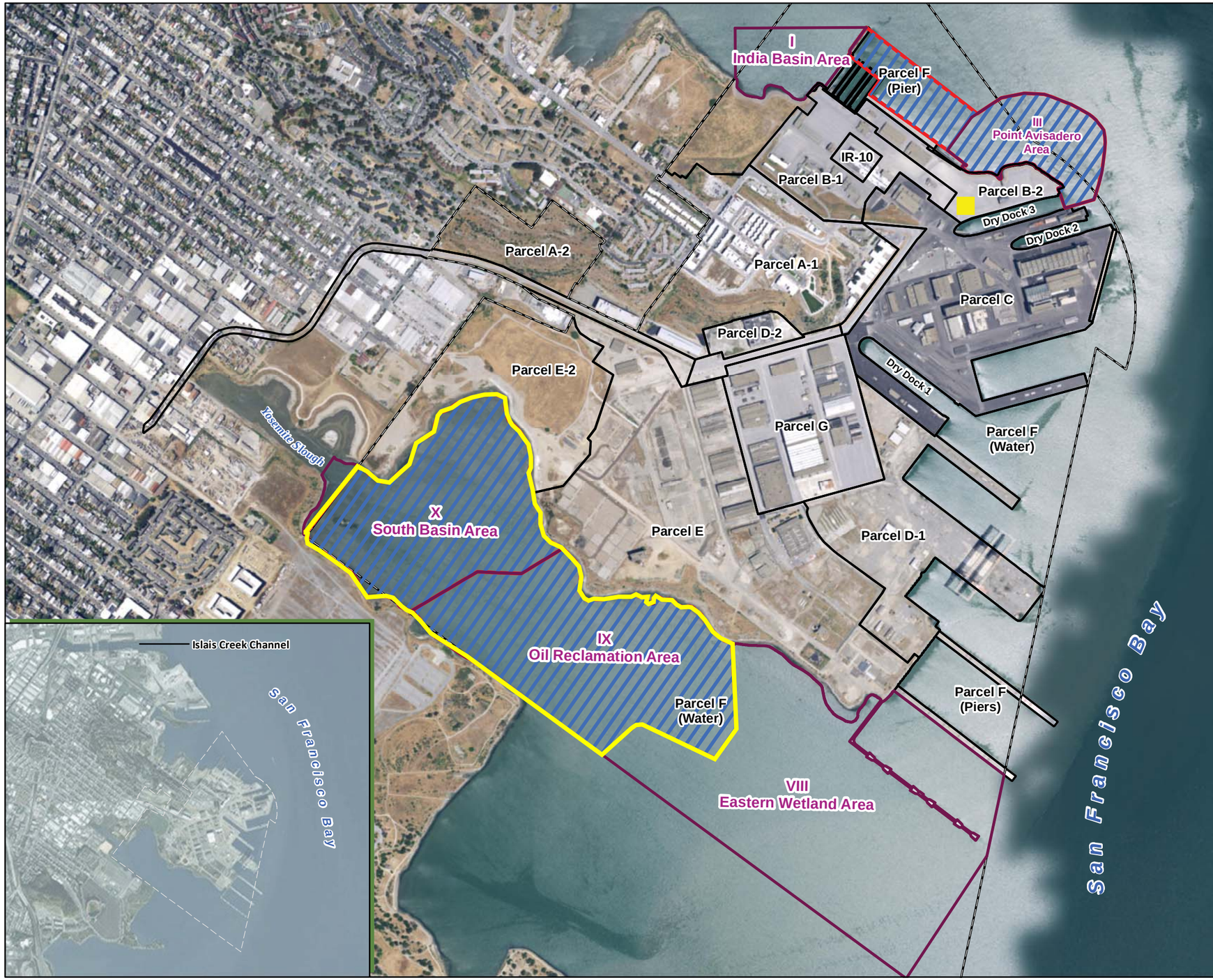
Hunters Point Naval Shipyard, San Francisco, California
U.S. Department of the Navy, BRAC PMO West

**Figure 2-3**
**South Basin**
**Conceptual Site Model**

Sediment Investigation and
Bathymetric Survey for Parcel F

schellerka G:\GIS\212737_HP_ParcelF\Task8_SAP\DraftFinal\Figure2-3_SouthBasinConceptualSiteModel.ai




## Legend

- Navy Property Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Parcel Boundary
- Approximate Bathymetric Survey Lines (100-foot spacing) and Areas
- Approximate Bathymetric Survey Lines (50-foot spacing) and Areas
- Staging Area

N

1,000 0 1,000
Feet

*1 inch = 1,000 feet*

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**U.S. Department of the Navy, BRAC PMO West**

**Figure 3-1**
**Proposed Bathymetric Survey Areas**

Sediment Investigation and
Bathymetric Survey for Parcel F

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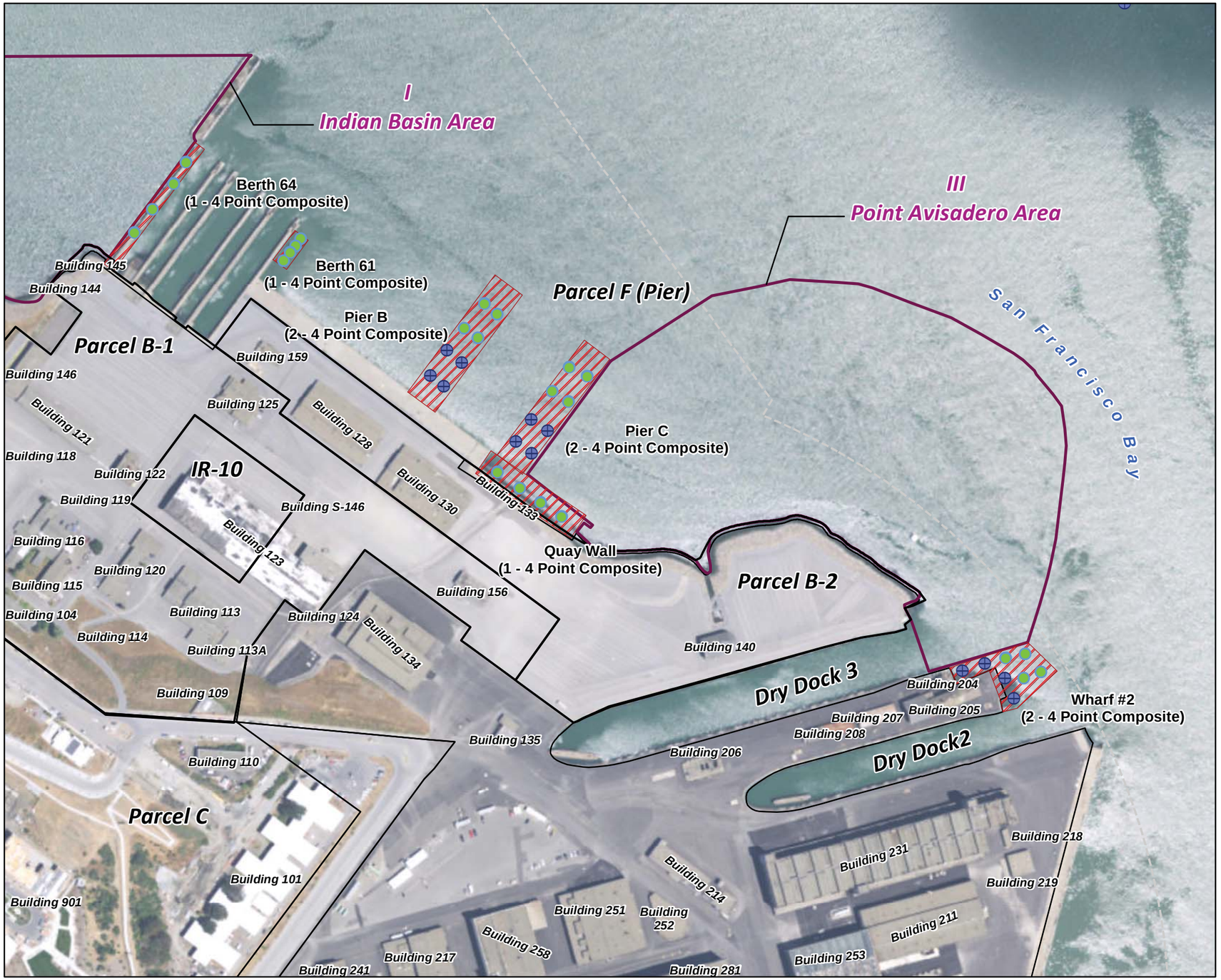


## Legend

- Navy Property Boundary
- Parcel Boundary
- Parcel F Subarea - Hunters Point Naval Shipyard
- Former Parcel B Pier Structure (Removed in 2011)
- Proposed Surface Sediment Sample Location (for Composite)
- Second Set of Four Sub-samples for Second Composite Sample for Pier B, Pier C, and Wharf #2

N

300 0 300
Feet
*1 inch = 300 feet*

ECC INSIGHT LLC
CDM Smith

**Hunters Point Naval Shipyard, San Francisco, California**
**U.S. Department of the Navy, BRAC PMO West**

**Figure 3-2**
**Proposed Sediment Sampling Locations**

Sediment Investigation and
Bathymetric Survey for Parcel F

schellerka G:\GIS\212737_HP_ParcelF\Task8_SAP\DraftFinal\Figure3-2_ProposedSedimentSamplingLocations.mxd 9/22/2017

# Attachment A1-1
# Field Forms
# (on Enclosed CD)

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## Instructional Notes for Daily Production Report Form

**WebCM Version of Report:**
To allow easier viewing, WebCM will allow the user to expand and collapse sections of the Production Report by clicking on the triangle to the left of each header. User can also expand or collapse all the sections by clicking on the Expand/Collapse All link provided at the top of the form.
All the fields in the form that are highlighted in blue with a white background are editable. Those fields that have an asterisk next to them are required to have data entered to allow the user to send the report on to the next person in the process chain. Fields that are red with a gray background are not editable and are provided for information only.
Make sure that the Contract Information is correct. If the non-editable information is not, contact the Web-CM specialist about the discrepancy.
Data continent of the applicable fields is described below.

**MS-Word Version of Report and WebCM Excel Importable Forms:**
Reports are required for each day that work is performed and shall accompany the submission of the Contractor QC Report prepared for the same day. This requirement shall commence at the beginning of the construction phase of work and continue through final completion of the Contract. Account for each calendar day throughout the life of the Contract. The reporting of work shall be identified by terminology consistent with the construction schedule. Contractor Production Reports are to be prepared, signed and dated by the Project Superintendent and shall contain the following information:

a. Date of report, report number, name of Contractor, Contract number, title and location of Contract and Superintendent present.
b. Weather conditions in the morning and in the afternoon including maximum and minimum temperatures.
c. Identify work performed by corresponding Schedule Activity No., PC#, Modification No., etc.
d. A list of Contractor and subcontractor personnel on the work site, their trades, employer, work location, description of work performed, hours worked by trade, daily total work hours on work site this date (incl. hours on continuation sheets), and total work hours from start of construction.
e. A list of job safety actions taken and safety inspections conducted. Indicate that safety requirements have been met including the results on the following:
    1) Was a job safety meeting held this date? (If YES, attach a copy of the meeting minutes.)
    2) Were there any lost time accidents this date? (If YES, attach a copy of the completed OSHA report.)
    3) Was crane/man lift/trenching/scaffold/high voltage electrical/high work/hazmat work done? (If YES, attach a statement or checklist showing inspection performed.)
    4) Was hazardous material/waste released into the environment? (If YES, attach a description of incident and proposed action.)
f. Identify Schedule Activity No. related to safety action and list safety actions taken today and safety inspections conducted.
g. Identify Schedule Activity No., Submittal # and list equipment/material received each day that is incorporated into the job.
h. Identify Schedule Activity No., Owner and list construction and plant equipment on the work site including the number of hours used.
i. Include a "remarks" section in this report which will contain pertinent information including directions received, problems encountered during construction, work progress and delays, conflicts or errors in the Contract documents, field changes, safety hazards encountered, instructions given and corrective actions taken, delays encountered and a record of visitors to the work site. For each remark given, identify the Schedule Activity No. that is associated with the remark.

**Contractor Production Report (Continuation Sheet)**
Additional space required to contain daily information on the Contractor Production Report will be placed on its Continuation Sheet(s). An unlimited number of Continuation Sheets may be added as necessary and attached to the Production Report.

# CONTRACTOR PRODUCTION REPORT

(ATTACH ADDITIONAL SHEETS IF NECESSARY)

| | | |
|---|---|---|
| | | DATE |
| CONTRACT NO | TITLE AND LOCATION | REPORT NO |
| CONTRACTOR | SUPERINTENDENT | |
| AM WEATHER | PM WEATHER | MAX TEMP (F) / MIN TEMP (F) |

## WORK PERFORMED TODAY

| Schedule Activity No. | DEFINABLE FEATURE OF WORK, WORK LOCATION AND DESCRIPTION | EMPLOYER | NUMBER | TRADE | HRS |
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**JOB SAFETY**

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| WAS A JOB SAFETY MEETING HELD THIS DATE? (If YES attach copy of the meeting minutes) | ☐ YES | ☐ NO |
| WERE THERE ANY LOST TIME ACCIDENTS THIS DATE? (If YES attach copy of completed OSHA report) | ☐ YES | ☐ NO |
| WAS CRANE/MANLIFT/TRENCHING/SCAFFOLD/HV ELEC/HIGH WORK/ HAZMAT WORK DONE? (If YES attach statement or checklist showing inspection performed.) | ☐ YES | ☐ NO |
| WAS HAZARDOUS MATERIAL/WASTE RELEASED INTO THE ENVIRONMENT? (If YES attach description of incident and proposed action.) | ☐ YES | ☐ NO |

| | |
|---|---|
| TOTAL WORK HOURS ON JOB SITE, THIS DATE, INCL CON'T SHEETS | |
| CUMULATIVE TOTAL OF WORK HOURS FROM PREVIOUS REPORT | |
| TOTAL WORK HOURS FROM START OF CONSTRUCTION | |

| Schedule Activity No. | DEFINABLE FEATURE OF WORK AND LIST SAFETY ACTIONS TAKEN TODAY/SAFETY INSPECTIONS CONDUCTED ☐ SAFETY REQUIREMENTS HAVE BEEN MET. |
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EQUIPMENT/MATERIAL RECEIVED TODAY TO BE INCORPORATED IN JOB (INDICATE SCHEDULE ACTIVITY NUMBER)

| Schedule Activity No. | Submittal # | Definable Feature of Work and Description of Equipment/Material Received |
|---|---|---|
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CONSTRUCTION AND PLANT EQUIPMENT ON JOB SITE TODAY. INDICATE HOURS USED AND SCHEDULE ACTIVITY NUMBER.

| Schedule Activity No. | Owner | Definable Feature of Work and Description of Construction Equipment Used Today (incl Make and Model) | Hours Used |
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| Schedule Activity No. | DEFINABLE FEATURE OF WORK AND REMARKS |
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CONTRACTOR/SUPERINTENDENT DATE

# CONTRACTOR PRODUCTION REPORT

(CONTINUATION SHEET)

| CONTRACT NO | TITLE AND LOCATION | DATE |
|---|---|---|
| | | REPORT NO |

## WORK PERFORMED TODAY

| Schedule Activity No. | DEFINABLE FEATURE OF WORK, WORK LOCATION AND DESCRIPTION | EMPLOYER | NUMBER | TRADE | HRS |
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| Schedule Activity No. | DEFINABLE FEATURE OF WORK AND LIST SAFETY ACTIONS TAKEN TODAY/SAFETY INSPECTIONS CONDUCTED |
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EQUIPMENT/MATERIAL RECEIVED TODAY TO BE INCORPORATED IN JOB (INDICATE SCHEDULE ACTIVITY NUMBER)

| Schedule Activity No. | Submittal # | Definable Feature of Work and Description of Equipment/Material Received |
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CONSTRUCTION AND PLANT EQUIPMENT ON JOB SITE TODAY. INDICATE HOURS USED AND SCHEDULE ACTIVITY NUMBER.

| Schedule Activity No. | Owner | Definable Feature of Work and Description of Construction Equipment Used Today (incl Make and Model) | Hours Used |
|---|---|---|---|
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| Schedule Activity No. | DEFINABLE FEATURE OF WORK AND REMARKS |
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INCLUDE ALL PERSONNEL WORK HOURS IN THE WORK PERFORMED SECTION ON THIS SHEET
INTO THE FRONT CONTRACTOR PRODUCTION REPORT

**Instructional Notes for QC Report Form**

**WebCM Version of Report:**
To allow easier viewing, WebCM will allow the user to expand and collapse sections of the QC Report by clicking on the triangle to the left of each header. User may also expand or collapse all the sections by clicking on the Expand/Collapse All link provided at the top of the form.
All the fields in the form that are highlighted in blue with a white background are editable. Those fields that have an asterisk next to them are required to have data entered to allow the user to send the report on to the next person in the process chain. Fields that are red with a gray background are not editable and are provided for information only.
Make sure that the Contract Information is correct. If the non-editable information is not, contact the Web-CM specialist about the discrepancy.
Data continent of the applicable fields is described below.

**MS-Word Version of Report and WebCM Excel Importable Forms:**
Reports are required for each day that work is performed, including design work, and for every seven consecutive calendar days of no-work and on the last day of a no-work period. Account for each calendar day throughout the life of the Contract. The reporting of work shall be identified by terminology consistent with the construction schedule. Contractor QC Reports are to be prepared, signed and dated by the Project QC Manager and shall contain the following information:

a. Date of report, report number, Contract Number, and Contract Title.
b. Indicate if Preparatory Phase work was performed today (Yes/No checkboxes).
c. If Preparatory Phase work was performed today (including on-site and off-site work), identify its Schedule Activity No. and DFOW. The Index # is a cross reference to the Preparatory Phase Checklist. An example of the Index # is: 0025-P01, where "0025" is the Contractor QC Report Number, "P" indicates Preparatory Phase, and "01" is the Preparatory Phase Checklist number(s) for this date. Each entry in this section must be accompanied with a corresponding Preparatory Phase Checklist.
d. Indicate if Initial Phase work was performed today (Yes/No checkboxes).
e. If Initial Phase work was performed today (including on-site and off-site work), identify its Schedule Activity No. and DFOW. The Index # is a cross reference to the Initial Phase Checklist. An example of the Index # is: 0025-I01, where "0025" is the Contractor QC Report Number, "I" indicates Initial Phase, and "01" is the Initial Phase Checklist number(s) for this date. Each entry in this section must be accompanied with a corresponding Initial Phase Checklist.
f. Results of the Follow-up Phase inspections held today (including on-site and off-site work), including Schedule Activity No., the location of the DFOW, Specification Sections, etc. Indicate in the report for this DFOW that the work complies with the Contract as approved in the Initial Phase, work complies with safety requirements, and that required testing has been performed and include a list of who performed the tests.
g. List the rework items identified, but not corrected by close of business; along with its associated Schedule Activity Number.
h. List the rework items corrected from the rework items list along with the corrective action taken and its associated Schedule Activity Number.
i. Include a "remarks" section in this report which will contain pertinent information including directions received, QC problem areas, deviations from the QC plan, construction deficiencies encountered, QC meetings held, acknowledgement that as-built drawings have been updated, corrective direction given by the QC Organization and corrective action taken by the Contractor. For each remark given, identify the Schedule Activity No. that is associated with the remark.
j. Contractor Quality Control Report certification, signature and date.

**Contractor Quality Control Report (Continuation Sheet)**

Additional space required to contain daily information on the Contractor QC Report will be placed on its Continuation Sheet(s). An unlimited number of Continuation Sheets may be added as necessary and attached to the Contractor QC Report.

**Reports from the QC Specialist(s)**

Reports are required for each day that work is performed in their area of responsibility. QC Specialist reports shall include the same documentation requirements as the Contractor QC Report for their area of responsibility. QC Specialist reports are to be prepared, signed and dated by the QC Specialists and shall accompany the submission of the Contractor QC Report prepared for the same day.

# CONTRACTOR QUALITY CONTROL REPORT

(ATTACH ADDITIONAL SHEETS IF NECESSARY)

DATE

REPORT NO

| PHASE | CONTRACT NO | CONTRACT TITLE |
|---|---|---|

## PREPARATORY

WAS PREPARATORY PHASE WORK PREFORMED TODAY? YES ☐ NO ☐

IF YES, FILL OUT AND ATTACH SUPPLEMENTAL PREPARATORY PHASE CHECKLIST.

| Schedule Activity No. | Definable Feature of Work | Index # |
|---|---|---|
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## INITIAL

WAS INITIAL PHASE WORK PREFORMED TODAY? YES ☐ NO ☐

IF YES, FILL OUT AND ATTACH SUPPLEMENTAL INITIAL PHASE CHECKLIST.

| Schedule Activity No. | Definable Feature of Work | Index # |
|---|---|---|
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## FOLLOW-UP

WORK COMPLIES WITH CONTRACT AS APPROVED DURING INITIAL PHASE? YES ☐ NO ☐

WORK COMPLIES WITH SAFETY REQUIREMENTS? YES ☐ NO ☐

| Schedule Activity No. | Definable Feature of Work, Description of Work, Testing Performed & By Whom, Definable Feature of Work, Specification Section, Location and List of Personnel Present |
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REWORK ITEMS IDENTIFIED TODAY (NOT CORRECTED BY CLOSE OF BUSINESS)

| Schedule Activity No. | Definable Feature of Work and Description |
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REWORK ITEMS CORRECTED TODAY (FROM REWORK ITEMS LIST)

| Schedule Activity No. | Definable Feature of Work and Description |
|---|---|
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REMARKS (Also Explain Any Follow-Up Phase Checklist Item From Above That Was Answered "NO"), Manuf. Rep On-Site, etc.

| Schedule Activity No. | Definable Feature of Work and Description |
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On behalf of the contractor, I certify that this report is complete and correct and equipment and material used and work performed during this reporting period is in compliance with the contract drawings and specifications to the best of my knowledge except as noted in this report.

AUTHORIZED QC MANAGER AT SITE DATE

## GOVERNMENT QUALITY ASSURANCE REPORT

DATE

QUALITY ASSURANCE REPRESENTATIVE'S REMARKS AND/OR EXCEPTIONS TO THE REPORT

| Schedule Activity No. | Definable Feature of Work and Description |
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GOVERNMENT QUALITY ASSURANCE MANAGER DATE

# CONTRACTOR QUALITY CONTROL REPORT

(CONTINUATION SHEET)
(ATTACH ADDITIONAL SHEETS IF NECESSARY)

DATE

REPORT NO.

| PHASE | CONTRACT NO | CONTRACT TITLE |
|---|---|---|

FOLLOW-UP

WORK COMPLIES WITH CONTRACT AS APPROVED DURING INITIAL PHASE? YES ☐ NO ☐

WORK COMPLIES WITH SAFETY REQUIREMENTS? YES ☐ NO ☐

| Schedule Activity No. | Definable Feature of Work, Description of Work, Testing Performed & By Whom, Definable Feature of Work, Specification Section, Location and List of Personnel Present |
|---|---|
| | |

REMARKS (Also Explain Any Checklist Item From Above That Was Answered "NO"), Manuf. Rep. On-Site, etc.

| Schedule Activity No. | Definable Feature of Work and Description |
|---|---|
| | |

# CHAIN-OF-CUSTODY RECORD

Number

2749 Saturn St.
Brea, CA 92821

| Project Name | Project No. |
|---|---|
| Project Location | |
| Project Contact | Contact Phone No. |

**Analyses**

Laboratory Name

**Lab Batch ID (SDG)**

**Project Information Section**
**For Project Personnel Only**
**Do Not Submit to Laboratory**

| | Sample ID | Date | Time | Number of Containers | QC Level | Type | TAT | | | | | | | | | | | Comments | Sample Location | QC | Depth Start | Depth End |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | | | | | | | | | | | | | | | | | | | | |
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| 11 | | | | | | | | | | | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | | | | | | | | | | | |

Samples Collected By:

Courier or Airbill Number:

Sampler Notes:

| Relinquished By | | | | Received BY | | | |
|---|---|---|---|---|---|---|---|
| Signature | Date | Time | Company | Signature | Date | Time | Company |
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Instructions and Notes for Laboratory:

QC: N=Normal, FD=Field Duplicate, MS=MS/MSD, TB=Trip Blank, EB=Equipment Rinse, SB=Source Blank

# DAILY DISRUPTION REPORT

Project Name: _____

G.C. Project No: _____

Owner Project No: _____

Foreman: _____

| Date | Description | | | Description | | | Description | | | Description | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sub Project No. | | | | | | | | | | | | | | | | |
| Day S M T W Th F S | | | | | | | | | | | | | | | | |
| Weather | | | | | | | | | | | | | | | | |
| Activity #: | | | | | | | | | | | | | SUMMARY | | | |
| | REG 1.0 | OVT 1.5 | DBL 2.0 | REG 1.0 | OVT 1.5 | DBL 2.0 | REG 1.0 | OVT 1.5 | DBL 2.0 | REG 1.0 | OVT 1.5 | DBL 2.0 | REG 1.0 | OVT 1.5 | DBL 2.0 | TOTAL |
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# DISRUPTION TRENDS

Page No. _____

G.C. Project No. _____

Project Name: _____ Sub Project No. _____

Project Manager: _____ Owner Project No. _____

| (A)<br>Date | (B)<br>Description | (C)<br>Hours Lost | (D)<br>Total Hours Per Day | (C/D)<br>Lost Productivity |
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# FIELD AUTHORIZATION FORM

Date: FA No.

To:

Sub Project No.

From: G.C. Project No.

Project Name: Owner Project No.

You are authorized to perform the following:

Signature: _____ Title: _____

This work is to be completed:

_____ On a time and material basis

_____ Pursuant to change order quotation # _____

_____ Pursuant to architectural bulletin quotation # _____

_____ Pursuant to modification quotation # _____

_____ Pursuant to a price to be negotiated within the next 7 days

_____ Other _____

# FIELD CHANGE REQUEST

Date: _____

Project Name: _____

Project Number: _____

Location: _____

Revision Number: _____

---

**Reference data**

Specification section No: _____ Page No: _____ Paragraph No: _____

Drawing No: _____ Details: _____

Sketch No: _____ Dated: _____ Other: _____

---

**Description**

1) Detailed identification of the problem: _____

2) Detailed solution proposed: _____

3) Is this a general problem or an isolated case? _____

4) See sketches on reverse side. ☐ Yes ☐ No

5) Affected contractor(s): _____

6) Cost and schedule impact: _____

Report completed by: _____ Title: _____

# FIELD MONITORING

Project Number: _____ Project Location: _____

Delivery Order No: _____ Tank No: _____

Site Location: _____ Date: _____

Monitoring Equipment Used: _____

Type of Activity: _____

| Time | PID (ppm) | LEL/$O_2$ Meter (%) | Remarks |
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# NONCONFORMANCE REPORT

Contract No. _____TO No. _____

| NCR Number: | Project Name and Number: | Date: | Page<br>1 of |
|---|---|---|---|

Nonconformance Description (include specific requirement violated):

Identified by: _____ Date: _____

Root Cause of Nonconforming Action:

Corrective Action(s) to be Taken (include date when action(s) will be complete):

To be Performed by: _____Date: _____

Action(s) to be Taken to Preclude Recurrence:

To be Performed by: _____Date: _____

Acceptance by:

Project Manager: _____Date: _____

CQC Manager: _____ Date: _____

| Corrective Action(s) Completed by and Date: | Verification Completed by and Date: |
|---|---|

# PHOTOGRAPHIC INVENTORY

Project Number: _____ Date: _____ Photographed by: _____

| Photo No. | Roll No. | Site No. | Directional View | Site Description |
|---|---|---|---|---|
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# TAILGATE SAFETY MEETING

Project No: _____ Project Name/Location: _____

Date: _____ Time: _____

Client: _____ Client Phone: _____

Type of Work: _____

Chemicals Used: _____

---

Protective Clothing/Equipment: _____

Chemical Hazards: _____

Physical Hazards: _____

Emergency Procedure: _____

Hospital: _____

Hospital Address: _____

Special Equipment: _____

Other: _____

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**ATTENDEES**

| Name (print) | Signature |
|---|---|
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Meeting Conducted By:

_____ _____

# Attachment A1-2
# Laboratory SOPs
# (on Enclosed CD)

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# CERTIFICATE OF ACCREDITATION

**ANSI-ASQ National Accreditation Board**
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

## Enthalpy Analytical, LLC
## 2323 Fifth Street
## Berkeley CA 94710

has been assessed by ANAB
and meets the requirements of

## ISO/IEC 17025:2005 and DoD-ELAP

while demonstrating technical competence in the field of

## TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of tests to which this accreditation applies.

L-2442
Certificate Number

ANAB Approval

Certificate Valid: 06/29/2017-06/29/2020
Issued: 06/29/2017

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated January 2009).

# Scope of Accreditation
# For
# Enthalpy Analytical, LLC

2323 Fifth Street
Berkeley CA 94710
Teresa Morrison
510-486-0900

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **Enthalpy Analytical, LLC** to perform the following tests:

Accreditation granted through: **June 29, 2020**

**Testing - Environmental**

| **Non-Potable Water** | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-FID | EPA 8015B/ 8015D | Gasoline Range Organics (GRO, TPH-G) |
| GC-FID | EPA 8015B/ 8015D | Diesel Range Organics (DRO, TPH-D) |
| GC-FID | RSK-175 | Acetylene |
| GC-FID | RSK-175 | Ethane |
| GC-FID | RSK-175 | Ethene |
| GC-FID | RSK-175 | Methane |
| GC-PID | EPA 8021B | MTBE |
| GC-PID | EPA 8021B | Benzene |
| GC-PID | EPA 8021B | Toluene |
| GC-PID | EPA 8021B | Ethylbenzene |
| GC-PID | EPA 8021B | m,p-Xylenes |
| GC-PID | EPA 8021B | o-Xylene |
| GC-ECD | EPA 8081A/ 8081B | Aldrin |
| GC-ECD | EPA 8081A/ 8081B | a-BHC |
| GC-ECD | EPA 8081A/ 8081B | b-BHC |
| GC-ECD | EPA 8081A/ 8081B | d-BHC |
| GC-ECD | EPA 8081A/ 8081B | g-BHC |
| GC-ECD | EPA 8081A/ 8081B | Chlordane (Technical) |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-ECD | EPA 8081A/ 8081B | a-Chlordane |
| GC-ECD | EPA 8081A/ 8081B | g-Chlordane |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDD |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDE |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDT |
| GC-ECD | EPA 8081A/ 8081B | Dieldrin |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan I |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan II |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan Sulfate |
| GC-ECD | EPA 8081A/ 8081B | Endrin |
| GC-ECD | EPA 8081A/ 8081B | Endrin Aldehyde |
| GC-ECD | EPA 8081A/ 8081B | Endrin Ketone |
| GC-ECD | EPA 8081A/ 8081B | Heptachlor |
| GC-ECD | EPA 8081A/ 8081B | Heptachlor Epoxide |
| GC-ECD | EPA 8081A/ 8081B | Methoxychlor |
| GC-ECD | EPA 8081A/ 8081B | Toxaphene |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1016 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1221 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1232 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1242 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1248 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1254 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1260 |
| GC-MS | EPA 8260B/ 8260C | 1,1,1,2-Tetrachloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,1-Trichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,2,2-Tetrachloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,2-Trichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | 1,2,3-Trichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2,3-Trichloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,2,4-Trichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2,4-Trimethylbenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dibromo-3-Chloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dibromoethane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichloropropane |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8260B/ 8260C | 1,3,5-Trimethylbenzene |
| GC-MS | EPA 8260B/ 8260C | 1,3-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,3-Dichloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,4-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 2,2-Dichloropropane |
| GC-MS | EPA 8260B/ 8260C | 2-Butanone |
| GC-MS | EPA 8260B/ 8260C | 2-Chlorotoluene |
| GC-MS | EPA 8260B/ 8260C | 2-Hexanone |
| GC-MS | EPA 8260B/ 8260C | 4-Chlorotoluene |
| GC-MS | EPA 8260B/ 8260C | 4-Methyl-2-Pentanone |
| GC-MS | EPA 8260B/ 8260C | Acetone |
| GC-MS | EPA 8260B/ 8260C | Benzene |
| GC-MS | EPA 8260B/ 8260C | Bromobenzene |
| GC-MS | EPA 8260B/ 8260C | Bromochloromethane |
| GC-MS | EPA 8260B/ 8260C | Bromodichloromethane |
| GC-MS | EPA 8260B/ 8260C | Bromoform |
| GC-MS | EPA 8260B/ 8260C | Bromomethane |
| GC-MS | EPA 8260B/ 8260C | Carbon Disulfide |
| GC-MS | EPA 8260B/ 8260C | Carbon Tetrachloride |
| GC-MS | EPA 8260B/ 8260C | Chlorobenzene |
| GC-MS | EPA 8260B/ 8260C | Chloroethane |
| GC-MS | EPA 8260B/ 8260C | Chloroform |
| GC-MS | EPA 8260B/ 8260C | Chloromethane |
| GC-MS | EPA 8260B/ 8260C | cis-1,2-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | cis-1,3-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | Dibromochloromethane |
| GC-MS | EPA 8260B/ 8260C | Dibromomethane |
| GC-MS | EPA 8260B/ 8260C | Ethylbenzene |
| GC-MS | EPA 8260B/ 8260C | Ethyl tert-Butyl Ether (ETBE) |
| GC-MS | EPA 8260B/ 8260C | Freon 113 |
| GC-MS | EPA 8260B/ 8260C | Freon 12 |
| GC-MS | EPA 8260B/ 8260C | Hexachlorobutadiene |
| GC-MS | EPA 8260B/ 8260C | Isopropylbenzene |
| GC-MS | EPA 8260B/ 8260C | Isopropyl Ether (DIPE) |
| GC-MS | EPA 8260B/ 8260C | m,p-Xylenes |
| GC-MS | EPA 8260B/ 8260C | Methylene Chloride |
| GC-MS | EPA 8260B/ 8260C | Methyl tert-Amyl Ether (TAME) |
| GC-MS | EPA 8260B/ 8260C | Methyl tert-Butyl Ether (MTBE) |
| GC-MS | EPA 8260B/ 8260C | Naphthalene |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8260B/ 8260C | n-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | o-Xylene |
| GC-MS | EPA 8260B/ 8260C | para-Isopropyl Toluene |
| GC-MS | EPA 8260B/ 8260C | Propylbenzene |
| GC-MS | EPA 8260B/ 8260C | sec-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | Styrene |
| GC-MS | EPA 8260B/ 8260C | tert-Butyl Alcohol (TBA) |
| GC-MS | EPA 8260B/ 8260C | tert-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | Tetrachloroethene |
| GC-MS | EPA 8260B/ 8260C | Toluene |
| GC-MS | EPA 8260B/ 8260C | trans-1,2-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | trans-1,3-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | Trichloroethene |
| GC-MS | EPA 8260B/ 8260C | Trichlorofluoromethane |
| GC-MS | EPA 8260B/ 8260C | Vinyl Acetate |
| GC-MS | EPA 8260B/ 8260C | Vinyl Chloride |
| GC-MS | EPA 8270C/ 8270D | 1,2,4-Trichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,2-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,3-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,4-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 2,4,5-Trichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4,6-Trichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dimethylphenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dinitrophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dinitrotoluene |
| GC-MS | EPA 8270C/ 8270D | 2,6-Dinitrotoluene |
| GC-MS | EPA 8270C/ 8270D | 2-Chloronaphthalene |
| GC-MS | EPA 8270C/ 8270D | 2-Chlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2-Methylnaphthalene |
| GC-MS | EPA 8270C/ 8270D | 2-Methylphenol |
| GC-MS | EPA 8270C/ 8270D | 2-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 2-Nitrophenol |
| GC-MS | EPA 8270C/ 8270D | 3,3'-Dichlorobenzidine |
| GC-MS | EPA 8270C/ 8270D | 3-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 4,6-Dinitro-2-methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Bromophenyl-phenylether |
| GC-MS | EPA 8270C/ 8270D | 4-Chloro-3-methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Chloroaniline |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8270C/ 8270D | 4-Chlorophenyl-phenylether |
| GC-MS | EPA 8270C/ 8270D | 4-Methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 4-Nitrophenol |
| GC-MS | EPA 8270C/ 8270D | Acenaphthene |
| GC-MS | EPA 8270C/ 8270D | Acenaphthylene |
| GC-MS | EPA 8270C/ 8270D | Anthracene |
| GC-MS | EPA 8270C/ 8270D | Azobenzene |
| GC-MS | EPA 8270C/ 8270D | Benzo(a)anthracene |
| GC-MS | EPA 8270C/ 8270D | Benzo(a)pyrene |
| GC-MS | EPA 8270C/ 8270D | Benzo(b)fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Benzo(g,h,i)perylene |
| GC-MS | EPA 8270C/ 8270D | Benzo(k)fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Benzoic acid |
| GC-MS | EPA 8270C/ 8270D | Benzyl alcohol |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroethoxy)methane |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroethyl)ether |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroisopropyl) ether |
| GC-MS | EPA 8270C/ 8270D | bis(2-Ethylhexyl)phthalate |
| GC-MS | EPA 8270C/ 8270D | Butylbenzylphthalate |
| GC-MS | EPA 8270C/ 8270D | Chrysene |
| GC-MS | EPA 8270C/ 8270D | Dibenz(a,h)anthracene |
| GC-MS | EPA 8270C/ 8270D | Dibenzofuran |
| GC-MS | EPA 8270C/ 8270D | Diethylphthalate |
| GC-MS | EPA 8270C/ 8270D | Dimethylphthalate |
| GC-MS | EPA 8270C/ 8270D | Di-n-butylphthalate |
| GC-MS | EPA 8270C/ 8270D | Di-n-octylphthalate |
| GC-MS | EPA 8270C/ 8270D | Fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Fluorene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorobenzene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorobutadiene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorocyclopentadiene |
| GC-MS | EPA 8270C/ 8270D | Hexachloroethane |
| GC-MS | EPA 8270C/ 8270D | Indeno(1,2,3-cd)pyrene |
| GC-MS | EPA 8270C/ 8270D | Isophorone |
| GC-MS | EPA 8270C/ 8270D | Naphthalene |
| GC-MS | EPA 8270C/ 8270D | Nitrobenzene |
| GC-MS | EPA 8270C/ 8270D | N-Nitrosodimethylamine |
| GC-MS | EPA 8270C/ 8270D | N-Nitroso-di-n-propylamine |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8270C/ 8270D | N-Nitrosodiphenylamine |
| GC-MS | EPA 8270C/ 8270D | Pentachlorophenol |
| GC-MS | EPA 8270C/ 8270D | Phenanthrene |
| GC-MS | EPA 8270C/ 8270D | Phenol |
| GC-MS | EPA 8270C/ 8270D | Pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 1,4-Dioxane |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Acenaphthylene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Acenaphthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(a)anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(a)pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(b)fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(g,h,i)perylene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(k)fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Chrysene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Dibenz(a,h)anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Fluorene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Indeno(1,2,3-cd)pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 1-Methylnaphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 2-Methylnaphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Naphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Phenanthrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Pyrene |
| HPLC-UV | EPA 8330/ 8330A | 2-Amino-4,6-dinitrotoluene |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| HPLC-UV | EPA 8330/ 8330A | 4-Amino-2,6-dinitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | 3,5-Dinitroaniline |
| HPLC-UV | EPA 8330/ 8330A | 1,3-Dinitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | 2,4-Dinitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | 2,6-Dinitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | HMX |
| HPLC-UV | EPA 8330/ 8330A | RDX |
| HPLC-UV | EPA 8330/ 8330A | Nitroglycerine |
| HPLC-UV | EPA 8330/ 8330A | Nitrobenzene |
| HPLC-UV | EPA 8330/ 8330A | 2-Nitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | 3-Nitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | 4-Nitrotoluene |
| HPLC-UV | EPA 8330/ 8330A | Pentaerythritol (PETN) |
| HPLC-UV | EPA 8330/ 8330A | RDX |
| HPLC-UV | EPA 8330/ 8330A | Tetryl |
| HPLC-UV | EPA 8330/ 8330A | 1,3,5-Trinitrobenzene |
| HPLC-UV | EPA 8330/ 8330A | 2,4,6-Trinitrotoluene |
| ICP-AES | EPA 6010B/ 6010C | Aluminum |
| ICP-AES | EPA 6010B/ 6010C | Antimony |
| ICP-AES | EPA 6010B/ 6010C | Arsenic |
| ICP-AES | EPA 6010B/ 6010C | Barium |
| ICP-AES | EPA 6010B/ 6010C | Beryllium |
| ICP-AES | EPA 6010B/ 6010C | Cadmium |
| ICP-AES | EPA 6010B/ 6010C | Calcium |
| ICP-AES | EPA 6010B/ 6010C | Chromium |
| ICP-AES | EPA 6010B/ 6010C | Cobalt |
| ICP-AES | EPA 6010B/ 6010C | Copper |
| ICP-AES | EPA 6010B/ 6010C | Iron |
| ICP-AES | EPA 6010B/ 6010C | Lead |
| ICP-AES | EPA 6010B/ 6010C | Magnesium |
| ICP-AES | EPA 6010B/ 6010C | Manganese |
| ICP-AES | EPA 6010B/ 6010C | Molybdenum |
| ICP-AES | EPA 6010B/ 6010C | Nickel |
| ICP-AES | EPA 6010B/ 6010C | Potassium |
| ICP-AES | EPA 6010B/ 6010C | Selenium |
| ICP-AES | EPA 6010B/ 6010C | Silver |
| ICP-AES | EPA 6010B/ 6010C | Sodium |
| ICP-AES | EPA 6010B/ 6010C | Thallium |
| ICP-AES | EPA 6010B/ 6010C | Vanadium |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| ICP-AES | EPA 6010B/ 6010C | Zinc |
| ICP-MS | EPA 6020/ 6020A | Aluminum |
| ICP-MS | EPA 6020/ 6020A | Antimony |
| ICP-MS | EPA 6020/ 6020A | Arsenic |
| ICP-MS | EPA 6020/ 6020A | Barium |
| ICP-MS | EPA 6020/ 6020A | Beryllium |
| ICP-MS | EPA 6020/ 6020A | Cadmium |
| ICP-MS | EPA 6020/ 6020A | Calcium |
| ICP-MS | EPA 6020/ 6020A | Chromium |
| ICP-MS | EPA 6020/ 6020A | Cobalt |
| ICP-MS | EPA 6020/ 6020A | Copper |
| ICP-MS | EPA 6020/ 6020A | Iron |
| ICP-MS | EPA 6020/ 6020A | Lead |
| ICP-MS | EPA 6020/ 6020A | Magnesium |
| ICP-MS | EPA 6020/ 6020A | Manganese |
| ICP-MS | EPA 6020/ 6020A | Molybdenum |
| ICP-MS | EPA 6020/ 6020A | Nickel |
| ICP-MS | EPA 6020/ 6020A | Potassium |
| ICP-MS | EPA 6020/ 6020A | Selenium |
| ICP-MS | EPA 6020/ 6020A | Silver |
| ICP-MS | EPA 6020/ 6020A | Sodium |
| ICP-MS | EPA 6020/ 6020A | Thallium |
| ICP-MS | EPA 6020/ 6020A | Vanadium |
| ICP-MS | EPA 6020/ 6020A | Zinc |
| CVAA | EPA 7470A | Mercury |
| Ion Chromatography | EPA 300.0 / 9056 | Bromide |
| Ion Chromatography | EPA 300.0 / 9056 | Chloride |
| Ion Chromatography | EPA 300.0 / 9056 | Fluoride |
| Ion Chromatography | EPA 300.0 / 9056 | Nitrate-N |
| Ion Chromatography | EPA 300.0 / 9056 | Nitrite-N |
| Ion Chromatography | EPA 300.0 / 9056 | Sulfate |
| Ion Chromatography | EPA 314 | Perchlorate |
| Ion Chromatography | EPA 7199 | Hexavalent Chromium |
| Ion Selective Electrode | SM 4500-NH3 D | Ammonia |
| Ion Selective Electrode | SM 5210B | Biochemical Oxygen Demand (BOD) |
| Ion Selective Electrode | EPA 9040B<br>SM 4500-H +B | pH |
| Ion Selective Electrode | SM 2510B | Specific Conductance |
| UV-VIS Spectrometer | SM 5220D | Chemical Oxygen Demand (COD) |

| Non-Potable Water | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| UV-VIS Spectrometer | SM 4500-CN E<br>EPA 9010B/ 9014 | Cyanide |
| UV-VIS Spectrometer | SM 4500-CN E<br>EPA 9010B/ 9014 | Cyanide, Amenable |
| UV-VIS Spectrometer | SM 3500-Fe B | Ferrous Iron |
| UV-VIS Spectrometer | EPA 7196A | Hexavalent Chromium |
| UV-VIS Spectrometer | SM 4500-P E | Total Phosphate-P |
| UV-VIS Spectrometer | SM 4500-S2 D | Sulfide |
| UV-VIS Spectrometer | SM 5310C | Total Organic Carbon (TOC) |
| Titration | SM 2320B | Alkalinity |
| Titration | SM 4500-NH3 C | Total Kjeldahl Nitrogen (TKN) |
| Gravimetric | SM 2540C | Total Dissolved Solids (TDS) |
| Gravimetric | SM 2540D | Total Suspended Solids (TSS) |
| Other | EPA 1010<br>ASTM D93 | Flash Point |
| **Preparation** | **Method** | **Analyte** |
| Purge & Trap | EPA 5030B/ 5030C | Preparation for Volatiles |
| Extraction | EPA 3520C | Continuous Liquid-Liquid Extraction for Semivolatile Organics (DRO, BNA, PCB, Pesticides, SIM) |
| Extraction | EPA 3535 | Solid Phase Extraction (for Nitroaromatics & Nitramines) |
| Digestion | EPA 200.8 | Water Digestion for ICP-MS Metals |
| Digestion | EPA 3010A | Water Digestion for ICP Metals |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-FID | EPA 8015B/ 8015D | Gasoline Range Organics (GRO, TPH-G) |
| GC-FID | EPA 8015B/ 8015D | Diesel Range Organics (DRO, TPH-D) |
| GC-PID | EPA 8021B | MTBE |
| GC-PID | EPA 8021B | Benzene |
| GC-PID | EPA 8021B | Toluene |
| GC-PID | EPA 8021B | Ethylbenzene |
| GC-PID | EPA 8021B | m,p-Xylenes |
| GC-PID | EPA 8021B | o-Xylene |
| GC-ECD | EPA 8081A/ 8081B | Aldrin |
| GC-ECD | EPA 8081A/ 8081B | a-BHC |
| GC-ECD | EPA 8081A/ 8081B | b-BHC |
| GC-ECD | EPA 8081A/ 8081B | d-BHC |
| GC-ECD | EPA 8081A/ 8081B | g-BHC |
| GC-ECD | EPA 8081A/ 8081B | Chlordane (Technical) |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-ECD | EPA 8081A/ 8081B | a-Chlordane |
| GC-ECD | EPA 8081A/ 8081B | g-Chlordane |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDD |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDE |
| GC-ECD | EPA 8081A/ 8081B | 4,4'-DDT |
| GC-ECD | EPA 8081A/ 8081B | Dieldrin |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan I |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan II |
| GC-ECD | EPA 8081A/ 8081B | Endosulfan Sulfate |
| GC-ECD | EPA 8081A/ 8081B | Endrin |
| GC-ECD | EPA 8081A/ 8081B | Endrin Aldehyde |
| GC-ECD | EPA 8081A/ 8081B | Endrin Ketone |
| GC-ECD | EPA 8081A/ 8081B | Heptachlor |
| GC-ECD | EPA 8081A/ 8081B | Heptachlor Epoxide |
| GC-ECD | EPA 8081A/ 8081B | Methoxychlor |
| GC-ECD | EPA 8081A/ 8081B | Toxaphene |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1016 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1221 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1232 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1242 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1248 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1254 |
| GC-ECD | EPA 8082/ 8082A | Arochlor 1260 |
| GC-MS | EPA 8260B/ 8260C | 1,1,1,2-Tetrachloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,1-Trichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,2,2-Tetrachloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1,2-Trichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | 1,1-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | 1,2,3-Trichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2,3-Trichloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,2,4-Trichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2,4-Trimethylbenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dibromo-3-Chloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dibromoethane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichloroethane |
| GC-MS | EPA 8260B/ 8260C | 1,2-Dichloropropane |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8260B/ 8260C | 1,3,5-Trimethylbenzene |
| GC-MS | EPA 8260B/ 8260C | 1,3-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 1,3-Dichloropropane |
| GC-MS | EPA 8260B/ 8260C | 1,4-Dichlorobenzene |
| GC-MS | EPA 8260B/ 8260C | 2,2-Dichloropropane |
| GC-MS | EPA 8260B/ 8260C | 2-Butanone |
| GC-MS | EPA 8260B/ 8260C | 2-Chlorotoluene |
| GC-MS | EPA 8260B/ 8260C | 2-Hexanone |
| GC-MS | EPA 8260B/ 8260C | 4-Chlorotoluene |
| GC-MS | EPA 8260B/ 8260C | 4-Methyl-2-Pentanone |
| GC-MS | EPA 8260B/ 8260C | Acetone |
| GC-MS | EPA 8260B/ 8260C | Benzene |
| GC-MS | EPA 8260B/ 8260C | Bromobenzene |
| GC-MS | EPA 8260B/ 8260C | Bromochloromethane |
| GC-MS | EPA 8260B/ 8260C | Bromodichloromethane |
| GC-MS | EPA 8260B/ 8260C | Bromoform |
| GC-MS | EPA 8260B/ 8260C | Bromomethane |
| GC-MS | EPA 8260B/ 8260C | Carbon Disulfide |
| GC-MS | EPA 8260B/ 8260C | Carbon Tetrachloride |
| GC-MS | EPA 8260B/ 8260C | Chlorobenzene |
| GC-MS | EPA 8260B/ 8260C | Chloroethane |
| GC-MS | EPA 8260B/ 8260C | Chloroform |
| GC-MS | EPA 8260B/ 8260C | Chloromethane |
| GC-MS | EPA 8260B/ 8260C | cis-1,2-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | cis-1,3-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | Dibromochloromethane |
| GC-MS | EPA 8260B/ 8260C | Dibromomethane |
| GC-MS | EPA 8260B/ 8260C | Ethylbenzene |
| GC-MS | EPA 8260B/ 8260C | Ethyl tert-Butyl Ether (ETBE) |
| GC-MS | EPA 8260B/ 8260C | Freon 113 |
| GC-MS | EPA 8260B/ 8260C | Freon 12 |
| GC-MS | EPA 8260B/ 8260C | Hexachlorobutadiene |
| GC-MS | EPA 8260B/ 8260C | Isopropylbenzene |
| GC-MS | EPA 8260B/ 8260C | Isopropyl Ether (DIPE) |
| GC-MS | EPA 8260B/ 8260C | m,p-Xylenes |
| GC-MS | EPA 8260B/ 8260C | Methylene Chloride |
| GC-MS | EPA 8260B/ 8260C | Methyl tert-Amyl Ether (TAME) |
| GC-MS | EPA 8260B/ 8260C | Methyl tert-Butyl Ether (MTBE) |
| GC-MS | EPA 8260B/ 8260C | Naphthalene |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8260B/ 8260C | n-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | o-Xylene |
| GC-MS | EPA 8260B/ 8260C | para-Isopropyl Toluene |
| GC-MS | EPA 8260B/ 8260C | Propylbenzene |
| GC-MS | EPA 8260B/ 8260C | sec-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | Styrene |
| GC-MS | EPA 8260B/ 8260C | tert-Butyl Alcohol (TBA) |
| GC-MS | EPA 8260B/ 8260C | tert-Butylbenzene |
| GC-MS | EPA 8260B/ 8260C | Tetrachloroethene |
| GC-MS | EPA 8260B/ 8260C | Toluene |
| GC-MS | EPA 8260B/ 8260C | trans-1,2-Dichloroethene |
| GC-MS | EPA 8260B/ 8260C | trans-1,3-Dichloropropene |
| GC-MS | EPA 8260B/ 8260C | Trichloroethene |
| GC-MS | EPA 8260B/ 8260C | Trichlorofluoromethane |
| GC-MS | EPA 8260B/ 8260C | Vinyl Acetate |
| GC-MS | EPA 8260B/ 8260C | Vinyl Chloride |
| GC-MS | EPA 8270C/ 8270D | 1,2,4-Trichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,2-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,3-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 1,4-Dichlorobenzene |
| GC-MS | EPA 8270C/ 8270D | 2,4,5-Trichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4,6-Trichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dichlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dimethylphenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dinitrophenol |
| GC-MS | EPA 8270C/ 8270D | 2,4-Dinitrotoluene |
| GC-MS | EPA 8270C/ 8270D | 2,6-Dinitrotoluene |
| GC-MS | EPA 8270C/ 8270D | 2-Chloronaphthalene |
| GC-MS | EPA 8270C/ 8270D | 2-Chlorophenol |
| GC-MS | EPA 8270C/ 8270D | 2-Methylnaphthalene |
| GC-MS | EPA 8270C/ 8270D | 2-Methylphenol |
| GC-MS | EPA 8270C/ 8270D | 2-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 2-Nitrophenol |
| GC-MS | EPA 8270C/ 8270D | 3,3'-Dichlorobenzidine |
| GC-MS | EPA 8270C/ 8270D | 3-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 4,6-Dinitro-2-methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Bromophenyl-phenylether |
| GC-MS | EPA 8270C/ 8270D | 4-Chloro-3-methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Chloroaniline |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8270C/ 8270D | 4-Chlorophenyl-phenylether |
| GC-MS | EPA 8270C/ 8270D | 4-Methylphenol |
| GC-MS | EPA 8270C/ 8270D | 4-Nitroaniline |
| GC-MS | EPA 8270C/ 8270D | 4-Nitrophenol |
| GC-MS | EPA 8270C/ 8270D | Acenaphthene |
| GC-MS | EPA 8270C/ 8270D | Acenaphthylene |
| GC-MS | EPA 8270C/ 8270D | Anthracene |
| GC-MS | EPA 8270C/ 8270D | Azobenzene |
| GC-MS | EPA 8270C/ 8270D | Benzo(a)anthracene |
| GC-MS | EPA 8270C/ 8270D | Benzo(a)pyrene |
| GC-MS | EPA 8270C/ 8270D | Benzo(b)fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Benzo(g,h,i)perylene |
| GC-MS | EPA 8270C/ 8270D | Benzo(k)fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Benzoic acid |
| GC-MS | EPA 8270C/ 8270D | Benzyl alcohol |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroethoxy)methane |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroethyl)ether |
| GC-MS | EPA 8270C/ 8270D | bis(2-Chloroisopropyl) ether |
| GC-MS | EPA 8270C/ 8270D | bis(2-Ethylhexyl)phthalate |
| GC-MS | EPA 8270C/ 8270D | Butylbenzylphthalate |
| GC-MS | EPA 8270C/ 8270D | Chrysene |
| GC-MS | EPA 8270C/ 8270D | Dibenz(a,h)anthracene |
| GC-MS | EPA 8270C/ 8270D | Dibenzofuran |
| GC-MS | EPA 8270C/ 8270D | Diethylphthalate |
| GC-MS | EPA 8270C/ 8270D | Dimethylphthalate |
| GC-MS | EPA 8270C/ 8270D | Di-n-butylphthalate |
| GC-MS | EPA 8270C/ 8270D | Di-n-octylphthalate |
| GC-MS | EPA 8270C/ 8270D | Fluoranthene |
| GC-MS | EPA 8270C/ 8270D | Fluorene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorobenzene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorobutadiene |
| GC-MS | EPA 8270C/ 8270D | Hexachlorocyclopentadiene |
| GC-MS | EPA 8270C/ 8270D | Hexachloroethane |
| GC-MS | EPA 8270C/ 8270D | Indeno(1,2,3-cd)pyrene |
| GC-MS | EPA 8270C/ 8270D | Isophorone |
| GC-MS | EPA 8270C/ 8270D | Naphthalene |
| GC-MS | EPA 8270C/ 8270D | Nitrobenzene |
| GC-MS | EPA 8270C/ 8270D | N-Nitrosodimethylamine |
| GC-MS | EPA 8270C/ 8270D | N-Nitroso-di-n-propylamine |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | EPA 8270C/ 8270D | N-Nitrosodiphenylamine |
| GC-MS | EPA 8270C/ 8270D | Pentachlorophenol |
| GC-MS | EPA 8270C/ 8270D | Phenanthrene |
| GC-MS | EPA 8270C/ 8270D | Phenol |
| GC-MS | EPA 8270C/ 8270D | Pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 1,4-Dioxane |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Acenaphthylene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Acenaphthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(a)anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(a)pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(b)fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(g,h,i)perylene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Benzo(k)fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Chrysene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Dibenz(a,h)anthracene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Fluoranthene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Fluorene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Indeno(1,2,3-cd)pyrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 1-Methylnaphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | 2-Methylnaphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Naphthalene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Phenanthrene |
| GC-MS | EPA 8270C-SIM<br>EPA 8270D-SIM | Pyrene |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 2-Amino-4,6-dinitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 4-Amino-2,6-dinitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 3,5-Dinitroaniline |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 1,3-Dinitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 2,4-Dinitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 2,6-Dinitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | HMX |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | RDX |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | Nitroglycerine |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | Nitrobenzene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 2-Nitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 3-Nitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 4-Nitrotoluene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | Pentaerythritol (PETN) |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | RDX |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | Tetryl |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 1,3,5-Trinitrobenzene |
| HPLC-UV | EPA 8330<br>EPA 8330A MOD<br>EPA 8330B MOD | 2,4,6-Trinitrotoluene |
| ICP-AES | EPA 6010B/ 6010C | Aluminum |
| ICP-AES | EPA 6010B/ 6010C | Antimony |
| ICP-AES | EPA 6010B/ 6010C | Arsenic |
| ICP-AES | EPA 6010B/ 6010C | Barium |
| ICP-AES | EPA 6010B/ 6010C | Beryllium |
| ICP-AES | EPA 6010B/ 6010C | Cadmium |
| ICP-AES | EPA 6010B/ 6010C | Calcium |
| ICP-AES | EPA 6010B/ 6010C | Chromium |
| ICP-AES | EPA 6010B/ 6010C | Cobalt |
| ICP-AES | EPA 6010B/ 6010C | Copper |
| ICP-AES | EPA 6010B/ 6010C | Iron |
| ICP-AES | EPA 6010B/ 6010C | Lead |
| ICP-AES | EPA 6010B/ 6010C | Magnesium |
| ICP-AES | EPA 6010B/ 6010C | Manganese |
| ICP-AES | EPA 6010B/ 6010C | Molybdenum |
| ICP-AES | EPA 6010B/ 6010C | Nickel |
| ICP-AES | EPA 6010B/ 6010C | Potassium |
| ICP-AES | EPA 6010B/ 6010C | Selenium |
| ICP-AES | EPA 6010B/ 6010C | Silver |
| ICP-AES | EPA 6010B/ 6010C | Sodium |
| ICP-AES | EPA 6010B/ 6010C | Thallium |
| ICP-AES | EPA 6010B/ 6010C | Vanadium |
| ICP-AES | EPA 6010B/ 6010C | Zinc |
| ICP-MS | EPA 6020/ 6020A | Aluminum |
| ICP-MS | EPA 6020/ 6020A | Antimony |
| ICP-MS | EPA 6020/ 6020A | Arsenic |
| ICP-MS | EPA 6020/ 6020A | Barium |
| ICP-MS | EPA 6020/ 6020A | Beryllium |
| ICP-MS | EPA 6020/ 6020A | Cadmium |
| ICP-MS | EPA 6020/ 6020A | Calcium |
| ICP-MS | EPA 6020/ 6020A | Chromium |
| ICP-MS | EPA 6020/ 6020A | Cobalt |

| Solid and Chemical Materials | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| ICP-MS | EPA 6020/ 6020A | Copper |
| ICP-MS | EPA 6020/ 6020A | Iron |
| ICP-MS | EPA 6020/ 6020A | Lead |
| ICP-MS | EPA 6020/ 6020A | Magnesium |
| ICP-MS | EPA 6020/ 6020A | Manganese |
| ICP-MS | EPA 6020/ 6020A | Molybdenum |
| ICP-MS | EPA 6020/ 6020A | Nickel |
| ICP-MS | EPA 6020/ 6020A | Potassium |
| ICP-MS | EPA 6020/ 6020A | Selenium |
| ICP-MS | EPA 6020/ 6020A | Silver |
| ICP-MS | EPA 6020/ 6020A | Sodium |
| ICP-MS | EPA 6020/ 6020A | Thallium |
| ICP-MS | EPA 6020/ 6020A | Vanadium |
| ICP-MS | EPA 6020/ 6020A | Zinc |
| CVAA | EPA 7471A/ 7471B | Mercury |
| Ion Chromatography | EPA 300.0 / 9056 | Bromide |
| Ion Chromatography | EPA 300.0 / 9056 | Chloride |
| Ion Chromatography | EPA 300.0 / 9056 | Fluoride |
| Ion Chromatography | EPA 300.0 / 9056 | Nitrate-N |
| Ion Chromatography | EPA 300.0 / 9056 | Nitrite-N |
| Ion Chromatography | EPA 300.0 / 9056 | Sulfate |
| Ion Selective Electrode | SM 4500-NH3 D | Ammonia |
| Ion Selective Electrode | EPA 9045C | pH |
| UV-VIS Spectrometer | SM 4500-CN E<br>EPA 9010B/ 9014 | Cyanide |
| UV-VIS Spectrometer | EPA 7196A | Hexavalent Chromium |
| Titration | EPA 9034 | Sulfide |
| **Preparation** | **Method** | **Analyte** |
| Purge & Trap | EPA 5035/ 5035A | Preparation for Volatiles in Soil |
| Extraction | EPA 3550B/ 3550C | Sonication Extraction for Semivolatile Organics (DRO, BNA, PCB, Pesticides, SIM) |
| Extraction | EPA 8330/ 8330A | Extraction of Nitroaromatics & Nitramines from Solids |
| Digestion | EPA 3060 | Alkaline Digestion for Hexavalent Chromium |
| Digestion | EPA 3050B | Soil Digestion for ICP & ICP-MS Metals |
| Leaching Procedure | EPA 1311 | TCLP – Toxicity Characteristic Leaching Procedure |
| Leaching Procedure | EPA 1312 | SPLP – Synthetic Precipitation Leaching Procedure |

| Air and Emissions | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-TCD | ASTM D1946-90 | Carbon Dioxide |
| GC-TCD | ASTM D1946-90 | Carbon Monoxide |
| GC-TCD | ASTM D1946-90 | Helium |
| GC-TCD | ASTM D1946-90 | Hydrogen |
| GC-TCD | ASTM D1946-90 | Methane |
| GC-TCD | ASTM D1946-90 | Nitrogen |
| GC-TCD | ASTM D1946-90 | Oxygen |
| GC-MS | TO-15 | 1,1,1-Trichloroethane |
| GC-MS | TO-15 | 1,1,2,2-Tetrachloroethane |
| GC-MS | TO-15 | 1,1,2-Trichloroethane |
| GC-MS | TO-15 | 1,1-Dichloroethane |
| GC-MS | TO-15 | 1,1-Dichloroethene |
| GC-MS | TO-15 | 1,2,4-Trichlorobenzene |
| GC-MS | TO-15 | 1,2,4-Trimethylbenzene |
| GC-MS | TO-15 | 1,2-Dibromoethane |
| GC-MS | TO-15 | 1,2-Dichlorobenzene |
| GC-MS | TO-15 | 1,2-Dichloroethane |
| GC-MS | TO-15 | 1,2-Dichloropropane |
| GC-MS | TO-15 | 1,3,5-Trimethylbenzene |
| GC-MS | TO-15 | 1,3-Butadiene |
| GC-MS | TO-15 | 1,3-Dichlorobenzene |
| GC-MS | TO-15 | 1,4-Dichlorobenzene |
| GC-MS | TO-15 | 2-Butanone |
| GC-MS | TO-15 | 2-Hexanone |
| GC-MS | TO-15 | 4-Ethyltoluene |
| GC-MS | TO-15 | 4-Methyl-2-Pentanone |
| GC-MS | TO-15 | Acetone |
| GC-MS | TO-15 | Acrolein |
| GC-MS | TO-15 | Benzene |
| GC-MS | TO-15 | Benzyl chloride |
| GC-MS | TO-15 | Bromodichloromethane |
| GC-MS | TO-15 | Bromoform |
| GC-MS | TO-15 | Bromomethane |
| GC-MS | TO-15 | Carbon Disulfide |
| GC-MS | TO-15 | Carbon Tetrachloride |
| GC-MS | TO-15 | Chlorobenzene |
| GC-MS | TO-15 | Chloroethane |
| GC-MS | TO-15 | Chloroform |
| GC-MS | TO-15 | Chloromethane |
| GC-MS | TO-15 | cis-1,2-Dichloroethene |
| GC-MS | TO-15 | cis-1,3-Dichloropropene |
| GC-MS | TO-15 | Cyclohexane |
| GC-MS | TO-15 | Dibromochloromethane |
| GC-MS | TO-15 | Ethyl Acetate |
| GC-MS | TO-15 | Ethylbenzene |

| Air and Emissions | | |
|---|---|---|
| **Technology** | **Method** | **Analyte** |
| GC-MS | TO-15 | Freon 113 |
| GC-MS | TO-15 | Freon 114 |
| GC-MS | TO-15 | Freon 12 |
| GC-MS | TO-15 | Hexachlorobutadiene |
| GC-MS | TO-15 | m,p-Xylenes |
| GC-MS | TO-15 | Methylene Chloride |
| GC-MS | TO-15 | MTBE |
| GC-MS | TO-15 | Naphthalene |
| GC-MS | TO-15 | n-Heptane |
| GC-MS | TO-15 | n-Hexane |
| GC-MS | TO-15 | o-Xylene |
| GC-MS | TO-15 | Propylene |
| GC-MS | TO-15 | Styrene |
| GC-MS | TO-15 | Tetrachloroethene |
| GC-MS | TO-15 | Tetrahydrofuran |
| GC-MS | TO-15 | Toluene |
| GC-MS | TO-15 | trans-1,2-Dichloroethene |
| GC-MS | TO-15 | trans-1,3-Dichloropropene |
| GC-MS | TO-15 | Trichloroethene |
| GC-MS | TO-15 | Trichlorofluoromethane |
| GC-MS | TO-15 | Vinyl Acetate |
| GC-MS | TO-15 | Vinyl Chloride |

Notes:

1) This laboratory offers commercial testing service.
2) This scope is formatted as part of a single document including Certificate of Accreditation No. L2442

Vice President

Curtis & Tompkins, Ltd.

# Acid Digestion of Soil & Solid Samples For Total Metals Analysis by ICP-AES and ICP-MS

# EPA 3050B DIGESTION

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Justin Buehner<br>Department Manager | | 1-28-16 |
| Teresa Morrison<br>QA Director | Teresa K Morrison | 01/28/16 |
| **Reapproved:** | | |
| Teresa Morrison<br>QA Director | Teresa K Morrison | 2/20/17 |
| | | |
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Acid Digestion of Soil & Solid Samples
For Total Metals Analysis by ICP-AES and ICP-MS

# EPA 3050B DIGESTION

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Acid Digestion of Soil & Solid Samples
For Total Metals Analysis by ICP-AES and ICP-MS

# EPA 3050B DIGESTION

## 1.0 SCOPE

This SOP details the acid digestion of solid samples for later analysis by ICP-AES and ICP-MS. This procedure is not applicable to the analysis of mercury (see EPA 7470 and 7471 for the mercury procedures). This procedure is *not* a total digestion technique but is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available".

The sample is digested with nitric acid and hydrogen peroxide under heat. The digestate is then refluxed with hydrochloric acid. This digestion procedure reduces interferences due to organic matter and converts metals that are adsorbed onto particulate matter into a form that can be determined by atomic absorption (flame AA) or inductively-coupled plasma spectroscopy (ICP).

The same procedure for both ICP-OES and ICP-MS is applied based on recommendation from the ICP-MS instrument manufacturer (Agilent). The use of HCl improves solubility of antimony, barium, lead, and silver. The ICP-MS instrument software includes correction factors for chloride and common chloride interferences.

***Multi-Incremental (MI) Sampling***:
Some projects require the use of a 10 gram sample size, sampled "multi-incrementally". Procedures specific to MI samples appear in each applicable section.

## 2.0 REFERENCES

*Sample Preparation:*
EPA 3050B, *Acid Digestion of Sediments, Sludges, and Soils,* SW-846, Dec 1996
ASTM D 6323-98, *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (Reapproved 2003)

*Subsequent Analytical Method:*
EPA 6010B, *Inductively Coupled Plasma-Atomic Emission*, SW-846, Dec 1996
EPA 6010C, *Inductively Coupled Plasma-Atomic Emission*, SW-846, Feb 2007
EPA 6020, *Inductively Coupled Plasma-Mass Spectrometry*, SW-846, Dec 1996
EPA 6020A, *Inductively Coupled Plasma-Mass Spectrometry*, SW-846, Feb 2007

*Additional SOPs and Guidance Documents:*
C&T SOP CS 2.3, *Subsampling & Compositing*
C&T SOP QA 1.4, *Balance Calibration Check & Maintenance*
C&T SOP QA 1.6, *Pipette Calibration Check Procedures*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL) & Limit of Detection (LOD)*
C&T SOP QA 4.6, *Limit of Quantitation (LOQ)*
C&T SOP CS 2.4, *Multi-Incremental Subsampling*

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Volume 1, TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2 October 2010
DoD/DoE Consolidated QSM, Version 5.0, July 2013

## 3.0 PRESERVATION & HOLDING TIME

Preservation: Store at > 0.0 to 6.0°C
Holding Time: 6 months

## 4.0 SAFETY

This procedure involves the use of strong acids and reagents that *will* cause injury if allowed to contact skin or eyes. Assume that all samples contain hazardous and/ or toxic chemicals. Wear a lab coat, gloves, and safety glasses whenever handling samples, standards, or reagents.

## 5.0 QC REQUIREMENTS

Client Quality Assurance Project Plans (QAPPs) may require different acceptance limits than those listed below; for samples from those projects, the QAPP requirements supersede C&T in-house requirements.

| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Method Blank | 1 for every batch of 20 or fewer samples | <1/2 RL<br><br>QSM 4.2:<br><1/2 LOQ, except common contaminants (ie: Zinc) <LOQ<br><br>QSM 5.0: <1/2 LOQ | Report samples that are <1/2 RL/LOQ or >10x contamination; redigest and rerun all others |
| Blank Spike (BS)/ Blank Spike Duplicate (BSD) | 1 for every batch of 20 or fewer samples | %Recovery & %RPD within statistically derived limits<br><br>QSM 4.2/5.0:<br>Within QSM limits, where available | Low recovery: Redigest and reanalyze all samples.<br><br>High recovery: Redigest and reanalyze samples with results >LOQ. Report results <LOQ.<br><br>High RPD: Report ND samples. Redigest & rerun all others |

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| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) | 1 for every batch of 20 or fewer samples | %Recovery & %RPD within statistically derived limits<br><br>QSM 4.2/5.0:<br>Within QSM LCS limits | Flag results as possible matrix interference. |
| Surrogates | Not applicable to Metals analyses | N/A | |
| Method Detection Limit (MDL) Study | Whenever new equipment is installed or significant changes are made to the process | <1/3 LOQ; see MDL SOP for additional details | See MDL SOP |
| Limit of Detection (LOD) | *QSM 4.2/ 5.0:*<br>Verify on each instrument, quarterly<br><br>*TNI-2009:*<br>Verify on each instrument annually | *QSM 5.0*:<br>Spike at 2-4x MDL and <LOQ; analytes detected<br><br>*TNI-2009:*<br>Spike at ≤ 4x MDL | See MDL SOP |
| Limit of Quantitation (LOQ) | *QSM 4.2/ 5.0:*<br>Digest quarterly; analyze on one instrument.<br><br>*TNI-2009:*<br>Digest and analyze on one instrument annually. | Spike at or above (up to 2x) the LOQ.<br><br>Recovery within 50-150% | See LOQ SOP |
| Mechanical Pipettes and Reagent Dispensers | *QSM 5.0:*<br>Verify upon receipt then daily, prior to use, using 3 replicates<br><br>*TNI-2009/ QSM 4.2:*<br>Verify upon receipt then quarterly | *QSM 5.0:*<br>Accuracy within ± 2% of nominal volume<br>RSD ≤ 1% of nominal volume | See QA SOP for Pipettes & Dispensers for details |
| Digestion Tubes | Purchase Class-A, or verify accuracy of each lot upon receipt, using 10 replicates. | *QSM 5.0:*<br>Accuracy: ± 3% of nominal volume<br>RSD ≤ 3% of nominal volume | See Volumetric Glassware SOP (QA 1.9) |

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| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Thermometers & Digestion Blocks | *QSM 4.2/ 5.0:* Verify calibration prior to 1st use then quarterly<br><br>*TNI-2009/ QSM4.2/5.0:*<br>Liquid-In-Glass Thermometers:<br>Verify calibration prior to 1st use then annually | See QA SOP for Temperature Controls for details | See QA SOP for Temperature Controls for details |
| Performance Testing (PT) Samples | Semiannually. | Each element within study limits | Any failures must be investigated, corrected, and a new PT analyzed and passed before end of the semi-annual time-frame. |
| Demonstration of Capability (DOC) | Each new analyst | Initial DOC:<br>4 consecutive LCS within C&T recovery and RPD limits<br><br>Continuing DOC:<br>PT sample within study limits or another 4 consecutive LCS within C&T limits | Retrain analyst and reanalyze DOC. |
| The following are not applicable to this digestion procedure: | | | |
| Surrogates | Not applicable to Metals analyses | | |
| Internal Standards | Added to every sample, QC, and standard by the analyst | See analysis SOPs for details. | See analytical SOP for requirements |
| Initial Calibration Curve (ICAL) | See analytical SOP for requirements | See analytical SOP for requirements | See analytical SOP for requirements |
| Initial Calibration Verification (ICV) | See analytical SOP for requirements | See analytical SOP for requirements | See analytical SOP for requirements |
| Continuing Calibration Verification (CCV) | See analytical SOP for requirements | See analytical SOP for requirements | See analytical SOP for requirements |
| Continuing Calibration Blank (CCB) | See analytical SOP for requirements | See analytical SOP for requirements | See analytical SOP for requirements |

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| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Serial Dilution (SER) | When a new matrix is encountered, or for any QSM project, or if requested by client:<br><br>1 for every batch of 20 or fewer samples | See analysis SOPs for requirements | See analysis SOPs for requirements |
| Post Digestion Spike (PDS) | When a new matrix is encountered, or for any QSM project, or if requested by client:<br><br>1 for every batch of 20 or fewer samples | See analysis SOPs for requirements | See analysis SOPs for requirements |
| Instrument Detection Limit (IDL) Study | See analytical SOP for requirements | See analytical SOP or QA SOP for requirements | See analytical SOP or QA SOP for requirements |

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## 6.0 QC DEFINITIONS

6.1 **Initial Calibration** curve is a series of standards that establishes the quantitation range of the instrument.

6.2 **Initial Calibration Verification (ICV)** standard is a “second source” standard that is obtained from a different manufacturer than the standards used for the initial calibration curve. The ICV is used to verify that both the working and source standards used for calibration were prepared correctly.

6.3 **Initial Calibration Blank (ICB)** is a reagent blank that follows the ICV and is used to demonstration that the instrument is free of contamination.

6.4 **Continuing Calibration Verification (CCV)** standards are analyzed throughout the sequence to demonstrate that the curve is still valid.
*Note:* The US Army Corp of Engineers recommends that the CCV’s be prepared from the same source standards as the ICAL curve in order to better differentiate between changing instrument response and simple differences between standards.

6.5 **Calibration Blanks (CCB)** are reagent blanks that follow every CCV and are used to verify that the instrument remains free of contamination.

6.6 **Batch QC:** The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples.

6.6.1 **Method Blank (MB):** An aliquot of deionized water is digested and analyzed with each batch of 20 or fewer samples to demonstrate that the glassware and reagents are free of contamination.

6.6.2 **Blank Spike / Blank Spike Duplicate (BS/BSD):** A blank spike and a blank spike duplicate are aliquots of deionized water to which a known amount of analyte is added. This solution is then digested and analyzed with every batch of 20 or fewer samples to demonstrate that the procedure is accurate (measured by the *recovery*) and precise (measured by the *RPD*) in the *absence* of matrix interferences.

6.6.3 **Matrix Spike (MS) / Matrix Spike Duplicate (MSD):** An MS and MSD are aliquots of real world samples to which a known amount of analyte is added prior to sample digestion. The MS/MSD are used to demonstrate the accuracy and precision of the process on environmental samples, which may include matrix interferences.

*Note:* Client- or project-specific quality assurance project plans (QAPPs) may require an a Sample Duplicate (SDUP) and Sample Spike (SSPIKE) in place of the MS/MSD. For batches containing samples from these projects, the QAPP requirement takes precedence over this SOP.

6.7 **Sample Interference Verifications**: The method recommends that whenever a new or unusual matrix is encountered, a series of tests be performed to ensure that neither positive nor negative interferences are distorting the accuracy of the reported values. These are optional steps that should be implemented for any batch containing “Level 3” or Level 4” samples, as nearly all Department of Defense project plans include this requirement. These should also be analyzed whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

6.7.1 **Serial Dilutions:** Analysis of a 5x dilution should agree with the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference, such as viscosity, should be suspected.

6.7.2 **Post Digest Spikes:** An analyte is added to a portion of a prepared sample digest, or its dilution, and analyzed to determine if matrix interferences that occur during digestion, are influencing the sample result.

6.8 **Instrument Detection Limit (IDL)**: An IDL study is determined by analyzing seven instrument blanks on three non-consecutive days to determine if the instrument is capable of meeting desired detection limits, before taking digestion procedures into account. The standard deviation of the calculated concentrations is determined for each day; the IDL is the sum of the three standard deviations.

6.9 **Method Detection Limit (MDL):** An MDL study is a set of at least 7 low-level blank spikes that are prepared and analyzed to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. See the QA SOP “Determining MDLs” for details.

6.10 **Limit of Detection (LOD)** samples are aliquots of deionized water that are spiked at or just above the calculated MDL, to verify that that the analyte can be detected at those

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levels. The LOD must be verified quarterly on every instrument for DoD (or annually for NELAC compliance). See the QA SOP 4.4 'Method Detection Limits' for details.

6.11 **Limit of Quantitation (LOQ)** samples are aliquots of deionized water that are spiked at 1-2x the lowest calibration standard and used to verify the accuracy of the procedure at the reporting limit. The LOQ must be determined quarterly on a single instrument for DoD (or annually for NELAC). See the QA SOP 4.6 'Limit of Quantitation' for details.

## 7.0 INTERFERENCES & TROUBLE-SHOOTING

7.1 Lead and zinc are common contaminants in the lab, they are present in dust particles; keeping the sample prep area clean and dusted will reduce contaminants, as will loosely covering the digestates during the heating steps.

7.2 Soils containing high levels of carbonates may foam when acid is added; add the acid slowly or use a smaller sample size.

7.3 Volatile elements (particularly antimony) may be lost during the digestion process if the digestate is superheated or allowed to go dry during heating.

7.4 Silver may precipitate during the digestion process if present in relatively high levels; samples submitted by photo-processing or reclamation clients (Dean X-Ray, Safety Kleen, etc.) should not be digested prior to analysis for silver. Standards should be stored away from light to prevent photo-induced precipitation of silver.

## 8.0 EQUIPMENT

Disposable 50 mL digestion tubes, SPC Science Catalog# 010-500-261
(or 250 mL beaker with watch glasses sized to fit the beakers)
Auto-pipette, adjustable to 0.5mL
Thermometer, minimum range 85-105°C
Digestion Block - adjustable and capable of maintaining a temperature of 90-95°C
Glass Funnels
Teflon boiling chips, Saint-Gobain Chemware Ultra-Pure PTFE Boiling Stones (26397-103)
Disposable watch glasses
Whatman # 541 Filter paper
(# 541 is "ashless", specifically for trace metals, & reduces sodium contamination)
50mL graduated plastic centrifuge tubes, VWR cat#21008-973

<u>Additional/alternate equipment for Multi-Incremental Samples</u>
600 mL beakers with ribbed watch glasses
500 mL graduated cylinder
Hotplate, adjustable and capable of maintaining a temperature of 90-95°C

## 9.0 PROCEDURE

9.1 Calibrate the autopipette and document the results in the pipette benchbook.

9.2 Calibrate the balance and document the results in the balance calibration benchbook.

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9.3 Turn on the digestion block. Place a digestion tube containing 50mL DI water in the block and place the "DigiProbe" temperature probe into this digestion tube. Allow the block to heat until the DigiProbe reads 95°C (usually takes 20 - 30 minutes). Document the temperature and thermometer location in the bench book. The thermometer should be rotated through each position in the block, each day the thermometer should be moved to the next position.

**MI samples**: Turn on the hotplate. Place a covered beaker with 200mL water and a calibrated thermometer on the hotplate. Allow to heat until the temperature reaches 95°C (usually takes 20 - 30 minutes).

9.4 While the block (or hotplate) is heating, check samples out of the coldroom and allow them to come to room temperature.

9.5 Write the sample number and bottle *letter* of each sample in the digestion log.

9.6 Use the "Batching" screen in LIMS to assign a batch number and create "QC" numbers for the batch QC. Document the batch and QC numbers in the prep log.

9.7 Label disposable digestion tubes (or beakers for MI samples) with the sample numbers, including a MB, BS, BSD, SSPIKE, and SDUP (or MS and MSD if needed). Place tubes in the same order in the rack as they are written in the digestion log.

9.8 Use a disposable wooden spatula.

9.9 Discard the top ~1cm of sample, to ensure that the aliquot used was not contaminated by the field equipment.

*Note:* Discard any leaves, twigs, large stones, etc and take a visually representative aliquot of each sample. Document your observations and actions (e.g.: "discarded leaves & twigs") in the prep log.

9.10 Using a new wooden spatula for each sample, thoroughly homogenize the next several cm of sample, to achieve homogeneity then weigh 1g (± 0.1g) of sample into the disposable digestion tube labeled with that sample number. Record the weight of sample (to 0.01g) in digestion benchbook.

*Note:* If a client requests that C&T "composite" the samples, see Appendix 3 for instructions.

**MI samples**: Follow the procedures in the Multi-Incremental Sub-sampling SOP to obtain a 10 gram sample.

9.11 For the Method Blank (BLANK), weigh out 1.0 g (± 0.1g) of Teflon boiling chips.

9.12 For the Blank Spike (BS), weigh out 1.0 g (± 0.1g) of Teflon boiling chips then use a calibrated autopipette to add the appropriate volumes of spiking solutions as listed in Appendix 1 below.

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9.13 Repeat, with the tube labeled "BSD", for the Blank Spike Duplicate (BSD). Record the volume added and the LIMS S# of the spike in the digestion log.

9.14 Review the job sheets to determine if any of the clients in the batch requested that matrix QC be done on their sample. If so, use that sample for the SSPIKE and SDUP (or MS/ MSD), otherwise choose a sample for batch QC so that matrix QC is rotated throughout the laboratory's clients and so that no one client's samples predominate over a period of time.

9.15 Thoroughly homogenize about 10g of the sample selected as the batch QC sample, before taking any aliquots. (Does not apply to MI samples)

9.16 For the sample spike (SSPIKE), weigh a second 1g (± 0.1g) aliquot of the sample chosen for batch QC into the labeled digestion tube.

Record the weight in the benchbook, then use a calibrated autopipette use a calibrated autopipette to add the appropriate volumes of spiking solutions as listed in Appendix 1 below.

9.17 For the SDUP, weigh a third 1g aliquot of the sample chosen for batch QC into the digestion tube labeled "SDUP". (MI samples: use 10 grams)

*Note:* If a client requests an MS/MSD on their sample, prepare two aliquots of the SSPIKE (and call them "MS" and "MSD" respectively) in place of the SSPIKE and SDUP.

9.18 Add 5 mL of 1:1 nitric acid ($HNO_3$) to each tube. Record the C&T ID of the nitric acid in the digestion benchbook.

MI Samples: Use 50 mL of 1:1 HNO3

9.19 Cover each tube with a disposable watch glass and place the tubes in the 90-95°C digestion block for 10-15 minutes *without boiling*, then remove from block and allow to cool to room temperature.

MI samples: Place beakers covered with ribbed watch glasses on the hotplate.

9.20 Add 2.5 mL concentrated nitric acid then cover and return to the digestion block (or hotplate) and reflux for 30 minutes. If brown fumes persist, repeat this step until no further brown fumes are produced.

MI samples: Use 25 mL concentrated HNO3

9.21 While the samples are digesting, record the C&T reagent ID of the 1:1 $HNO_3$, the manufacturer and lot# of the nitric acid in the digestion benchbook.

9.22 Reduce the volume to about 5 mL without boiling or allowing it to go to dryness.

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MI samples: Reduce to 50 mL

*Note:* If the sample volume will not reduce to 5 mL (50 mL for MI samples), digest for an additional 2 hours, then continue with the steps below.

9.23 Remove the test tubes from the block digester (or beakers from the hotplate) and cool to room temperature.

9.24 Using a calibrated autopipette, add 1 mL water and 1.5 mL of 30% (as purchased) hydrogen peroxide and swirl tube. Record the C&T ID of the peroxide in the benchbook.

MI samples: Add 10mL water + 15 mL 30% $H_2O_2$

9.25 Return covered tubes to digestion block (or hotplate) and warm until effervescence subsides.

*Note:* If the sample foams over on additional of hydrogen peroxide, re-digest the sample using a smaller sample weight and note the problem in the digestion log.

9.26 Continue adding 0.5 mL portions of 30% hydrogen peroxide and heating until sample appearance is does not change (do not exceed 5 mL).

MI samples: Use 5.0 mL aliquots of 30% $H_2O_2$ (do not exceed 50mL)

9.27 Reduce the sample volume to 5 mL again, without boiling or allowing it to go dry.

MI samples: Reduce to 50 mL again

9.28 Add 5mL concentrated HCl and swirl tube to mix thoroughly. Record the HCl manufacturer and lot# in the benchbook.

MI samples: Use 50mL concentrated HCl

9.29 Return the covered tubes to the digestion block (or hotplate) and heat at 95°C for 15 minutes.

9.30 Remove the tubes from the digestion block (or hotplate) and cool to room temperature.

9.31 Cool and bring to 50 mL volume with deionized water.

MI samples: Bring to 500 mL in a clean, acid-rinsed 500 mL graduated cylinder.

*Method Modification Note:* C&T uses a digestion block in place of the hot plate digestion discussed in 3050B because the block provides better temperature control and uniform heating across the samples. The final volume by this procedure is 50mL instead of 100mL as discussed in 3050b, because using the 50mL disposable digestion tubes for the entire

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process provides a complete digestion while eliminating potential cross-contamination. The reagent volumes have been adjusted for the 1g sample weight and lower final volume.

9.32 Cap, invert and shake tube several times to ensure good mixing.

MI samples: Cover cylinder with parafilm.

9.33 Label clean, dry 50 mL disposable centrifuge tubes with the sample IDs.

9.34 Line up centrifuge tubes in the rack, in the same order as written in the bench book.

9.35 Check sample ID of digestion tube against centrifuge tube and make sure they match before filtering the sample.

9.36 Filter the sample through Whatman # 541 filter paper into the appropriate centrifuge tube, or allow sample to settle and then decant the top portion (excluding any settled matter) into the centrifuge tube.

*Note:* If any of the samples are filtered, the batch QC must also be filtered and the filtration must be documented in the digestion benchbook.

9.37 Complete benchbook entries then use the 'Prep Entry' screen in LIMS to enter all prep information, including spiking standards and volumes.

9.37 Scan the completed benchbook page into LIMS then verify that the scanned copy is legible.

9.38 Transfer custody of the samples to the analyst.

**10.0 DOCUMENTATION**

The benchbook entries must include:

Date sample digestion
C&T sample ID's (including container letter), initial and final volumes digested
Identity of QC Samples (spikes, duplicates & LCS)
Record of whether or not the digests were filtered
LIMS numbers of all spiking solutions, Volume of spikes added
*Unique* ID of all reagents used (mfg lot# or C&T assigned ID)
IDs of pipettes, thermometers and digestion block(s) used
Any unusual occurrences during digestion

**11.0 POLLUTION PREVENTION**

The digestion should be performed in a fume hood. No other pollution prevention measures are currently applicable to this analysis, except for the proper disposal of the samples.

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## 12.0 WASTE DISPOSAL

All containers holding chemical waste (liquid or solid) must be kept closed except when necessary to add or remove waste. Waste container must be compatible with the type of material being stored and must be in good condition (ie: not rusted or cracked).

Each container must be clearly labeled "Hazardous Waste", with a description of the composition, physical state of the waste ('Liquid/Solid'), the type of hazard (Corrosive, Flammable Solvent, etc. ), and the date that the first contents are added to the container

After the analysis is completed, the excess digestate and any expired standards volume must be properly discarded by:

12.1 Sample digests and acidified filtrates should be kept for at least 6 months prior to disposal. After 6 months, the digests should be transferred to the 55-gallon tight-head HDPE ("poly") Acid Waste drum in the garage.

12.2 Expired or discontinued spiking standards and instrument waste should also be accumulated in the metals lab for no more than 9 months before being transferred to the 55-gallon tight-head HDPE ("poly") Acid Waste drum in the garage.

## 13.0 REVISION HISTORY

The previous document (revision 14) was changed as follows:

- Section 5: Updated support equipment calibration details to match QSMv5
- Section 8: Added Teflon boiling chips to equipment list
- Section 9: Added requirement to use boiling chips for the method blank and blank spikes to represent a solid matrix. Updated spiking instructions to refer to the details in Appendix_1. Added requirement to use watch glasses during digestion. Added instruction to "batch" the samples in LIMS and use the 'prep entry' screen to record prep information.
- Section 10: Add traceability requirement for support equipment
- Section 12: Expanded waste accumulation & disposal details
- Appendix_1: Updated spiking information to match new standards
- Appendix_2: Updated spike information to list new standards

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**APPENDIX_1: ICP or ICP-MS SOIL DIGESTION SUMMARY**

EPA 3050B

| | | |
|---|---|---|
| Sample Weight: | 1g (± 0.1g) | |
| Spike: | 6010 (ICP) | + 0.5 mL of each spiking standard (A,B,C) |
| | 6020 (ICPMS) | + 0.25 mL of each spiking standard (A,B,C) |
| 1:1 Digestion: | + 5 mL 1:1 $HNO_3$<br>95 °C<br>10-15 minutes | |
| Conc. $HNO_3$ Digestion: | + 2.5 mL concentrated $HNO_3$<br>95 °C<br>30 minutes<br>repeat until no brown fumes are produced<br>reduce volume to < 5mL (or digest for 2 hours) | |
| $H_2O_2$ Digestion: | + 1.0 mL DI water and 1.5 mL 30% $H_2O_2$<br>95 °C<br>add 0.5 mL 30% $H_2O_2$ until no effervescence and appearance is stable<br>reduce volume to < 5mL (or digest for 2 hours) | |
| HCl Digestion: | + 5mL concentrated HCl<br>95 °C<br>15 minutes | |
| Final Volume: | 50 mL | |
| Filtration: | if particulates present in sample<br>Whatman # 541<br>filter QC samples if any sample filtered | |

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ICP or ICP-MS SOIL DIGESTION SUMMARY for
**MULTI-INCREMENTAL SAMPLING**

| | |
|---|---|
| Sample Weight: | 10g (± 10g) |
| Spike: | For 6010 (ICP) use 5.0 mL of each spiking standard (A,B,C)<br>For 6020 (ICPMS) use 2.5 mL of each spiking standard (A,B,C) |
| 1:1 Digestion: | + 50 mL 1:1 $HNO_3$<br>95 °C<br>10-15 minutes |
| Conc. $HNO_3$ Digestion: | + 25 mL concentrated $HNO_3$<br>95 °C<br>30 minutes<br>repeat until no brown fumes are produced<br>reduce volume to < 50mL (or digest for 2 hours) |
| $H_2O_2$ Digestion: | + 10 mL DI water and 15 mL 30% $H_2O_2$<br>95 °C<br>add 5.0 mL 30% $H_2O_2$ until no effervescence and appearance is stable<br>reduce volume to < 50mL (or digest for 2 hours) |
| HCl Digestion: | + 50mL concentrated HCl<br>95 °C<br>15 minutes |
| Final Volume: | 500 mL |
| Filtration: | if particulates present in sample<br>Whatman # 541<br>filter QC samples if any sample filtered |

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## APPENDIX_2: REAGENTS & STANDARDS

Alternate supplies may be used so long as they are of equivalent quality and all other quality control, and traceability requirements are met.

### REAGENTS

Those reagents that are used as purchased should be labeled with the date opened & initials of chemist who opened it and the expiration date. If an aliquot of a reagent is decanted into another bottle (to reduce changes of contamination, for example), that bottle must be labeled with the contents, concentration, date on which it was decanted, prep chemist's initials, and expiration date of the original reagent.

For those reagents that require additional preparation, including dilutions into DI water, the prep must be documented in the reagent prep benchbook. Assign each reagent a unique ID, based on the manufacturer and the date prepared. Any reagents that are not prepared daily should be labeled with the contents, reagent ID, concentration, date prepared, prep chemist's initials, and expiration date.

Each reagent lot should be checked prior to analysis to verify that the levels of impurities are within acceptable levels. Place a copy of the vendor's Certificate of Analysis in the reagents benchbook.

Nitric acid ($HNO_3$), concentrated, Instra-Analyze (trace metals) grade,
JT Baker, VWR catalog # JT9598-34
Store at room temperature for up to 1 year.

1:1 Nitric acid ($HNO_3$) *Warning*: Always add acid to water, as reversing the process may cause hot acid to splatter and cause chemical burns.

Partially fill an empty $HNO_3$ bottle with 500mL of DI water. Slowly add 500mL of concentrated $HNO_3$ to the deionized water. Cap tightly then carefully invert 3 times to mix. Store at room temperature for up to 1 year.

Hydrochloric acid (HCl), concentrated, Instra-Analyze (trace metals) grade,
JT Baker, VWR catalog # JT9530-33
Store at room temperature for up to 1 year.

30% Hydrogen Peroxide ($H_2O_2$) as received,
VWR, 500mL, catalog # VW3690-1
Store at room temperature for up to 1 year.

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**SOURCE STANDARDS**

Source standards are those purchased from a chemical manufacturer or vendor, and should be NIST traceable. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#. If you need more details, log into the LIMS browser; follow the 'LAB MENU' link and click on the "New Standards System (March 2005)" link for details on the system.

Each standard should be traceable to NIST. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is **1 year** from the date received, or sooner if comparison with check standards indicates a problem.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory".

Write the S#, date received, and expiration date on the certificate (if the vendor did not list the date) then scan it into LIMS. Query the certificate to verify that the scanned copy is complete and legible.

Store source standards at room temperature, away from light (to prevent photo-induced precipitation of silver).

Spiking Solutions are purchased as custom standards and used without an intermediate dilution.

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**Spiking Solution A** (ESI, High Purity Standards # C1-151015RH01)

| Element | Concentration (µg/mL) | Element | Concentration (µg/mL) | Element | Concentration (µg/mL) |
|---|---|---|---|---|---|
| Aluminum | 100 | Cobalt | 100 | Thallium | 100 |
| Antimony | 100 | Copper | 100 | Tin | 100 |
| Arsenic | 100 | Manganese | 100 | Titanium | 100 |
| Barium | 100 | Molybdenum | 100 | Uranium | 100 |
| Beryllium | 50 | Nickel | 100 | Vanadium | 100 |
| Boron | 100 | Lead | 100 | Zinc | 100 |
| Cadmium | 100 | Selenium | 100 | | |
| Chromium | 100 | Strontium | 1,000 | | |

**Spiking Solution B** (ESI, High Purity Standards # C1-151015RH02)

| Element | Concentration (µg/mL) | Element | Concentration (µg/mL) |
|---|---|---|---|
| Calcium | 1,000 | Phosphorus | 1,000 |
| Iron | 1,000 | Potassium | 1,000 |
| Magnesium | 1,000 | Sodium | 1,000 |

**Spiking Solution C** (Inorganic Ventures # MSAG-10PPM)

| Element | Concentration (µg/mL) |
|---|---|
| Silver | 10 |

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**APPENDIX_3: COMPOSITING SOIL SAMPLES**

Clients may ask C&T to "composite" samples during preparation, to yield an average result instead of running each sample as a discrete sample. If a composite is requested, use the following steps to create a representative sample and document the composite:

1.) Verify that the balance has been calibrated earlier in the day. If it has not, calibrate it before continuing. Use a minimum of 1g of each sample being added to the composite, as we need to take subsamples that are representative of the entire contents of each core or bottle.

2.) Group the samples to be included in the composite and determine what size container will be needed to create more than enough of the composite for all of the analyses needed.

3.) Label a pre-cleaned container with the C&T sample number of the composite.

4.) Place the container on the scale and tare the scale.

5.) If samples were received in brass or steel sleeves/cores, use a clean spatula or equivalent tool to remove and discard the top ~1cm from the first sample sleeve. Discard any leaves, twigs, large stones, etc and take a visually representative aliquot of each sample. Document your observations and actions (ie: "discarded leaves & twigs") in the prep log.

6.) Using the same spatula, thoroughly homogenize the next several cm of sample then weigh the necessary aliquot out of this homogenized fraction.

7.) Clean the spatula or tool between samples using deionized water and a clean paper towel, to ensure that there is no contamination between the discrete samples.

8.) Repeat Steps 5-7 for each of the remaining samples to be included in the composite, using exactly the same weight for each aliquot.

9.) In the appropriate analysis or Soil Aliquot benchbook, write the C&T sample number of the composite, along with the sample numbers, bottle letters, and weight used from each of the discrete samples being included in the composite.

| | | |
|---|---|---|
| Example: | 242689-001 | comp -001 A-D, 15.0g of each |
| | 242714-001 | comp -1A, -2A, -3A, 20.0g of each |

*Note:* When using composites that have been previously prepared, write "premade comp", "xlab comp", etc. under the Comments/Observations heading.

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# METALS BY ICP-MS

## EPA 6020, EPA 6020A, EPA 200.8

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Justin Buehner<br>Department Manager | | 8.30.13 |
| Teresa Morrison<br>QA Director | | 8/30/13 |
| **Reapproved:** | | |
| Teresa Morrison<br>QA Director | | 9/18/14 |
| Teresa Morrison<br>QA Director | | 10/15/15 |
| Teresa Morrison<br>QA Director | | 11/09/16 |
| | | |
| | | |

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# METALS by ICP/MS

EPA 6020, EPA 6020A, EPA 200.8

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This SOP contains information that may only be disseminated to C&T staff, clients, and regulators.

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# METALS by ICP/MS

EPA 6020, EPA 6020A, EPA 200.8

## 1.0 SCOPE

This document describes the analysis of waters, soils, and hazardous waste for inorganic elements by ICP-MS methods EPA 6020, EPA 6020A and EPA 200.8. Methods 6020 and 6020A were written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 200.8 which was written by the EPA's Office of Water specifically for wastewater. EPA 200.8 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix_12 for a summary of EPA 200.8 requirements.

In this analysis, analytes in solution are nebulized and transported to a plasma torch. The ions produced by inductively coupled plasma torch are then measured by mass spectrometry. Background subtraction is used to reduce interferences contributed by the plasma gas, reagents, and sample matrix. No digestion required for dissolved metals. Acid digestion prior to analysis is required for total metals in water, soil, and wastes.

See Appendix_1 for reporting limits and Appendix_8 for a discussion of interferences.

## 2.0 REFERENCES

*Analytical Methods:*
EPA 6020, Test Methods for Evaluating Solid Waste, SW-846 Update 0, Sep 1994
EPA 6020A, Test Methods for Evaluating Solid Waste, SW-846 Update 4, Feb 2007
EPA 200.8, Methods for Analysis of Water & Wastewater, Rev. 5.4, 1994

*Sample Preparation Methods:*
EPA 3010A, Acid digestion of Aqueous Samples, SW-846, Update 1, Jul 1992
EPA 3050B, Acid Digestion of Solid Samples, SW-846 Update 3, Dec.1996
EPA 200.8, Methods for Analysis of Water & Wastewater, Rev. 5.4, 1994

*Additional SOP's and Guidance Documents:*
C&T SOP QA 1.6, *Pipette Calibration Check Procedures*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)*
C&T SOP QA 4.5, *Instrument Detection Limits (IDL) for ICP & ICP-MS*
C&T SOP QA 4.6, *Limit of Quantitation (LOQ)*
TNI Standard, Volume 1, EL-V1-2009, September 2009
DoD Quality Systems Manual, version 4.2 October 2010
Consolidated DoD/DoE Quality Systems Manual (QSM), DoD version 5.0, July 2013

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## 3.0 PRESERVATION & HOLDING TIME

Bottles: Samples are usually received in preserved, polyethylene bottles; glass bottles are also acceptable (unless Boron or Silica are requested) but are rarely submitted

Preservation: $HNO_3$ to pH $\leq$ 2
If samples are to be analyzed for Mercury as well as the ICP-MS targets, the samples should be stored at >0.0 to 6.0°C

Holding Time: 6 months

### 3.1 Filtration:

Per 40CFR136 Table II Footnote 7, sample filtration should be performed in the field, within 15 minutes of sample collection, however if an unfiltered sample is submitted for dissolved metals, the sample must be filtered through a 0.45 µm Cellulose-Ester membrane filter as soon as possible after receipt, then acidified to pH <2 with $HNO_3$. An aliquot of DI water should also be filtered for use as a prep blank, to verify that the filters did not contaminate the sample. Document the filtration date & time in a sample prep log; the case narrative will note that filtration was done outside the recommended 15 minute window.

### 3.2 Sample Preservation:

Per 40CFR136 Table II Footnote 7, sample preservation should be performed in the field, within 15 minutes of sample collection, however if sample preservation (acidification) is done by C&T and not by the sampling crew, the sample control technician should add sufficient nitric acid to bring the pH to < 2 as soon as possible after receipt, noting the preservation date and time in the LIMS login "comment" field. The metals prep analyst should then wait at least 24 hours before verifying the pH and digesting the samples. If the pH is <2, document this verification in the comment section of the bench book. If the pH is >2, the preservation and 24 hour wait must be repeated one more time; document this verification on the job sheet and in the comment section of the bench book. If the samples are being analyzed for dissolved metals and are filtered by the lab, the 24 hour hold time post acidification is not needed.

## 4.0 SAFETY

Allow digests to cool to room temperature prior to analysis. Sample digests contain concentrated acids and should be handled with caution. Assume all samples, reagents and standards contain hazardous and/ or toxic material and take necessary precautions.

*Caution:* The torch and interface are hot and should to cool for about 10 minutes before handling.

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## 5.0 QC REQUIREMENTS

| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Internal Standards | Add to every sample, batch QC, and standard | 6020:<br>%Recovery: 30-120<br><br>6020A:<br>%Recovery: 30-120<br><br>200.8:<br>%Recovery: 60-125<br><br>QSM 4.2/5.0:<br>%Recovery: 30-120 | Dilute and reanalyze for elements associated with failing IS |
| Tune Check | Beginning of analytical sequence (and every 12 hours) | 6020/6020A Require:<br>• Precision: ≤ 5% RSD over at least 4 exposures (CT uses 5)<br>• Resolution: < 0.9 amu @ 10% Peak Height<br>• Mass Calibration: ±0.1 amu<br><br>200.8 Requires:<br>• Precision: ≤ 5% RSD over 5 exposures<br>• Resolution: < 1 amu @ 5% Peak Height<br>• Mass Calibration: ±0.1 amu | Retune instrument and rerun check; if still out, perform instrument maintenance and retune instrument, then rerun tune check. |

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| | | | |
|---|---|---|---|
| Initial Calibration Curve (ICAL) | Prior to sample analysis or whenever CCVs fail and maintenance does not correct the problem.<br><br>Minimum 5 points + a blank; | 6020: Correlation Coefficient (r) ≥ 0.995<br><br>6020A: Correlation Coefficient (r) ≥ 0.998<br><br>200.8: Correlation Coefficient (r) ≥ 0.995<br><br>QSM 4.2/5.0: Coefficient (r) ≥ 0.995 – or $r^2$ ≥ 0.99 | Perform instrument maintenance and reanalyze curve. |
| Initial Calibration Verification (ICV) | Mid-level standard run after ICAL curve and before samples | 6020/ 6020A/ 200.8: %D ≤ 10<br><br>QSM 4.2/5.0: %D ≤ 10 | Perform maintenance and rerun; if still out, reanalyze curve. |
| 6020A Low Level ICV (LLICV, CRI) | **For 6020A only:** Concentration ≤LOQ; run after mid-level ICV and before samples | 6020A: %D ≤ 30<br><br>QSM 4.2/5.0: %D ≤ 20% | Perform maintenance and rerun; if still out, reanalyze curve. |
| Initial Calibration Blank (ICB) | After ICV and before samples | 6020: <3xIDL<br><br>6020A: <LOQ<br><br>200.8: <2.2xMDL<br><br>QSM 4.2/5.0: <LOD | Report samples that are ND or >10x contamination; rerun all others |
| Interference Check Standard A (ICS-A) | 6020/ 6020A/ 200.8: Methods do not specify the frequency<br><br>DoD QSM 4.2: Beginning of each sequence, after the ICV and before samples, every 12 hours, and at end of the sequence | C&T inhouse criteria: Unspiked elements < \| LOQ \|<br><br>6020/6020A: No limit specified in methods.<br><br>200.8: Does not include this standard<br><br>QSM 4.2/5.0: Unspiked elements <LOD | Perform instrument maintenance and rerun any elements that are not verified trace impurities from one of the spiked analytes. |

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| | | | |
|---|---|---|---|
| Interference Check Standard AB (ICS-AB) | 6020/ 6020A/ 200.8: Methods do not specify the frequency<br><br>DoD QSM 4.2: Beginning of each sequence, after the ICV and before samples, every 12 hours, and at end of the sequence | C&T inhouse limit: %D ≤ 20<br><br>6020/6020A: No limit specified in methods.<br><br>200.8: Does not include this standard<br><br>QSM 4.2/5.0: %D ≤ 20 | Perform instrument maintenance and rerun any samples requiring affected elements. |
| Method Blank | 1 for every batch of 20 or fewer samples | 6020/ 6020A: <1/2 LOQ<br><br>200.8: <2.2xMDL<br><br>QSM 4.2/5.0: <1/2 LOQ (common lab contaminants, i.e. Zinc, <LOQ) | Report samples that are ND or >10x contamination; redigest and rerun all others |
| Blank Spike/ Blank Spike Duplicate (BS/BSD) | 1 for every batch of 20 or fewer samples | 6020/ 6020A: %Recovery & %RPD within statistically derived limits<br><br>200.8: %Recovery within 85-115, %RPD within statistically derived limits<br><br>QSM 4.2/5.0: Within DoD recovery limits. | Low recovery: Redigest and reanalyze all samples.<br><br>High recovery: Redigest and reanalyze samples with results ≥LOQ. Report results <LOQ.<br><br>High RPD: Report ND samples, rerun all others |

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| | | | |
|---|---|---|---|
| Matrix Spike/ Matrix Spike Duplicate (MS/MSD) | 1 for every batch of 20 or fewer samples | 6020/6020A: %Recovery & %RPD within statistically derived limits<br><br>200.8: %Recovery within 70-130; %RPD within statistically derived limits<br><br>Arizona: %RPD ≤20<br><br>QSM 4.2/5.0: Within DoD LCS recovery limits. %RPD ≤ 20 | Flag results as possible matrix interference. |
| Serial Dilution (SER) | For any DoD QSM project, new matrix type, or when otherwise requested,<br><br>1 for every batch of 20 or fewer samples | For a 5x dilution:<br><br>6020/ 6020A/ 200.8: %D ≤ 10<br><br>QSM 4.2/5.0: %D ≤ 10 | Flag results as possible matrix interference |
| Post Digestion Spike (PDS) | For any DoD QSM project, new matrix type, or when otherwise requested,<br><br>1 for every batch of 20 or fewer samples | 6020: %Recovery within 75-125<br><br>6020A: %Recovery within 80-120<br><br>200.8: Not discussed<br><br>QSM 4.2: %Recovery within 75-125<br><br>QSM 5.0: %Recovery within 80-120% | Dilute and reanalyze; if again outside limits, flag as possible matrix interference |
| Sample Analysis | 6020/ 6020A/ 200.8: Minimum 3 integrations; report average | C&T inhouse criteria: %RSD ≤ 20<br><br>6020/ 6020A/ 200.8: No criteria discussed in methods | Rerun sample; if again fails, report the run with the lower RPD. |

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| | | | |
|---|---|---|---|
| Continuing Calibration Verification (CCV) | Mid-level concentration run at beginning of each sequence, after every ten samples (excluding MB, LCS/BS/BSD), and at end of the sequence | 6020/ 6020A/ 200.8: %D ≤ 10<br><br>200.8: CCV not required by method<br><br>QSM 4.2/5.0: %D ≤ 10 | Perform maintenance and rerun; if still out, reanalyze curve. See Section 11 below for sample guidance. |
| 6020A Low Level CCV (LLCCV) | **For 6020A only:** Concentration ≤LOQ;<br><br>6020A: At end of sequence (recommend more frequently)<br><br>QSM 4.2/5.0FD: Not required (only requires LLICV daily) | 6020A: %D ≤ 30<br><br>QSM 4.2/5.0: LLCCV not required | Report any samples with failing analyte higher than mid-level (passing) CCV and rerun samples for failing element(s).<br><br>Perform instrument maintenance; if still out, reanalyze curve. |
| Continuing Calibration Blank (CCB) | After each CCV | 6020: <3xIDL<br><br>6020A: <LOQ<br><br>200.8: <2.2xMDL<br><br>QSM 4.2/5.0: <LOD | Report samples that are ND or >10x contamination; rerun all others |
| Instrument Detection Limit (IDL) Study | Per QSM 4.2: Quarterly on each instrument | C&T inhouse criteria: ≤ H2O MDL<br><br>QSM 4.2: ≤ LOD (not listed in QSM5.0) | Perform instrument maintenance and rerun |
| Method Detection Limit (MDL) Study | When new instrument installed, or significant change made to process | ≤1/3 LOQ; see MDL SOP for additional details | See MDL SOP |
| Limit of Detection (LOD) | Analyze on each instrument, quarterly (DoD QSM requirement) | Spike at 1-4x MDL and <LOQ; analytes detected | See MDL SOP |

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| | | | |
|---|---|---|---|
| Limit of Quantitation (LOQ) | Analyze on one instrument, quarterly (DoD QSM requirement) | Spike at or above (up to 2x) the LOQ. %Recovery: 50-150. | See LOQ SOP |
| Mechanical Pipettes and Reagent Dispensers | Check calibration quarterly | See QA SOP for Pipettes & Dispensors for details | See QA SOP for Pipettes & Dispensors for details |
| Performance Testing (PT) Samples | Semiannually. Drinking Water PT samples (account: QC-PE, project: ERA WS-Study) do not have to be digested but Wastewater PT samples (account: QC-PE, project: ERA WP-Study) must be digested. | Each element within study limits | Any failures must be investigated, corrected, and a new PT analyzed and passed before end of the semi-annual time-frame. |
| Demonstration of Capability (DOC) | Each new analyst; annually thereafter | Initial: 4 consecutive LCS within C&T recovery and RPD limits.<br><br>Continuing: Acceptable PT results or 4 consecutive LCS within C&T recovery and RPD limits. | Retrain analyst and reanalyze DOC. |

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5.1 *Method Modification:* C&T does not use the concept of **Linear Dynamic Range**, in which the upper limit of quantitation is established through extrapolation. Unlike ICP-AES methods (6010 and 200.7), the upper limit of the ICP-MS calibration range is highly dependent on the voltage of the electron multiplier in the detector, which may change any time maintenance is performed or the instrument tuned. For this reason, the upper end of the quantitation range must be established by a daily multi-point calibration curve. The highest calibration standard, which is at a much lower concentration than those used as the “high standard” in an extrapolation scenario, determines the top of the quantitation range for each mass/charge (m/z) ratio utilized, by determining the signal response from four different concentrations across the range.

5.2 **Interference equations** must be verified daily and updated whenever the mass axis and resolution are updated or after the detector is cleaned. The Agilent software includes auto-correct programs for the elements listed in the methods. Fine tuning may be performed during the tuning sequence.

**6.0 EQUIPMENT** (see Appendix_9 for instrument conditions):
Agilent 7500ce ICP-MS with Octopole Reaction System
Environmental Science FAST autosampler, Model SC-4DXS
0.45um x 25 mm GD/X Disposable PTFE syringe filter, VWR, Whatman # 28138-164
10 ml disposable syringe, BD syringe, VWR # BD309604

**7.0 PROCEDURE**

7.1 **Daily Maintenance**

- Replenish the rinse-water reservoir daily with acidified (1% HCl plus 1% $HNO_3$) deionized water.
- Verify that the Drain Vessel is empty. If it is not, empty the vessel and rinse it thoroughly with DI water.
- Change the Sample and Internal Standard pump windings daily. Soak Tygon Sample and Internal Standard pump windings in acidified (5%HCl plus 5%$HNO_3$) deionized water for at least 2 hours prior to use.
- Change the drain pump winding every monthly, or whenever pump winding become flattened or damaged.

7.2 **Daily Instrument Sequence**

7.2.1 Turn the plasma on.

7.2.2 Allow at least 30 minutes for the instrument to equilibrate.

7.2.3 Start the sequence by analyzing the 10 µg/L 6020 Tuning Check Standard. The instrument must meet the criteria listed in Section 5 above before analysis can continue.

7.2.4 If EPA 200.8 samples will be included in the sequence, analyze the 10 µg/L 200.8 Tuning Check Standard. The instrument must meet the criteria listed in Section 5 above before samples can be analyzed by EPA 200.8.

7.2.5 Calibrate the ICPMS by running a calibration blank followed by at least five calibration standards, in increasing order of concentration, at levels that bracket the quantitation range; the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range (see Appendix_5 for preparation of these standards).

*200.8 Method Modification:* Method 200.8 only requires a 3-point calibration however C&T standard practice is to run 5 or more points, to better establish the curve.

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7.2.6 Send the ICAL data to LIMS.

7.2.7 Review the ICAL summary to determine if the sequence can be continued:

- The correlation coefficient must meet the limits specified below for the methods being run on the day's sequence (see Section 5 for details).
- The highest concentration standard may be omitted so as long as there are at least three points remaining and the remaining highest point defines the top of the calibration range (any digests which exceed this concentration must be diluted and reanalyzed).
- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least three points remaining.
- Mid-point standards may not be omitted simply to improve the correlation coefficient. They may, however, be reanalyzed if poor aspiration is suspected. The reanalysis must occur immediately after the curve so long as no sample digests were analyzed since the last calibration standard and all elements are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular element.

7.2.8 Analyze a mid-level Initial Calibration Verification (ICV) standard, obtained from a second manufacturer, at a different concentration than the calibration standard. The result must be within the limits listed in Section 5 above, for the method being run on the day's sequence, or the instrument must be recalibrated.

7.2.9 **For Method 6020A analysis**: Analyze a CRI standard (low level ICV, LLICV) after running the initial calibration. The CRI should be prepared at the limit of quantitation (LOQ), using the same source standards as the calibration curve. The CRI should meet the limits listed in Section 5 above before any samples are analyzed for EPA 6020A. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing CRI only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing CRI if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing CRI run at a higher concentration.

7.2.10 Analyze an Initial Calibration Blank (ICB) consisting of deionized water acidified with 1% HCl and 1% $HNO_3$. The ICB must meet the criteria listed in Section 5 above for the method being run on the day's sequence.

7.2.11 Analyze the ICS-A to demonstrate that high levels of interferents are not biasing low-level quantitation. The determined concentration of the non-interferent should

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be within the limits listed in Section 5 above, except for those elements that are considered by the manufacturer to be 'trace' contaminants of the high level elements and are listed on the Certificate of Analysis.

If this standard fails acceptance criteria**,** adjust either the mass resolution, or parameters in the Hydrogen or Helium modes.

7.2.12 Analyze the ICS-AB to demonstrate that quantitation in the presence of high-level interferents is acceptably accurate. The %D of the non-interferent elements in the ICS-AB must be within the limits listed in Section 5 above. If this standard does not meet the acceptance criteria, the analysis must be terminated, the method corrected for the interference, and the analysis restarted from the Tuning Check Standard.

7.2.13 Now actual samples may be run. Collect the digests, job sheets, LIMS prep sheet, and copy of the benchbook page from the prep chemists. Sign the "Received by" line on the LIMS prep sheet to maintain internal chain-of-custody of the digests. Review the LIMS prep entry against the benchbook and when everything matches, sign off on it in LIMS.

7.2.14 Decant the digests into autosampler tubes labeled with the sample number and dilution factor. Depending on which instrument is being run, digestates may need to be diluted to compensate for salinity.

Water samples are typically analyzed at a 1:10 dilution, and soil samples at a 1:50 dilution; the dilutions should be made with acidified (1% HCl plus 1% $HNO_3$ ).

Prepare dilutions as described in Appendix 2. Don't use the auto-dilution feature on the instrument, as results obtained using this feature have shown poor agreement with additional dilutions or undiluted results obtained by optical ICP-AES.

*Note:* If any of the digests contain suspended particles, which could clog the injection tubing, filter the digests *and* the associated method blank and LCS or BS/BSD through acid-washed syringe filters prior to adding them to the sequence.

7.2.15 Nebulize each sample for 30 seconds or until a steady signal is observed, prior to collecting data, and then collect 3 integrations. The instrument will flush the system for approximately 90 seconds between samples and will go on to the next autosampler tube once the signal has stabilized.

7.2.16 Analyze a continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the sequence.

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The CCV result must be within the limits listed in Section 5 above for the method(s) being run on the day's sequence or the instrument must be recalibrated and any samples analyzed after the last passing CCV must be reanalyzed.

Target elements should not be detected in the CCB above the levels listed in Section 5 above for the method(s) needed for the samples bracketed by the CCV; any samples bracketed by a contaminated CCB and being analyzed for a detected element must be reanalyzed.

7.3.17 **For EPA Method 6020A analysis**: Analyze a low level CCV (LLCCV) following the ICV. The LLCCV should be prepared at the limit of quantification (LOQ) for the calibration, using standards from the same source as the calibration standards. The %D should be within the limits listed in Section 5 above. If any element falls outside these limits, all samples affected by the failure must be reanalyzed. An element may be reported in the event of a high failing LLCCV only if the sample result is determined to be less than the specified RL An element may also be reported in the event of a failing LLCCV if the LOQ used for the sample result is greater than or equal to the level of a passing midlevel CCV or a passing LLCCV run at a higher concentration.

*DoD QSM:* The DoD QSM v4.2/5.0 does not require the analysis of low-level calibration verifications except the opening LLICV.

7.2.18 Analyze the Tune Check Standard, ICS-A, and ICS-AB at the beginning of every 12-hour shift. The standards must pass the acceptance criteria listed in Section 5 above or any standards analyzed after the failing standard must be reanalyzed.

7.2.19 Analyze the ICS-A, and ICS-AB at the end of every 12-hour shift. The standards must pass the acceptance criteria listed in Section 5 above or any samples analyzed after the last passing set of ICS standards must be reanalyzed.

7.2.20 **Analytical Sequence:** A typical analytical sequence looks like the following:

Tuning Solution
200ppb Pulse/Analog Solution (or 50ppb for MET06)
10ppb Tune Check Standard
Initial Calibration Blank
Initial Calibration Standards (0.25, 0.5, 1, 10, 100, 200 ug/L)
Initial Calibration Verification (ICV)
Initial Calibration Blank (ICB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)
Method Blank (MB)
Blank Spike (BS)
Blank Spike Duplicate (BSD)
QC Sample (MSS)
Matrix Spike (MS)

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Matrix Spike Duplicate (MSD)
Serial Dilution (SER)
.. 3 more samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
…more samples and CCV's (until 12-hour tune clock runs out)
Tune Check Standard
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)
.. 10 samples ..
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
… etc.
Continuing Calibration Verification Standard (CCV)
Continuing Calibration Blank (CCB)
Interference Check Standard A (ICSA)
Interference Check Standard A/B (ICSAB)

The sequence must end with a CCV, CCB, ICS-A, and ICS-AB regardless of the number of samples that have been analyzed.

After the sequence is complete, the Agilent software will automatically turn off the instrument.

LIMs automatically analyzes 2 ICBs and CCBs and always chooses the second run, even when specific elements fail blank criteria, we continue running samples for passing elements. Corrective action is taken when the analyst judges it to be warranted.

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7.3 **Quantitative Analysis**

The Agilent ICP-MS automatically adds internal standard, which helps compensate for viscosity and transport interferences. The sample is then transported through the nebulizer and vaporized in the plasma. The spectrometer measures the mass-to-charge ratio (m/z) of the resulting ions. For all samples and standards, the instrument collects information for 3 integrations and reports the average intensity of these integrations, along with the %RSD between the integrations. The RSD between the integrations must be $\leq$ 20% or the sample must be reanalyzed. If RSD for the reanalysis is again greater than 20%, report the analysis with the lowest RSD and narrate the problem on the Data Review Checklist; LIMS will apply a qualifier flag to reported result on the final form.

*Method Modification Note:* Methods 6020, 6020A, and 200.8 do not specify a maximum RSD, so C&T has chosen 20% as the RSD limit to standardize ICP criteria.

Quantitation is based on comparison of the intensity of the target element and internal standard to the initial calibration curve for that element, with adjustments for the sample preparation concentration factor and instrument dilution factor. See Appendix_11 for example calculations. Concentrations are expressed as micrograms per liter (µg/L) or milligrams per kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

7.3.1 **Evaluate the Internal Standard Recoveries**

Internal standards are added to the samples and standards at the time of analysis by a second on-line channel of the peristaltic pump. See Appendix_4 for internal standard assignment and comments.

The internal standard recoveries are determined by dividing the intensity observed in the sample by that observed in the first Initial Calibration Standard in the ICAL, which is a zero standard. The internal standard recoveries associated with all required/ reported elements should be within the recovery limits listed in Section 5.0 for the method required for each sample, or the sample should be diluted and reanalyzed.

The CCV and CCB internal standard recoveries associated with all reported sample results must be within limits required for the methods requested for the samples bracketed by the CCV/CCB, or the instrument must be recalibrated and any samples analyzed after the last passing CCV reanalyzed. Note: If an internal standard fails but is not assigned to any of the requested elements for that sample, the sample does not have to be reanalyzed.

7.3.2 **Evaluate the CCV Results**

The concentration of the CCV must be within the calibration range, with %D within the limits listed in Section 5 above. If the %D for any element is outside this acceptance window, LIMS will use the following to determine if the associated results are reportable:

a. If the failing element is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b. If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

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c. If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

d. If the compound fails the %D criterion due to a low response, the sample must be reanalyzed as the low bias may result in false negatives or misquantitation.

7.3.3 **For 6020A-only, Evaluate the LLCCV Results**:
The LLICV recovery should be within the method-specified limits (see Section 5 above) for any target elements, otherwise:

a. If a high bias is observed, samples bracketed by the failing standard may be reported if that element was not detected above the LOQ; concentrations between the MDL and LOQ may be reported with a J-flag (estimated).

b. If a high recovery is observed and that element is found in the associated sample(s) at levels greater than or equal to the concentration in the associated, passing, mid-level CCV, the data may be reported.

c. Any other samples being analyzed for the failing element must be reanalyzed.

7.3.4 **Evaluate the CCB Results**
Target elements should not be detected in the calibration blank at any level greater than 3 times the Instrument Detection Limit. If target elements are detected, use the following to determine if the associated results are reportable:

a. If the detected element is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b. If the detected element was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c. If the detected element *was* also detected in any of the associated samples at levels above the reporting limit but the sample concentrations were greater than 10 times the concentration in the instrument blank, the samples may be reported.

d. If the detected element *was* also detected in any of the associated samples at levels between the reporting limit and 10 times the reporting limit, the samples must be reanalyzed.

7.3.5 **Evaluate the Batch QC Results**

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7.3.5.1 **Method (Prep) Blank:** Target analytes should not be detected in the method blank at any level above that listed in Section 5 for the method required for the batch being run, or the blank should be reanalyzed. If reanalysis confirms the contamination, use the following steps to determine if the sample results may be reported:

a. If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

b. If the target element(s) found in the blank was not detected in the associated samples, the data may be reported and the problem narrated.

c. If the target element(s) found the method blank was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If the target element(s) detected in the blank were also detected in the associated samples, but at levels less than 10x the level in the blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-batched and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-digestion can begin within the clients requested turn-around time, if necessary.

7.3.5.2 **Blank Spike (BS) and Blank Spike Duplicate (BSD):** The recoveries and RPD should fall within the limits listed in Section 5 for the methods needed for the batch being run. If these limits are not met, the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine if the sample results may be reported:

a. If the samples are being analyzed for only a subset of the target element list (ie: lead only, LUFT 5, etc.) and those elements all pass acceptance criteria, the data may be reported without further corrective action.

b. If a high recovery is observed but that element was not detected in the associated samples, note the failure on the Data Review Checklist and report the data without re-digestion, as the potential high bias does not affect the sample results.

c. If a high recovery is observed and the samples contain that element at levels above the reporting limits, the samples containing that element must be re-digested.

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d. If a high RPD is observed but the recoveries are within acceptance limits and the samples do not contain that element, note the failure on the Data Review Checklist and report the data without re-digestion, as the lack of good precision data does not affect ND samples.

e. If a high RPD is observed and the samples contain that element at levels above the reporting limits, those samples containing that element must be re-extracted.

7.3.5.3 **Matrix QC (SSPIKE/SDUP or MS/MSD):** The recoveries and RPD should fall within the limits listed in Section 5 for the methods needed for the batch being run. If these limits are not met, the samples associated with it may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

a. If the concentration of a target element in the sample is greater than the linear range and the sample needs to be rerun for just that compound, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries, without reanalysis.

b. If the concentration of a target element in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

c. If the concentration of a target element is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

*Note:* If the concentration of a target compound is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis (except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

7.3.5.4 **Serial Dilutions:** Analysis of a 5x dilution should agree within the limits listed in Section 5 if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference, such as viscosity of the digestate, should be suspected. If for a given

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project, all elements repeatedly show an increased response in the Serial Dilution, when compared to the initial run, discuss the problem with the Department Manager and/or the Project Manager for that account, as the samples may require routine dilution to overcome viscosity effects.

7.3.5.5 **Post Digest Spikes:** An analyte spike added to a portion of a prepared sample digest, or its dilution, should be recovered to within the criteria listed in Section 5 above. The spike addition should produce a minimum level of 10 times and a maximum level of 100 times the instrumental detection limit. If the recoveries of all elements show a uniform bias in either direction, make and analyze a new spike to verify that it was prepared correctly. If the spike is not recovered within the specified limits a matrix effect should be suspected and the sample should be diluted (to dilute out the matrix interference).

7.3.6 **Evaluate the Sample Results**

Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

Examine the sample results to verify that all requested elements are reported on the internal user report and that the results are within the linear range. If the concentration of any requested target element is greater than the highest calibration standard for that element, use volumetric pipettes to prepare a dilution of the digestate so that the highest target element is in the upper half of the calibration range.

If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

Examine the sample results to verify that the RSD between exposures is less than 20%. Any sample result with a duplicate exposure RSD greater than 20% must be reanalyzed. If the RSD is still greater than 20%, report the exposure with the lower %RSD. Any sample with requested element concentrations above the linear range must be diluted and reanalyzed.

*Method Modification Note:* Methods 6020, 6020A, and 200.8 do not specify a maximum RSD, so C&T has chosen 20% as the RSD limit to standardize ICP criteria.

7.4 **Assemble the Data Package**

7.4.1 After all samples and necessary dilutions have been analyzed, print the final report forms to the printer designated “Nowhere”; this will send the final reporting forms to LIMS.

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7.4.2 Review these forms to make sure that the correct results were reported and that there are no elements marked “N/A” (results “not available”).

7.4.3 Verify that the prep sheets are complete and signed, that all necessary sample information is present (see section below for details), then complete and sign the “Data Review Checklist”.

7.4.4 Submit the data package to the Department Manager or QC Chemist for second-party review. Any changes made by the second-party reviewer must be individually signed by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative and sign the completed checklist.

## 8.0 DOCUMENTATION REQUIREMENTS

a. **Instrument Raw Data**
The raw ICP-MS data is directly captured by LIMS; no hardcopy data is printed or filed.

b. **Sample Prep Documentation**
A copy of the digestion benchbook page for the sample digestion must be scanned into LIMS for inclusion in final data packages and archiving. The digestion benchbook entries should include:

- C&T sample ID's and unique container identifier,
- date of sample digestion, initial volume or weight of sample, and final digestate volume,
- identity of QC samples (spikes, duplicates & LCS),
- amount of spikes added and LIMS identification numbers of all spiking solutions,
- a list of all reagents used (C&T ID or manufacturer and lot number),
- indication of whether or not the digests were filtered after digestion,
- any unusual occurrences observed during the digestion procedure

## 9.0 POLLUTION PREVENTION

Prepare only sufficient standard and reagent volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

## 10.0 WASTE DISPOSAL

All digests are kept for at least 6 months prior to disposal. After 6 months, the digests are included in the ‘Corrosives’ waste stream. Expired standards should also be discarded into the ‘Corrosives’ waste stream.

## 11.0 REVISION HISTORY

The previous document (revision 9) was changed as follows:

- Cover page and numbering were reformatted
- Section 2: Updated references

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- Section 3: Added bottle guidance. Updated storage temperature conditions to match current TNI/NELAP and 40CFR136 guidance. Added additional note that samples submitted for lab filtration will have comment regarding 15 minute “holding time” for filtration added to the case narrative.
- Section 5: Reformatted for ease of use. Clarified differences between requirements for various methods and guidance documents. Added QSM 5.0 requirements. Added 40CFR136 required items plus other commonly referenced criteria.
- Section 6: Updated equipment list
- Section 7: Replaced specific acceptance criteria with reference to Section 5, to reduce opportunities for conflicting criteria. Moved the pulse/analog program notes to Appendix_10, as this is required after maintenance but not on a daily basis. Minor changes associated with paperless reporting. Removed reference to outdated USACE exposure RSD requirement.
- Section 8: Removed list of Level 2/ Level 3/ Level 4 data packages, as this is now all handled by LIMS.
- Appendix_1: Updated method modifications note to include current methods (EPA 6020A and 200.8-Rev.5.4)
- Appendix_3: Added MET-26 to instruments listed.
- Appendix_4: Added MET-26 to instruments listed. Added 6020A list of internal standards
- Appendix_5: Added requirement to scan standard Certificate of Analysis and prep log entries into LIMS. Added LLICV/CRI information; updated other standards information to match current practice.
- Appendix_6: Aligned 200.8 and 6020/6020A tuning criteria to include 5 exposures. Corrected 200.8 Tune Check resolution acceptance criteria to match method. Updated tuning frequency from daily to ‘as needed’; tune check remains required beginning of every 12 hour shift. Removed initial calibration and calibration verification discussion and criteria, as those are listed in the main body of the procedure and in Section 5; renamed section appropriately.
- Appendix_12: Deleted this appendix, as the information is now incorporated into Section 5 and Appendix_5

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## APPENDIX_1: ELEMENTS & STANDARD REPORTING LIMITS

### ICP-MS Metals
### EPA 6020/ EPA 6020A/ EPA 200.8

| CAS # | | Element | Reporting Limit* | | CAS # | | Element | Reporting Limit | |
|---|---|---|---|---|---|---|---|---|---|
| | | | µg/L | mg/Kg | | | | µg/L | mg/Kg |
| 7429-90-5 | Al | Aluminum | 50 | 10 | 7439-95-4 | Mg | Magnesium | 50 | 10 |
| 7440-36-0 | Sb | Antimony | 1 | 0.25 | 7439-96-5 | Mn | Manganese | 1 | 0.25 |
| 7440-38-2 | As | Arsenic | 1 | 0.25 | 7439-98-7 | Mo | Molybdenum | 1 | 0.25 |
| 7440-39-3 | Ba | Barium | 1 | 0.25 | 7440-02-0 | Ni | Nickel | 1 | 0.5 |
| 7440-41-7 | Be | Beryllium | 1 | 0.25 | 7440-09-7 | K | Potassium | 50 | 25 |
| 7440-43-9 | Cd | Cadmium | 1 | 0.25 | 7782-49-2 | Se | Selenium | 1 | 0.25 |
| 7440-70-2 | Ca | Calcium | 50 | 10 | 7440-22-4 | Ag | Silver | 1 | 0.25 |
| 7440-47-3 | Cr | Chromium | 1 | 0.50 | 7440-23-5 | Na | Sodium | 50 | 25 |
| 7440-48-4 | Co | Cobalt | 1 | 0.25 | 7440-28-0 | Tl | Thallium | 1 | 0.25 |
| 7440-50-8 | Cu | Copper | 1 | 0.25 | 7440-62-2 | V | Vanadium | 1 | 0.25 |
| 7439-89-6 | Fe | Iron | 50 | 10 | 7440-66-6 | Zn | Zinc | 1 | 1.0 |
| 7439-92-1 | Pb | Lead | 1 | 0.25 | | | | | |

***Method Modification:*** C&T includes the elements listed in 6020 (1994) plus the following elements that were added to the 6020A (2007): Ca, Fe, Mg, K, Se, Na, and V. With the exception of Se, the same elements were not included in 200.8 (1994):

### Low Level ICP-MS Metals
### EPA 6020 (by special request)

| CAS # | | Element | Reporting Limit* | | CAS # | | Element | Reporting Limit* | |
|---|---|---|---|---|---|---|---|---|---|
| | | | µg/L | mg/Kg | | | | µg/L | mg/Kg |
| 7429-90-5 | Al | Aluminum | 50 | 10 | 7439-95-4 | Mg | Magnesium | 50 | 10 |
| 7440-36-0 | Sb | Antimony | 0.25 | 0.25 | 7439-96-5 | Mn | Manganese | 0.25 | 0.25 |
| 7440-38-2 | As | Arsenic | 0.5 | 0.25 | 7439-98-7 | Mo | Molybdenum | 0.5 | 0.25 |
| 7440-39-3 | Ba | Barium | 0.25 | 0.25 | 7440-02-0 | Ni | Nickel | 0.25 | 0.5 |
| 7440-41-7 | Be | Beryllium | 0.25 | 0.25 | 7440-09-7 | K | Potassium | 50 | 25 |
| 7440-43-9 | Cd | Cadmium | 0.25 | 0.25 | 7782-49-2 | Se | Selenium | 0.5 | 0.25 |
| 7440-70-2 | Ca | Calcium | 50 | 10 | 7440-22-4 | Ag | Silver | 0.25 | 0.25 |
| 7440-47-3 | Cr | Chromium | 0.5 | 0.50 | 7440-23-5 | Na | Sodium | 50 | 25 |
| 7440-48-4 | Co | Cobalt | 0.25 | 0.25 | 7440-28-0 | Tl | Thallium | 0.5 | 0.25 |
| 7440-50-8 | Cu | Copper | 0.5 | 0.25 | 7440-62-2 | V | Vanadium | 1 | 0.25 |
| 7439-89-6 | Fe | Iron | 50 | 10 | 7440-66-6 | Zn | Zinc | 1 | 1.0 |
| 7439-92-1 | Pb | Lead | 0.25 | 0.25 | | | | | |

* These lower reporting limits may be analyzed upon special request and may only be used if the current MDL is <1/3 the requested RL and the lowest ICAL is at or below the requested RL.

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**APPENDIX_2: SAMPLE DILUTIONS**

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using autopipettes, dilute the following volumes of sample digestate in a disposable centrifuge tube; adjust the acid concentration as needed so that the final acid concentration in the diluted aliquot is 1% $HNO_3$ and 1% HCl. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

**DILUTIONS**

| Instrument Dilution Factor (IDF) | Digestate Volume | Add Volume (mL) Acidified DI Water | Final Volume (mL) |
|---|---|---|---|
| 2 | 5.0 mL | 5.0 | 10 |
| 5 | 2.0 mL | 8.0 | 10 |
| 10 | 1.0 mL | 9.0 | 10 |
| 20 | 0.50 mL | 9.5 | 10 |
| 50 | 0.20 mL | 9.8 | 10 |
| 100 | 0.10 mL | 9.9 | 10 |

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

**SERIAL DILUTIONS**

| Instrument Dilution Factor | Using Primary Dilution | Volume of Primary (100x) Dilution | Add Volume (mL) Acidified DI Water | Final Volume (mL) |
|---|---|---|---|---|
| 500 | 100x | 2.0 mL | 8.0 | 10 |
| 1,000 | 100x | 1.0 mL | 9.0 | 10 |
| 2,000 | 100x | 0.5 mL | 9.5 | 10 |
| 5,000 | 100x | 0.2 mL | 9.8 | 10 |
| 10,000 | 100x | 0.1 mL | 9.9 | 10 |

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**APPENDIX_3: QUANTITATION ISOTOPES**

| **Element** | Quantitation Isotope | Secondary Isotopes |
|---|---|---|
| Aluminum | 27 | |
| Antimony | 121 | |
| Arsenic | 75 | 77 [a], 83 [a] |
| Barium | 137 [**] | 135 |
| Beryllium | 9 | |
| Bismuth (IS) | 209 | |
| Cadmium | 111 [**] | 106 [a], 108 [a], 114 |
| Calcium | 44 | 43, 88 [a] |
| Chlorine [a] | | 37 [a], (77, 82) [a] |
| Chromium | 52 | 53, 54 |
| Cobalt | 59 | |
| Copper | 65 | 63 [**] |
| Erbium (dilution std) [e] | 166 | |
| Germanium (IS) * | 72 | |
| Indium (IS) | 115 | 113, 118 [a] |
| Iron [f] | 56 | 57, 54, 58 |
| Lead [b] | 208, 207, 206 | 204 |
| Lithium (IS) | 6 | |
| Magnesium | 24 | 25, 26 |
| Manganese | 55 | |
| Molybdenum | 98 | 97, 96, 92, 94, (108), 99[a] |
| Nickel | 60 | 58, 62, 61, 64 |
| Potassium | 39 | |
| Scandium (IS) | 45 | |
| Selenium [c f] | 78 | 80, 78, 76, 77, 74 |
| Silver | 107 | 109 |
| Sodium | 23 | |
| Terbium (IS) | 159 | |
| Thallium | 205 | |
| Thorium [d] | 232 | |
| Tin | 118 | |
| Uranium [d] | 238 | |
| Vanadium [c] | 51 | 238, 50 |
| Yttrium (IS) | 89 | |
| Zinc | 66 | 64, 68, 67, 70 |

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*Method Modifications:*
The internal standard Germanium (*) is not listed in either 6020 or 200.8 but is recommended by the instrument manufacturer to improve the recovery of the difficult-to-ionize elements (As, Cd, Se, Zn) in the presence of high concentrations of easily ionized elements such as Na and K. Germanium is also included in 6020A.

A double-asterisk or underscore indicates the isotope suggested by the method by 200.8 (**) or 6020 (_) that is not used by C&T for quantitation.

[a] These masses are used for interference correction.

[b] Lead is quantitated using the sum of m/z 206, 207, and 208 to compensate for any differences in the abundances of these isotopes between samples and standards. The Agilent software includes the calculation based on all three isotopes, but reports them as mass 208.

[c] Target analyte listed in 6020A (Rev.1, 2007) but not in 6020 (Rev.0, 1994).

[d] Target analyte listed in 200.8 but not in either version of 6020.

[e] Erbium is the dilution standard, used by the data system to apply the correct instrument (auto-) dilution factor.

[f] Agilent 7500CE instruments (MET-06, MET-16, MET-26): Hydrogen is used in the octopole to eliminate ArAr (m/z 78) and ArO (m/z 56) interference on masses 56 (Iron) and 78 (Selenium).

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## APPENDIX_4: INTERNAL STANDARD ASSIGNMENTS

The isotope used for target element quantitation must be within 50 mass units of the associated internal standard (6020 Section 5.4).

### AGILENT 7500CE (MET-06, MET-16, MET-26)

| Lithium (6) | Scandium (45) | Germanium (72) | Yttrium (89) |
|---|---|---|---|
| Beryllium | Aluminum | Arsenic | Molybdenum |
| | Calcium | Copper | |
| | Chromium | Selenium | |
| | Cobalt | Zinc | |
| | Iron | | |
| | Magnesium | | |
| | Manganese | | |
| | Nickel | | |
| | Potassium | | |
| | Sodium | | |
| | Vanadium | | |

| Indium (115) | Terbium (159) | Bismuth (209) |
|---|---|---|
| Antimony | Barium | Thallium |
| Cadmium | Iridium | |
| Erbium (dilution std) | Lead | |
| Palladium | Platinum | |
| Rhodium | | |
| Ruthenium | | |
| Silver | | |

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*Notes:*
The lithium is enriched with $^{6}$Li to reduce interferences from native lithium in the samples. The other internal standards are elements that are not typically encountered in environmental samples.

*Method Modification:* Curtis & Tompkins uses the internal standards recommended by the instrument manufacturer (Agilent). This mix meets the method criterion that the isotope used for sample quantitation is within 50 amu of the associated internal standard, and improves method performance by reducing matrix interferences.

| | |
|---|---|
| C&T ISTD List: | $^{6}$Li, $^{45}$Sc, $^{72}$Ge, $^{115}$In, $^{159}$Tb, $^{209}$Bi |
| 6020 Recommends: | $^{6}$Li, $^{45}$Sc, $^{89}$Y, $^{103}$Rh, $^{115}$In, $^{159}$Tb, $^{165}$Ho, $^{209}$Bi |
| 6020A Recommends: | $^{6}$Li, $^{45}$Sc, $^{74}$Ge, $^{89}$Y, $^{103}$Rh, $^{115}$In, $^{159}$Tb, $^{165}$Ho, $^{209}$Bi |
| 200.8 Recommends: | $^{6}$Li, $^{45}$Sc, $^{89}$Y, $^{103}$Rh, $^{115}$In, $^{159}$Tb, $^{165}$Ho, $^{175}$Lu, $^{209}$Bi |

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## APPENDIX_5: REAGENTS & STANDARDS PREPARATION

The standards and reagents listed below are those in use at the time this procedure was written. Alternate supplies may be used so long as they are of equivalent quality and all calibration, quality control, and traceability requirements are met.

### REAGENTS

The preparation of all liquid or solid reagents, including dilutions into DI water, must be documented in the reagent prep benchbook. Each reagent is assigned a unique ID, based on the manufacturer and the date prepared. This ID is then recorded in the digestion benchbook each time the reagent is used.

Label each reagent with the reagent ID, concentration, prep date, and expiration date. All reagents should be prepared and stored in freshly cleaned glassware. Expired, discolored, or contaminated reagents should be discarded and the bottle cleaned before reuse.

Deionized water, ASTM Type II (ASTM D1193)
Argon, Purity: 99.99%
Air, C&T house compressed air
He, compressed, Purity: 99.9999%
$H_2$, compressed, Purity: 99.9999%

Aqua Regia: *prepare daily*

Prepare immediately before use in a glass bottle by adding 3 volumes of concentrated HCl to one volume $HNO_3$, typically preparing about 125mL aqua regia per batch ( 32mL HCl + 96mL $HNO_3$ => 128mL total ). Aqua Regia must be prepared daily.

Nitric Acid ($HNO_3$), concentrated, InstraAnalyzed grade

JT Baker catalog # 7697-37-2
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to two years.

Hydrochloric Acid (HCl), concentrated, InstraAnalyzed grade

JT Baker catalog # 7647-01-0
Store unopened bottles in the corrosives cabinet and open bottles under the fume hood for up to two years.

### STANDARDS

All source standards must be NIST-traceable and be documented in LIMS upon receipt, through the Standards Menu. The LIMS S-name is unique to the vendor that the source is obtained from; if a source standard is obtained from a different vendor, a new S-name must be assigned and the information entered in the “Standard Entry” table before the standard can be assigned a unique S# (standard number).

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The LIMS Standard Maintenance database includes the catalog#, lot#, expiration date, and concentration of the standards as they are received from the vendor. Write the S# and the date received on both the standard vial and on the 'Certificate of Analysis' that accompanied the standard, then scan the Certificate into LIMS. If the supplier did not provide a certificate, call and request that a copy be faxed to C&T.

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Document the preparation in the standards benchbook. Enter the prep information into LIMS through the "Standard Maintenance" menu; LIMS will then assign a unique S# to that standard. Write the LIMS S# and expiration date in the benchbook along with the prep information then scan the benchbook entry into LIMS. Label the standard vial with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

If the Certificate of Analysis or bottle label did not include an expiration date, assign an expiration date of one year from the date received.

Store standards at room temperature, away from light, to prevent photo-induced precipitation of silver. Working standards expire after 90 days except ICS-AB, which must be made weekly (Method 6020 requires a short expiration time because silver falls out of solution).

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**CALIBRATION BLANKS**

Prepare calibration blanks (ICB/ CCB) daily by adding 1.0 mL concentrated $HNO_3$ and 1.0 mL HCl to DI water and diluting to 100 mL.

**INTERNAL STANDARD SOLUTION**

Source Standard (10ppm $^{6}Li$, $^{45}Sc$, $^{72}Ge$, $^{89}Y$, $^{115}In$, $^{159}Tb$, $^{209}Bi$)
Agilent Catalog# 5183-4681

1.0 µg/mL (ppm) Working Standard: Make a 1:10 dilution of the Source Standard in 1%HCl and 1%$HNO_3$

**TUNE SOLUTION**

The Tune Solutions are made from the following source standards containing individual elements at 1,000µg/mL:

| | |
|---|---|
| Ce | Cerium, SepxCertiprep Catalog # PLCE2-2X |
| Co | Cobalt, SepxCertiprep Catalog # PLCO1-2X |
| Li | Lithium, SpexCertiprep Catalog # PLLI1-2X |
| Tl | Thallium, SpexCertiprep Catalog # PLTL2-2X |
| Y | Yttrium, SpexCertiprep Catalog # PLY2-2X |

**Agilent 7500CE instruments (MET-06, MET-16, MET-26)**

10 µg/L Ce, Co, Li, Tl, Y – Final Concentration

Make an acidified (1% $HNO_3$ plus 1% HCl) 10,000 µg/L intermediate:

1.) Add about 500mL deionized water to a 1L Class-1 volumetric flask.
2.) Carefully measure 10mL of nitric acid into the volumetric flask then swirl to mix.
3.) Measure 10mL of hydrochloric acid into the volumetric flask then swirl to mix.

4.) Using an autopipette, add 10mL of each (Ce, Co, Li, Tl, Y) 1,000 µg/mL standard to the flask.
5.) Swirl to mix then fill to the mark with DI water.
6.) Cap & invert 3 times to mix, allowing sufficient time for mixing with each inversion.

From this intermediate, make an acidified 10 µg/L solution:

7.) Add about 500mL deionized water to a second 1L Class-1 volumetric flask.
8.) Carefully measure 10mL of nitric acid into this volumetric flask then swirl to mix.
9.) Measure 10mL of hydrochloric acid into this volumetric flask then swirl to mix.
10.) Using an autopipette, add 1.0 mL of the 10,000 µg/L intermediate to the flask
11.) Swirl to mix then fill to the mark with DI water.
12.) Cap & invert 3 times to mix, allowing sufficient time for mixing with each inversion.
13.) Store at room temperature for up to 1 year.

**TUNING CHECK STANDARDS**

6020 / 6020A Source Standard: 10,000 µg/L of Li, Co, In, Tl
LIMS S-Name: 6020TS
Inorganic Ventures Cat.# 6020TS

6020 / 6020A Working Standard: 10 µg/L of Li, Co, In, Tl
LIMS S-Name: ICPMS TUNE
Dilute 0.05 mL of the 6020TS source standard into 0 mL of acidified (1% HNO3 plus 1% HCl) deionized water. This standard expires after 3 months.

200.8 Source Standard: 10,000 µg/L of Li, Co, In, Tl
LIMS S-Name: 2008TS
Inorganic Ventures Cat.# 2008TS

6020 / 6020A Working Standard: 10 µg/L of Li, Co, In, Tl
LIMS S-Name: 200.8 TUNE
Dilute 0.05 mL of the 200.8 source standard into 50 mL of acidified (1% HNO3 plus 1% HCl) deionized water. This standard expires after 3 months.

**CALIBRATION STANDARDS**

**Source Standards:**

CS-CT02 1,000 µg /mL of Al, Ca, Fe, Mg, K, Na
CPI Cat # 4400-10829AM02 10 ug/mL of Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, V, Zn
5 µg /mL of Tl

CS-CT03 100 µg /mL of B, Sn, Ti
CPI Cat # 4400-10829AM01 10 µg /mL of Sb, Mo

**Calibration Working Standards:** Dilute the following volumes of the source standard (ICS) or the indicated working standard into 50mL acidified (1% HCl plus 1% $HNO_3$) deionized water to make standards at the listed levels. These calibration standards expire after 90 days.

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| | Element | ICPMS ICS 1 (µg/L) | ICPMS ICS 2 (µg/L) | ICPMS ICS 3 (µg/L) | ICPMS ICS 4 (µg/L) | ICPMS ICS 5 (µg/L) | ICPMS ICS 6 (µg/L) |
|---|---|---|---|---|---|---|---|
| Volume (mL) of Source | | 0.050 ICS5 (1000x) | 0.25 ICS5 (200x) | 0.50 ICS5 (100x) | 5.0 ICS5 (10x) | 0.5 CS-CT02/03 (100x) | 1.0 CS-CT02/03 (50x) |
| Al | Aluminum | 10. | 50 | 100 | 1,000 | 10,000 | 20,000 |
| Sb | Antimony | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| As | Arsenic | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Ba | Barium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Be | Beryllium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| B | Boron | 1.0 | 5.0 | 10. | 100 | 1,000 | 2,000 |
| Cd | Cadmium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Ca | Calcium | 10. | 50 | 100 | 1,000 | 10,000 | 20,000 |
| Cr | Chromium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Co | Cobalt | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Cu | Copper | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Fe | Iron | 10. | 50 | 100 | 1,000 | 10,000 | 20,000 |
| Pb | Lead | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Mg | Magnesium | 10. | 50 | 100 | 1,000 | 10,000 | 20,000 |
| Mn | Manganese | 0.1 | 0.50 | 1.0 | 10. | 100 | 200 |
| Mo | Molybdenum | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Ni | Nickel | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| K | Potassium | 10. | 50. | 100. | 1,000 | 10,000 | 20,000 |
| Se | Selenium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Ag | Silver | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Na | Sodium | 10. | 50. | 100 | 1,000 | 10,000 | 20,000 |
| Tl | Thallium | 0.05 | 0.25 | 0.50 | 5.0 | 50. | 100 |
| Sn | Tin | 1.0 | 5.0 | 10. | 100 | 1,000 | 2,000 |
| Ti | Titanium | 1.0 | 5.0 | 10. | 100 | 1,000 | 2,000 |
| V | Vanadium | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |
| Zn | Zinc | 0.10 | 0.50 | 1.0 | 10. | 100 | 200 |

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**ICV / CCV**

**Source Standards:**

| | |
|---|---|
| ICVMS4A<br>Inorganic Ventures<br>Catalog# CTL-CAL-4A | 1,000 µg/mL of Al, Ca, Fe, Mg, K, Na<br>10 µg/mL of Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, V, Zn<br>5 µg/mL of Tl |
| ICVMS2A<br>Inorganic Ventures<br>Catalog# CTL-CAL-5 | 100 µg/mL of B, Sn, Ti<br>10 µg/mL of Mo, Sb |
| IV CA<br>Inorganic Ventures<br>Catalog# CGCA1-1 | 1,000 µg/mL Calcium |
| IV MG<br>Inorganic Ventures<br>Catalog# CCMG1-1 | 1,000 µg/mL Magnesium |
| IV K<br>Inorganic Ventures<br>Catalog# CGK1-1 | 1,000 µg/mL Potassium |
| IV NA<br>Inorganic Ventures<br>Catalog# CGNA1-1 | 1,000 µg/mL Sodium |
| IV ZN<br>Inorganic Ventures<br>Catalog# CGZN1-1 | 1,000 µg/mL Zinc |

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**ICV/CCV Working Standards:** Use acidified (1% HCl plus 1% $HNO_3$) deionized water to make the standards below:

| Working Standard & Conc. | WS Conc. (µg/mL) | Final Vol (mL) | Using "Source" Std | Add Vol (mL) Source Std | LIMS S-Name | Shelf Life |
|---|---|---|---|---|---|---|
| Mid-level ICV | See table below | 50 | ICVMS2A | 0.50 | 6020ICV1 | 30 days |
| | | | ICVMS4A | 0.50 | | |
| | | | | | | |
| Mid-level CCV | See table below | 50 | ICVMS2A | 0.50 | 6020CCV1 | 30 days |
| | | | ICVMS4A | 0.50 | | |
| | | | | | | |
| Intermediate for Low Level ICV Prep | See table below | 50 | CS-CT02 | 0.50 | LLICV STOCK | 180 days |
| | | | CS-CT03 | 0.50 | | |
| | | | IV CA | 2.0 | | |
| | | | IV K | 2.0 | | |
| | | | IV MG | 2.0 | | |
| | | | IV NA | 2.0 | | |
| | | | IV ZN | 0.02 | | |
| | | | | | | |
| Low Level ICV (CRI) | See table below | 50 | LLIVC STOCK | 0.50 | CRI6020A | 30 days |

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## Calibration Verification Levels

| | LIMS Name: | 6020ICV1 (µg/L) | 6020CCV1 (µg/L) | LLICV STOCK (µg/L) | CRI6020A (µg/L) |
|---|---|---|---|---|---|
| Ag | Silver | 100 | 100 | 100 | 1.0 |
| Al | Aluminum | 10,000 | 10,000 | 10,000 | 100 |
| As | Arsenic | 100 | 100 | 100 | 1.0 |
| B | Boron | 1,000 | 1,000 | 1,000 | 10. |
| Ba | Barium | 100 | 100 | 100 | 1.0 |
| Be | Beryllium | 100 | 100 | 100 | 1.0 |
| Ca | Calcium | 10,000 | 10,000 | 50,000 | 500 |
| Cd | Cadmium | 100 | 100 | 100 | 1.0 |
| Co | Cobalt | 100 | 100 | 100 | 1.0 |
| Cr | Chromium | 100 | 100 | 100 | 1.0 |
| Cu | Copper | 100 | 100 | 100 | 1.0 |
| Fe | Iron | 10,000 | 10,000 | 10,000 | 100 |
| K | Potassium | 10,000 | 10,000 | 50,000 | 500 |
| Mn | Manganese | 100 | 100 | 100 | 1.0 |
| Mg | Magnesium | 10,000 | 10,000 | 50,000 | 500 |
| Mo | Molybdenum | 100 | 100 | 100 | 1.0 |
| Na | Sodium | 10,000 | 10,000 | 50,000 | 500 |
| Ni | Nickel | 100 | 100 | 100 | 1.0 |
| Pb | Lead | 100 | 100 | 100 | 1.0 |
| Sb | Antimony | 100 | 100 | 100 | 1.0 |
| Se | Selenium | 100 | 100 | 100 | 1.0 |
| Sn | Tin | 1,000 | 1,000 | 1,000 | 10. |
| Ti | Titanium | 1,000 | 1,000 | 1,000 | 10. |
| Tl | Thallium | 50 | 50 | 50 | 0.5 |
| V | Vanadium | 100 | 100 | 100 | 1.0 |
| Zn | Zinc | 100 | 100 | 500 | 5.0 |

*Note:* Although the mid-level ICV and CCV are identical in composition, they are prepared and assigned separately because LIMS applies different rules to the instrument sequence.

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**ICS-A and ICS-AB**

**Stock Standards:**
Inorganic Ventures, Catalog# 6020ICS-9A, contains interferent elements
Inorganic Ventures, Catalog# 6020ICS-9B, contains other elements

**Working Standards:** Prepare monthly by diluting the source standards or more frequently if low silver recoveries are observed.

ICS-A (LIMS Name: ICPMSICSA):
Using a 50mL volumetric flash, dilute 1.0mL HCl, 1.0 mL $HNO_3$ and 5.0 mL of the 6020ICS-9A source standard in deionized water.

ICS-AB (LIMS Name: ICPMSICSAB):
Using a 50mL volumetric flask, dilute 1.0mL HCl, 1.0 mL $HNO_3$ and 5.0 mL of the 6020ICS-9A source standard and 0.05 mL of the 6020ICS-9B source standard in deionized water.

| Element | | ICPMSICSA (mg/L) | ICPMSICSAB (mg/L) |
|---|---|---|---|
| Aluminum | Al | 100 | 100 |
| Calcium | Ca | 300 | 300 |
| Carbon | C | 200 | 200 |
| Chloride | Cl | 2,121.5 | 2,121.5 |
| Iron | Fe | 250 | 250 |
| Magnesium | Mg | 100 | 100 |
| Molybdenum | Mo | 2.0 | 2.0 |
| Phosphorous | P | 100 | 100 |
| Potassium | K | 100 | 100 |
| Sodium | Na | 250 | 200 |
| Sulfur | S | 100 | 100 |
| Titanium | Ti | 2.0 | 2.0 |
| | | | |
| Arsenic | As | 0 | 0.10 |
| Cadmium | Cd | 0 | 0.10 |
| Chromium | Cr | 0 | 0.20 |
| Cobalt | Co | 0 | 0.20 |
| Copper | Cu | 0 | 0.20 |
| Manganese | Mn | 0 | 0.20 |
| Nickel | Ni | 0 | 0.20 |
| Selenium | Se | 0 | 0.10 |
| Silver | Ag | 0 | 0.05 |
| Vanadium | V | 0 | 0.20 |
| Zinc | Zn | 0 | 0.10 |

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**APPENDIX_6: TUNING & INTERFERENCE CHECKS**

## 1.0 TUNING

1.1 The mass spectrometer must be "tuned" whenever the tune-check solution does not meet acceptance criteria or when maintenance has been performed on the detector, to verify the sensitivity, resolution, and mass alignment of the detector.

For Agilent 7500CE instruments (MET-06, MET-16, MET-26), the Tuning Solution contains 100 ug/L Cerium (Ce), Cobalt (Co), Lithium (Li), Thallium (Tl) and Yttrium (Y). The following guidelines should be met before any analyses are performed:

| | |
|---|---|
| 6020, 6020A & 200.8 Mass Calibration | ± 0.1 amu |
| 6020/6020A: Resolution at 10% peak height | < 0.75 amu |
| 200.8: Resolution at 5% peak height | < 0.75 amu |
| Oxide Ratio (Cerium Oxide: Cerium) | < 1% |
| Double-Charge Ions (m/z 140:70 ratio) | < 3% |

| | | Agilent 7500CE |
|---|---|---|
| Sensitivity | Li (m/z 7) | > 10,000 counts |
| | Co (m/z59) | > 20,000 counts |
| | Y (m/z 89) | > 20,000 counts |
| | Tl (m/z 205) | > 20,000 counts |
| Hydrogen-Mode Sensitivity | Co (m/z 59) | > 5,000 counts |
| | Se (m/z 78) | < 10 counts |
| | Fe (m/z 56) | Confirm Fe presence |
| | FeH:Fe (ratio m/z 57 to m/z 56) | ~ 3% |
| Helium-Mode Sensitivity | Co (m/z 59) | > 5,000 counts |
| | ArCl or false As (m/z 75) | < 10 counts |
| | ClO or false V (m/z 51) | < 100 counts |
| | Ratio (m/z 51 to m/z 59) | < 1% |

***EPA 200.8*** *Method Modification:* The tuning solution described in EPA 200.8 includes only Magnesium (m/z 24, 25 & 26) and Lead (m/z 206, 207 & 208). C&T uses the tuning solution described in 6020, because it includes lighter elements ($Li^6$) and additional mid-range elements.

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1.2 **EPA 6020 / 6020A Tune Check Standard (ICPMS TUNE)** contains 10 ug/L of Li (m/z 7), Co (m/z 59), In (m/z 115), and Tl (m/z 205). This standard must be analyzed at the beginning of the analytical sequence (and at the beginning of each 12-hour shift) to demonstrate that the detector hardware is correctly calibrated; no samples or calibration standards may be analyzed until the tune has passed acceptance criteria.

| | |
|---|---|
| Frequency: | Beginning of 12-hour shift and every 12 hours |
| Precision: | ≤ 5% RSD over at least 4 exposures (*Note: C&T typically uses 5 exposures to match the 200.8 requirement and allow both methods to be run on same sequence)* |
| Resolution: | < 0.9 amu @ 10% Peak Height |
| Mass Calibration: | ± 0.1 amu |

1.3 **EPA 200.8 Tune Check (200.8 TUNE)** contains 10 ug/L of Be, Co, In, Mg, Pb. This standard must be analyzed at the beginning of the analytical sequence in which samples submitted for EPA 200.8 are run (and at the beginning of each 12-hour shift) to demonstrate that the detector hardware is correctly calibrated; no samples or calibration standards may be analyzed until the tune has passed acceptance criteria.

| | |
|---|---|
| Frequency: | Beginning of 12-hour shift and every 12 hours |
| Precision: | ≤ 5% RSD over 5 exposures |
| Resolution: | < 1 amu @ 5% Peak Height |
| Mass Calibration: | ± 0.1 amu |

*6020 & 200.8 Method Modification*: The EPA methods only require a tune check standard at the beginning of each sequence however C&T analyzes a tune check standard at the beginning of each 12-hour "tune shift" to standardize Mass Spectrometry criteria across the lab.

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## 2.0 INTERFERENCE CHECK STANDARDS (ICS)

Interference Check Standards contain known concentrations of interfering elements and are used to demonstrate the magnitude of the interferences and that the inter-element correction factors correctly compensate for these interferences. These standards are analyzed at the beginning, every 12 hours, and end of every sequence. The recoveries for the requested elements must be within the acceptance limits or the instrument must be recalibrated and any samples analyzed since the last passing CCV reanalyzed.

Chloride is included in the ICS to evaluate software corrections for chloride-related interferences. Iron is used to verify that the resolution of the spectrometer is adequate for quantitation of manganese. Molybdenum evaluates oxide effects on cadmium isotopes.

2.1 **ICS-A** contains only the elements that typically cause interferences when present at relatively high concentrations. The affected elements (which are not included in this standard) should not be observed at levels above the limits that are or will be required for the methods contained in the day's sequence; see Section 5 for specific limits.

Note that, according to the standard manufacturers (Agilent, CPI and SPEX), low levels of some elements (notably chromium) will be present in the ICS-A due to impurities that cannot be removed from the vendors' cation sources. C&T controls only those elements to which Inter-element correction factors are applied.

2.2 **ICS-AB** contains the ICS-A elements at relatively high levels and all other target elements at lower levels (typical of those seen in environmental samples). None of the EPA ICP-MS methods (200.8, 6020, or 6020A) specify control limits for this standard, so C&T has chosen to standardize the ICP methods by using the EPA 6010 limits; see Section 5 for acceptance criteria.

*Method Modification*: C&T requires that the ICSAB be re-analyzed (and pass acceptance criteria) at the end of each analytical sequence and pass the acceptance criteria listed above; if the standard fails, any samples analyzed for the failing element after the last passing ICS-AB must be reanalyzed. The limits listed above are those typically required in DoD project plans and have been adopted by C&T because method 200.8 does not require these interference check standards and EPA methods 6020 and 6020A do not specify recovery limits for this standard and the instrument manufacturer (Agilent) recommends it "for informational purposes only".

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**APPENDIX_7: USING CHEMSTATION FOR INSTRUMENT CONTROL**

**Instrument Control Diagram**
Diagram displays a schematic of the instrument, allowing you to check the gauges, meters, and ON/OFF switches. Instrument control is accessed through this diagram.

*Instrument >> Instrument Control*

**Meter Control Diagram**
Diagram displays additional details for specific components (vacuum, water, gases, plasma Rf, housing, S/C) associated with instrument control.

*Instrument >> Instrument Control*
*Meters >> Meter Control*

**Tune Panel**
Tune Panel displays detailed information regarding system parameters.

*Instrument >> Tune…*

**'Standby' Mode to 'Analysis' Mode**
The instrument will go to 'Standby' mode, after maintenance, when the vacuum has pumped down and is ready. Then turn on the instrument by:

Select *Instrument >> Instrument Control*.
*Plasma >> Plasma On*
*YES*

**'Standby' Mode to 'Shutdown' Mode**
To do any maintenance in the vacuum chamber, the plasma must be turned off and the vacuum shut down.

Select *Instrument >> Instrument Control.*
*Plasma >> Plasma Off*
*YES*
*Vacuum >> Vacuum Off*

**'Shutdown' Mode to 'Standby' Mode**
Open ICPMS TOP program,
Select: *Instrument >> Instrument Control*
*Vacuum >> Vacuum On*
*YES*

Allow between 15 minutes to 2 hours to pump the vacuum down to $5x10^{-4}$ kPa.
Once the instrument has reached the 'Standby' Mode:

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Verify that the instrument exhaust rate is correct ( 5 m3/min ).
Verify that the water chiller is turned on.
Verify that the Argon pressure is correct ( 700 + 20 kPa ).

**Turn Off the Instrument**
To do any major maintenance (on the cones, extraction lenses, source, etc.), the plasma must be turned off and the vacuum shut down.

Put the instrument into 'Standby' Mode as described above.
Turn off the power switch on the front panel.
Turn off the rotary pump power switches on instrument.
Turn off the main power breaker on the back of the instrument.
Turn off the rotary pump power switch on the actual pump.

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**AGILENT 7500ce GENERAL STARTUP & ANALYTICAL PROCEDURE _____**

*Restart the Agilent 7500ce Instrument from Overnight Stand-by Mode*

1.) Replace the tubing around the peristaltic pumps.
2.) Refill the DI rinse-water bottle with acidified (1% HCl plus 1% $HNO_3$) DI water.
3.) Turn on the computer then click the ICPMS TOP icon.
4.) Open the Instrument Control Diagram.
5.) Verify that the instrument is in 'Standby' mode.
6.) Click YES when asked to verify the tubing connection; this will turn on the plasma and warm up the instrument. The warm-up will take approximately 5 minutes and the instrument icons will turn green as each system reaches 'ready'.

*Tune the Agilent 7500ce Instrument:*

7.) Open Tune Panel.
8.) Select: nogas.u from the dropdown menu.
9.) Select: ALS >> Go To >> '2'(Tune Solution).
10.) Select: Tune >> Sensitivity, then START.
11.) Verify that the RSD and Counts results are in the correct range.
Note: RSD's should be <5%, not to exceed 10%.
12.) Verify that the height of the $Li^{6+}$ peak is less than ½ the $Li^{7+}$ peak; once it reaches approximately ½, preventative maintenance (cleaning the cones) should be scheduled.
13.) Click STOP.

14.) Select: File >> Save Tune Values…(save to nogas.u)
15.) Select: H2.u from the dropdown menu
16.) Select: Tune >> Sensitivity, then START..
17.) Verify that the RSD and Counts results are in the correct range
18.) Click STOP

19.) Select: File >> Save Tune Values…(save to H2.u)

20.) Select: He.u from the dropdown menu
21.) Select: Tune >> Sensitivity, then START
22.) Verify that the RSD and Counts results are in the correct range
23.) Click STOP

24.) Select: File >> Save Tune Values…(save to He.u)
25.) Select: nogas.u from the dropdown menu
26.) Select: Tune >> Oxide Ion, then START.
27.) Verify that the oxide ratio is ≤ 1.0%.
28.) Click STOP.

29.) Select: Tune >> Doubly Charged Ion, then START.
30.) Verify that the ratio is ≤ 3%.
31.) Click STOP.

32.) Select: Tune >> Resolution/ Axis button, then START.
33.) Verify that the 'Axis' reading is within ± 0.1 amu from the true value for each element.
34.) Verify that the peak width (W-10%) is between 0.7 - 0.8 (must be < 0.9).
35.) Click STOP.

36.) Select: File >> Generate Multi-mode Report.
37.) Verify that all of the listed parameters are within acceptance limits.

*Analyze the Pulse/ Analog Standard:*

38.) Select: ALS >> Go To >> rinse
39.) Select: ALS >> Go To >> '5'(50ppb P/A Factor solution)
40.) Select: Tune >> P/A Factor
41.) Click 'Load Masses from aqu. Method'
42.) Click RUN
43.) After P/A Factor is RUN, Chemstation asks, '"Would you like to adopt new P/A Factors? (Refer to the report displayed on notepad.)"
44.) Observe intensities. Click 'Yes'
Note:If nessessary, adjust tuning parameters and reanalyze P/A Factor solution to aquire ,at a minimum, all of the internal standards
45.) Select: File >> Print, then close the screen.
46.) Select: File >> Copy Tune Parameters…
47.) Select: Export to button.
48.) Click Browse…, Select H2.u
49.) Check the Detector Parameters and P/A Factors box
50.) Click Copy
51.) Click Browse…, Select He.u
52.) Check the Detector Parameters and P/A Factors box
53.) Click Copy
54.) Click Close

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**APPENDIX_8: INTERFERENCES**

**Polyatomic Interferences** occur when an isotope of an interferent occurs at the same mass as the primary mass of an element of interest. Corrections can be calculated, based on the *observed* ratio of isotopes in a standard. See Section 3.2 of SW-846 method 6020 for further details and example equations.

Specific interferences which have been identified in the referenced methods include $ArCl^+$ with the $^{75}As$ signal and $MoO^+$ with the Cd isotopes.

For the Agilent 7500ce instruments (MET-06, MET-16, MET-26), most isobaric molecular interferences are reduced by the octopole reaction system running in Hydrogen Mode or Helium Mode.

**Memory Interferences**, also called 'carry-over', may occur when a low-level sample is analyzed immediately after a high-level sample. These interferences are minimized by rinsing the system between all samples and standards.

**Physical Interferences** are typically those occurring during the digestion, nebulization, and transport. Common physical interferences include:

1. - Loss of volatile elements (antimony) during the digestion process if the digestate is superheated or allowed to go dry during heating.
2. - Precipitation of certain elements (silver) during the digestion process if present in relatively high levels.
3. - Zinc contamination due to dusty surroundings.
4. - Chromium contamination in purchased ICS-A standards (a problem for all ICS suppliers – per Agilent Technologies).
5. - Differences in sample viscosity and surface tension due to high levels of dissolved solids. Serial dilutions may be used to identify this type of interference, which may clog the nebulizer and tubing.

This method includes the use of internal standards, which minimize the effects of these interferences on sample results by closely matching the physical properties of the associated internal standard to those of the target elements.

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**APPENDIX_9: INSTRUMENT CONDITIONS**

**AGILENT 7500ce** (MET-06, MET-16, MET-26)

| | | | |
|---|---|---|---|
| Argon | Purity (%): | 99.99 | |
| | Pressure (kPa): | 700 ± 20 | (Concentric Nebulizer) |
| | Flow Rate (L/min): | 1.2 | |
| Helium: | Purity (%): | 99.9999 | |
| Hydrogen: | Purity (%): | 99.9999 | |
| Water Cooler | Temperature (°C): | Set Point: 20°C (acceptable between 15°C to 25°C) | |
| | Pressure: | 40 psi | |
| | Flow Rate: | WC/IF: 1.1 to 2.0<br>RF/TP: 1.1 to 3.0 | |
| MSD Vacuum | Pressure (kPa): | $5 \times 10^{-4}$ | |
| Exhaust Flow | Rate ($m^3$/min): | 5 | |

Quantitation Channels (Octopole Reaction System):

| | |
|---|---|
| Hydrogen Mode (LIMS Channel H): | Fe, Se |
| Helium Mode (LIMS Channel E): | As, Cr, Co, Cu, Mn, Ni, Na, V, Zn |
| Vacuum only (LIMS Channel A): | Al, Sb, Ba, Be, Cd, Ca, Pb, Mg, Mo, K, Ag, Tl |

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**APPENDIX_10: MAINTENANCE & TROUBLE-SHOOTING**

**ROUTINE MAINTENANCE**

Any maintenance performed on the instrument must be documented in the instrument maintenance logbook. Whenever preventative or trouble-shooting maintenance is performed, document:

1.) the reason the maintenance was necessary,
2.) the action taken, and
3.) the resolution of the maintenance ("passed CRI", "RSD's OK", etc.).

Each benchbook entry must be initialed and dated by the analyst performing the maintenance. If an outside service contractor performs the maintenance, the identity of the contractor's company and a summary of the maintenance performed should be documented in the benchbook.

Detailed instrument maintenance instructions are described in Chapter 4 of the Agilent 7500 Hardware Manual. Maintenance and part numbers for the Integrated Sample Introduction System (ISIS) are described in the ISIS Manual.

1. - **Peristaltic Pump Tubing** should be replaced whenever the tubing becomes yellow, discolored, or cracked; typically the sample tube and ISTD tubing should be changed daily and the rinse vessel tube (drain tube) every month. High RSD's in the tuning mode typically demonstrate that peristaltic sample and ISTD pump windings need to be replaced. The type of tubing can be identified by the color-coded tabs fixed to the piece. Turn off the peristaltic pump, then replace the tubing. Order replacement tubing from CPI and keep at least one spare set on-hand.

| | | |
|---|---|---|
| Sample Tube | Tygon (Orange-Orange, 0.89mm ID) | CPI Cat # 4062-535 |
| ISTD Tube | Tygon (Orange-Red, 0.19mm ID) | CPI Cat # 4062-5008 |
| Drain Tube | PharMed (Yellow-Blue, 1.52mm ID) | CPI Cat # 4062-01916 |
| ISIS Pump Tube | Tygon(White-White, 0.60ml/min) | CPI Cat # 4062-440 |

Soak Tygon tubing in acidified (5% HCl plus 5% $HNO_3$) deionized water for at least 2 hours. Verify that any contamination has been rinsed from the tubing by running an instrument blank.

2. - The **nebulizer, spray chamber,** and **torch** may also need to be cleaned or replaced when elements begin routinely failing the RSD or CCV requirements. Allow the torch and interface to cool for at least 10 minutes before handling. Always wear gloves when handling the nebulizer and associated parts; if the surface becomes contaminated with oils, the signal will become unstable.

3. - The **cones** may need to be cleaned or replaced when elements begin routinely failing the RSD or CCV requirements, or any of the following are observed: low response, distorted

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peak shape, or interface vacuum pressure >5 x $10^2$ kPa. The sampling cone typically requires more frequent maintenance than the skimmer cone, due to its proximity to the torch. See pages 4-59 to 4-66 of the Hardware Manual for complete details.

*Caution:* Do not touch the skimmer cone orifice or allow it to come into contact with other instrument parts, as even slight pressure can damage the orifice. If the orifice of either cone becomes damaged or distorted, the cone must be replaced.

| | |
|---|---|
| Nickel Skimmer Cone | Agilent Cat# G1820-65050 |
| Nickel Sampling Cone | Agilent Cat# G1820-65238 |

After cleaning or changing the cones, run the Pulse/ Analog program (using the 50 µg/L calibration standard) to establish the concentration (intensity) at which the instrument should switch from Pulse to Analog mode.

*Note:* The Agilent-7500 operates in two modes, pulse-mode for typical low-level samples and analog-mode for high-level samples. The instrument automatically switches between the two modes based on the voltages encountered during sample analysis; the mode used for quantitation may vary on an analyte-by-analyte basis. The raw instrument data will include a data qualifier flag indicating which mode was used for quantitation, A(nalog), P(ulse), or M(ixed).

4. - The **extraction lens** and **Einzel lens assembly** may need to be cleaned when the instrument cannot be tuned or the ion signal is unstable. *Always* wear clean powder-free latex or cotton gloves when handling any vacuum chamber parts.

| | |
|---|---|
| Polishing Paper | Agilent Cat# G1833-65404 |

For Agilent 7500ce Instruments:
Only the Einzel lens assembly is inside the vacuum chamber. Reduce the maintenance time by first cleaning the Einzel lens assembly with specified Agilent polishing paper, reinstalling it (see pages 4-146 to 4-147 of the Hardware Manual) and beginning the vacuum pump-down, then cleaning the extraction-omega lens assembly in the same fashion. For the Agilent 7500ce, see pages 4-87 to 4-94 of the Hardware Manual for detailed instructions

Clean the o-ring with methanol and check for damage whenever re-installing the lenses; any dust or nicks in the o-ring will cause a vacuum leak when the instrument is pumped down.

5. - The **Octopole Reaction System** in the Agilent 7500ce may need to be cleaned when the counts in the normal mode (nogas.u) do not recover instantaneously when switched from the hydrogen mode ($h_2$.u) to the normal mode.

5.1 Remove the octopole from the instrument (see Hardware Manual pp. 4-97 to 4-99).
5.2 Sonicate entire octopole assembly (NOT INCLUDING the cell upper case, cell lower case, or cell assembly screws) in deionized water for 5 minutes.
5.3 Change deionized water and repeat sonication two more times.
5.4 Dry the octopole with nitrogen gas.

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5.5 Remove the Plate Bias Lens (see Hardware Manual pp. 4-102 to 4-103).
5.6 Clean the plate bias lens with specified Agilent polishing paper and deionized water.
5.7 Dry with nitrogen gas.
5.8 Reinstall octopole reaction system.

6. - The **electron multiplier (EM)** normally operates in pulse mode but automatically switches to analog mode when analyzing high concentration samples. The EM should be changed when adjusting increasing the EM voltage to meet sensitivity requirements results in an Analog voltage of ~3300 or a Pulse voltage of ~1800. See pages 4-85 to 4-88 of the Hardware Manual for detailed instructions.

| | |
|---|---|
| Electron Multiplier | Agilent Cat# G1833-65420 or -65575 |

7. - The **vacuum pump oil** should be changed every 6 months or if the pressure gauge reads 10 microns Hg or above. It typically takes 1-2 days after the oil change to pump the vacuum back down and remove all traces of oil from the system. See pages 4-95 to 4-97 of the Hardware Manual for detailed instructions.

| | |
|---|---|
| Rotary Pump Oil | Agilent Cat# 6040-0834 |

8. - The water **filter** for the re-circulator should be changed about every 6 months, or whenever the warning lights go on.

| | |
|---|---|
| Water filter | Agilent Cat# G1820-65018 |

9. - **Lubricate the autosampler** tracks approximately every six months by wiping the tracks with a Kim-Wipe saturated with 1-in-3 or clear oil.

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## TROUBLE-SHOOTING

For trouble-shooting purposes, the ICPMS can be separated into three distinct sections:
1.) The sample introduction system, from the autosampler sipper through the tubing and connections to the nebulizer and spray chamber and ending with the torch.
2.) The second consists of the cones back through the lenses.
3.) The third begins at the quadrupole (or octopole) and ends with the detector.

To begin trouble shooting, run a sequence of 5 sets of a high standard followed by a blank, while monitoring counts for two IS and two analytes. The counts for IS and high standard should not vary more than 5% between runs, and the IS should not drift more than 30% between the blank and high standard. If the IS counts are stable but the analyte counts drift or if the IS counts drift and the analyte counts are stable this indicates that there is problem in section one. Begin replacing and/or adjusting components in section one until this behavior discontinues.

If both counts for the IS and analyte are drifting through the run this indicates a problem in section two. Clean or replace components of section two until this behavior ceases.

To trouble shoot section three monitor drift of two plasma gases, such as mass 80 and 76 while aspirating no sample. A drift in counts of these readings indicate a problem with the Electron Multiplier or Mass Analyzer.

**Problem:** High RSD's in the tuning mode.
Solution: Peristaltic sample and ISTD pump windings need to be replaced.

**Problem:** Instrument cannot be tuned or the ion signal is unstable.
Solution: The extraction lens and Einzel lens assembly may need to be cleaned

**Problem:** Internal standard recoveries are stable for 1-2 hours after starting up instrument but then begin slowly rising and do not drop back to the baseline after repeated rinsing.

Solution: The plastic lens spacers in the source may have developed a charge. To check this possibility, go to the Tune screen voltages. Read the Tune Solution then change the voltages to zero. Let the instrument run for a few minutes to clear any temporary charge, then go back to the normal settings. Check the Tune Solution voltages - if the voltages change significantly, the spacers should be replaced.

**Problem:** Higher mass elements (particularly Molybdenum and heavier) fail calibration criteria.

Solution: Run the Pulse/Analog program described above, to reset the concentrations at which the instrument switches acquisition modes.

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**APPENDIX_11: CALCULATIONS**

The Agilent software automatically performs internal standard correction on the analyte intensities. These corrected intensities are then used to determine the calibration curve and analyte concentrations.

Instructions for printing Uncorrected Intensities:

1. Open Offline Data Analysis program.
2. From the 'Tools' drop down menu, select 'Configure Do List…', select 'FullQ-Summary-Printer', Add to the Do List.
3. From the 'Tools' drop down menu, select 'Do List'.
4. Select 'FullQ-Summary-Printer'.
5. Select file to be printed, 'Add file', then process file.
6. Selected file will print to the associated printer.

The corrected intensities are calculated by dividing the area count of the target ion by the ratio of the counts in the internal standard divided by the concentration of the internal standard in the first level of the calibration curve. These equations, as described in the Agilent Operator Manual, are:

$$y = y_\sigma / ( y_i / x_i ) \qquad (y = y_\sigma \text{ if } x_i = 0)$$

Where: $x_i$ = concentration of the internal standard in the first level of the calibration curve
$y_i$ = count of the internal standard
$y_\sigma$ = count of the target ion

Moisture Corrected Results
Dry Weight Concentration (ug/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

**Using Linear Regression:**
The corrected intensities are then used to calculate the following parameters from the initial calibration: slope (s) and intercept (I). The slope and intercept define a relationship between the concentration and instrument response of the form:

$$y = s\,x + I$$

Where: y = predicted instrument response
s = response slope
x = concentration of standard
I = intercept

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Rearrangement of the above equation yields the concentration corresponding to an "on-column" instrumental measurement:

$$x = (y - I) / s$$

Where: x = calculated on-column concentration for a sample
y = actual instrument response for a sample
s and I are calculated slope and intercept from calibration.

Final sample concentration is then corrected for any sample prep dilution factors:

$$\text{Sample Conc (mg/L)} = x * pdf * idf$$

Where: x = calculated on-column concentration for a sample
pdf = prep dilution factor
idf = instrument dilution factor

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## BATCH QC CALCULATIONS

**Percent Recovery (%R):**
The recovery is the measured concentration divided by the true concentration of the spike.

$$\%Recovery = (Cf - Cs) / (Cws * Vws / S) * 100$$

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

**Relative Percent Difference (RPD):**
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

$$\%RPD = |(Cs - Cdup)| / ((Cs + Cdup)/2) * 100$$

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSD's where the sample weights are not weight-targeted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

$$\%RPD = |( (Wms/Wmsd)*Cms - Cmsd )| / (( (Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

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## INITIAL CALIBRATION

### Correlation Coefficient

For each compound, the correlation coefficient for the initial calibration curve is calculated by:

$$\text{Correlation coefficient} = \frac{\sum_{i=1}^{n} (Yobs - Ymean)^2 - ((n-1)/(n-p)) * \sum_{i=1}^{n} (Yobs - Yi)^2}{\sum_{i=1}^{n} (Yobs - Ymean)^2}$$

Where:
Yobs = observed intensity for each ICAL std conc.
Ymean = mean observed intensity from the ICAL standards
Yi = calculated (or predicted) intensity for each ICAL std conc.
n = total number of ICAL points
p = number of adjustable parameters in equation (linear= 1, quadratic= 2)

### First Order Coefficient (Slope) - For Linear Regression ICAL

$$\text{Slope} = \frac{n * \sum (Cx * Ax) - (\sum Cx) * (\sum Ax)}{n * \sum (Cx^2) - (\sum Cx)^2}$$

Where:
n = total number of ICAL points
Ax = Area or Intensity of the compound being measured
Cx = Concentration of the compound being measured

## CALIBRATION VERIFICATION

$$\%\ \text{Difference}\ (\%D) = \frac{AVG\ RF - RFc}{AVG\ RF} * 100$$

Where:
AVG RF = Average response factor from initial calibration
RFc = Response factor from current verification check standard

$$\%\text{Drift}\ (\%D) = \frac{(C1-Cc)}{C1} * 100$$

Where:
C1 = Calibration Check Compound standard concentration
Cc = Measured concentration of CCC

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**STANDARDS PREPARATION**

To determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where:
Vss = Vol (mL) of Source Standard needed to make Working Standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

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Digestion & Analysis of Solid Samples for

# MERCURY

Cold Vapor Atomic Absorption (CVAA) Methods
EPA 7471A / 7471B

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Justin Buehner<br>Group Leader | | 01/18/16 |
| Teresa Morrison<br>QA Director | | 01/18/16 |
| **Reapproved:** | | |
| | | |
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Digestion & Analysis of Solid Samples for

# MERCURY

Cold Vapor Atomic Absorption (CVAA) Methods
EPA 7471A / 7471B

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Digestion & Analysis of Solid Samples for

# MERCURY

Cold Vapor Atomic Absorption (CVAA) Methods
EPA 7471A / 7471B

## 1.0 SCOPE

This procedure describes the sample preparation and analysis for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. The reporting limit for this procedure, using a 0.6g sample, is 0.02 mg/Kg.

In this procedure, organometallic compounds are broken down and converted to mercuric ions by digesting the sample with permanganate and persulfate under heated conditions. The concentration of mercury in the resulting digestate is then determined by cold vapor atomic absorption (CVAA). This procedure is not applicable to Waters or Liquid Samples (see Metals SOP 5.1, Method 7470A/ 245.1).

## 2.0 REFERENCES

EPA 7471A, Mercury in Solid or Semisolid Waste *(Manual Cold-Vapor Technique)*, SW-846, Sept 1994
EPA 7471B, Mercury in Solid or Semisolid Waste *(Manual Cold-Vapor Technique)*, SW-846, Feb 2007

*Additional SOP's and Guidance Documents:*
EPA 7000A, *Atomic Absorption Methods*, SW-846, Feb 2007
TNI Standard, Volume 1, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2 October 2010
Consolidated DoD/DoE QSM, Version 5.0, July 2013
C&T SOP QA 1.4, *Balance Calibration Check & Maintenance*
C&T SOP QA 1.5, *Calibrating & Maintaining Temperature Controls*
C&T SOP QA 1.6, *Pipette Calibration Check Procedures*
C&T SOP QA 1.9, *Calibration of Volumetric Glassware*
C&T SOP QA 4.1, *Establishing Control Limits*
C&T SOP QA 4.4, *Determining Method Detection Limits (MDL)+Limit of Detection (LOD)*
C&T SOP QA 4.6, *Limit of Quantitation (LOQ)*
C&T SOP CS 2.3, Subsampling & Compositing
Perkin-Elmer FIMS-100 Flow Injection Mercury System Hardware Guide (C&T MN# 317)
Perkin-Elmer FIMS-100 Setting Up and Performing Analyses (C&T MN# 318)

## 3.0 SAMPLE PRESERVATION & HOLDING TIME

| | |
|---|---|
| Preservative: | No chemical preservation.<br>Store at >0.0°C to 6.0°C |
| Holding time: | 28 days from sample collection until digestion & analysis |

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## 4.0 SAFETY

Mercury is a highly toxic element that is also somewhat volatile. The reagents used in this procedure are corrosive and can be harmful if inhaled. Always perform the digestions under a hood and take steps to avoid inhaling vapors generated in the process.

This procedure uses concentrated acids that will cause injury if allowed to contact skin or eyes.

Gloves and safety glasses and appropriate clothing should be worn at all times when handling samples, standards, or reagents. Aqua Regia must be used in the fume hood due to toxic and corrosive fumes.

## 5.0 QC REQUIREMENTS

Client Quality Assurance Project Plans (QAPPs) may require different acceptance limits than those listed below; for samples from those projects, the QAPP requirements supersede C&T in-house requirements.

| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Initial Calibration Curve (ICAL) | Prior to sample analysis or whenever CCVs fail and maintenance does not correct the problem<br><br>*C&T Requirement:*<br>Minimum 5 points + a blank;<br><br>*QSM 4.2/ 5.0:*<br>Minimum 5 points + a blank | Correlation Coefficient (r) $\geq$ 0.995<br><br>*7000A (7471A/ 7471B):*<br>Not specified<br><br>*QSM 4.2:*<br>Correlation Coefficient r $\geq$ 0.995<br><br>*QSM 5.0:*<br>$r^2 \geq$0 .99 | Perform instrument maintenance and reanalyze curve |
| Initial Calibration Verification (ICV) | After ICAL curve and prior to sample analysis | *7000A (7471A/ 7471B):*<br>%D $\leq$ 10<br><br>*QSM 4.2/ 5.0:*<br>%D$\leq$10 | Perform maintenance and rerun; if still out, reanalyze curve. |
| Continuing Calibration Verification (CCV) | After every ten samples (excluding MB, LCS/BS/BSD), and at end of the sequence | *7000A (7471A/ 7471B):*<br>%D $\leq$ 20<br><br>*QSM 4.2:*<br>%D $\leq$ 20<br><br>*QSM 5.0:*<br>%D $\leq$ 10 | Perform maintenance and rerun; if still out, reanalyze curve. See Section 11 below for sample guidance. |

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| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Calibration Blanks (ICB/CCB) | After each ICV and CCV | <RL/LOQ<br><br>*QSM4.2 / 5.0:*<br><LOD | Report samples that are ND or >10x contamination; rerun all others |
| Method Blank | 1 for every batch of 20 or fewer samples | <1/2 RL<br><br>*QSM 4.2/ 5.0:*<br><1/2 RL | Report samples that are ND or >10x contamination; rerun all others |
| Blank Spike (BS)/ Blank Spike Duplicate (BSD) | 1 for every batch of 20 or fewer samples | %Recovery & %RPD within statistically derived limits<br><br>*QSM 4.2 / 5.0:*<br>Within QSM LCS limits | Low recovery: reprep and reanalyze all samples.<br><br>High recovery: Reprep and reanalyze samples with results ≥LOQ. Report results <LOQ.<br><br>High RPD: Report ND samples, rerun all others |
| Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) | 1 for every batch of 20 or fewer samples | %Recovery & %RPD within statistically derived limits<br><br>*QSM 4.2 / 5.0:*<br>Within QSM LCS recovery limits; RPD ≤ 20% | Flag results as possible matrix interference. |
| Serial Dilution | *7000A:* One per batch, if native concentrations ≥ 25x RL, using a 5x dilution. If all native concentrations are <10xRL, run a Post-Digestion Spike<br><br>*DoD 4.2/5.0:* Not discussed. | *7000A:* %D ≤ 10 | Flag results as possible matrix interference. |
| Method Detection Limit (MDL) Study | When new instrument installed, or significant change made to process | ≤1/3 LOQ; see MDL SOP for additional details | See MDL SOP (QA 4.4) |

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| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Limit of Detection (LOD) | Analyze on each instrument, quarterly (DoD QSM requirement) | *QSM 4.2/ 5.0 :* Spike at 2-3x MDL and <LOQ; analytes detected | See MDL SOP (QA 4.4) |
| Limit of Quantitation (LOQ) | Analyze on one instrument, quarterly (DoD QSM requirement) | Spike at or above (up to 2x) the lowest ICAL standard. Recovery within 50-150%. | See LOQ SOP (QA 4.6) |
| Mechanical Pipettes and Reagent Dispensers | *QSM 5.0:* Verify upon receipt then daily, prior to use, using 3 replicates<br><br>*TNI-2009/ QSM 4.2:* Verify upon receipt then quarterly | *QSM 5.0:* Accuracy within ± 2% of nominal volume RSD ≤ 1% of nominal volume | See SOP for Pipettes & Dispensors (QA 1.6) for details |
| Glassware (Digestion Tubes) | Purchase Class-A, or verify accuracy of each lot upon receipt. | *QSM 5.0:* Accuracy within ± 3% of nominal. RSD ≤ 3% of nominal volume | See Volumetric Glassware SOP (QA 1.9) |
| Thermometers & Digestion Blocks | Check Liquid-in-Glass thermometer calibration annually | See QA SOP for Temperature Controls for details | See SOP for Temperature Controls (QA 1.5) for details |
| Performance Testing (PT) Samples | Semiannually. | Within study limits | Any failures must be investigated, corrected, and a new PT analyzed and passed before end of the semi-annual time-frame. |
| Demonstration of Capability (DOC) | Each new analyst; annually thereafter | 4 consecutive LCS within C&T recovery and RPD limits, or acceptable PT results | Retrain analyst and reanalyze DOC. |
| The following are not applicable to the Mercury analysis | | | |
| Post Digestion Spike (PDS) | QSM 4.2/ 5.0: Not required for the Mercury analysis | | |
| Surrogates | Not applicable to Metals analyses | | |
| Instrument Detection Limit (IDL) study | Not applicable to the Mercury analysis | | |
| Internal Standards | Not applicable to the Mercury analysis | | |

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## 6.0 QC DEFINITIONS

6.1 **Initial Calibration** curve is a series of standards that establishes the quantitation range of the instrument.

6.2 **Initial Calibration Verification (ICV)** standard is a "second source" standard that is obtained from a different manufacturer than the standards used for the initial calibration curve. The ICV is used to verify that both the working and source standards used for calibration were prepared correctly.

6.3 **Initial Calibration Blank (ICB)** is a reagent blank that follows the ICV and is used to demonstration that the instrument is free of contamination.

6.4 **Continuing Calibration Verification (CCV)** standards are analyzed throughout the sequence to demonstrate that the curve is still valid.

*Note:* The US Army Corp of Engineers recommends that the CCV's be prepared from the same source standards as the ICAL curve in order to better differentiate between changing instrument response and simple differences between standards.

6.5 **Calibration Blanks (CCB)** are reagent blanks that follow every CCV and are used to verify that the instrument remains free of contamination.

6.6 **Batch QC:** The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples.

6.6.1 **Method Blank (MB):** An aliquot of Teflon boiling chips is digested and analyzed with each batch of 20 or fewer samples to demonstrate that the glassware and reagents are free of contamination.

6.6.2 **Blank Spike / Blank Spike Duplicate (BS/BSD):** A blank spike and a blank spike duplicate are aliquots of Teflon boiling chips to which a known amount of analyte is added. This is then digested and analyzed with every batch of 20 or fewer samples to demonstrate that the procedure is accurate (measured by the *recovery*) and precise (measured by the *RPD*) in the *absence* of matrix interferences.

6.6.3 **Matrix Spike (MS) / Matrix Spike Duplicate (MSD):** An MS and MSD are aliquots of real world samples to which a known amount of analyte is added prior to sample digestion. The MS/MSD are used to demonstrate the accuracy and precision of the process on environmental samples, which may include matrix interferences.

*Note:* Client- or project-specific quality assurance project plans (QAPPs) may require an a Sample Duplicate (SDUP) and Sample Spike (SSPIKE) in place of the MS/MSD. For batches containing samples from these projects, the QAPP requirement takes precedence over this SOP.

6.7 **Sample Interference Verifications**: The method recommends that whenever a new or unusual matrix is encountered, a series of tests be performed to ensure that neither positive nor negative interferences are distorting the accuracy of the reported values.

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These should be analyzed whenever the analyst suspects that sample viscosity, salt content, or other matrix interferences are likely.

6.7.1 **Serial Dilutions:** Analysis of a 5x dilution should agree with the original determination if the concentration of the element in diluted aliquot is greater than the reporting limit. If not a chemical or physical interference, such as viscosity, should be suspected.

6.7.2 **Post Digest Spikes:** An analyte is added to a portion of a prepared sample digest, or its dilution, and analyzed to determine if matrix interferences that occur during digestion, are influencing the sample result.

6.8 **Instrument Detection Limit (IDL)**: An IDL study is determined by analyzing seven instrument blanks on three non-consecutive days to determine if the instrument is capable of meeting desired detection limits, before taking digestion procedures into account. The standard deviation of the calculated concentrations is determined for each day; the IDL is the sum of the three standard deviations.

6.9 **Method Detection Limit (MDL):** An MDL study is a set of at least 7 low-level blank spikes that are prepared and analyzed to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. See the QA SOP "Determining MDLs" for details.

6.10 **Limit of Detection (LOD)** samples are aliquots of deionized water that are spiked at 2-3x the calculated MDL, to verify that that the analyte can be detected at those levels. The LOD must be verified quarterly on every instrument for DoD (or annually for NELAC compliance). See the QA SOP for Method Detection Limits for details.

6.11 **Limit of Quantitation (LOQ)** samples are aliquots of deionized water that are spiked at 1-2x the lowest calibration standard and used to verify the accuracy of the procedure at the reporting limit. The LOQ must be determined quarterly (DoD requirement) on a single instrument. See the QA SOP for Limit of Quantitation for details.

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7.0 **INTERFERENCES**

Mercury is a relatively volatile element that may be vaporized if the digestion temperature is not closely monitored. Sulfide interferences are reduced through use of potassium permanganate in the digestion step. Samples with high organic content may reduce the potassium permanganate and require additional permanganate be added during digestion.

8.0 **EQUIPMENT** (See Appendix_5 for instrument conditions)

Perkin-Elmer FIMS-100 Mercury Analysis Systems
Perkin-Elmer AS-93plus Autosampler (MET-44)
CETAC ASX-520 Autosampler (MET-54)
Heating block capable of maintaining a temperature of 95°C ± 3°C (7471B Section 4.10)
Auto-pipette, adjustable to 0.5mL
Thermometer, minimum range 85-105°C
Teflon boiling chips, Saint-Gobain Chemware Ultra-Pure PTFE Boiling Stones (26397-103)
Disposable 70 mL digestion tubes, CPI Catalog # 4370-010010
0.45 µm Syringe Filters, Sartorius Catalog # 16537Q

*Note:* If the digestion tubes are not certified by the manufacturer or vendor as Class-A, the accuracy of each lot must be verified prior to use according to QA SOP for Volumetric Labware. This is done by selecting 5 digestion tubes from the lot and filling them to 50mL with 20°C deionized water and weighing it on an analytical (4 decimal) balance; the weights must fall within 3% of the expected weights (48.5 – 51.5g) or the lot may not be used for direct measurement of sample or digestate volume.

The equipment listed above was in use at the time this document was written; alternate equipment and supplies may be used at the laboratory's discretion, so long as all QA/QC requirements are met.

See the Perkin-Elmer FIMS-100 guides for hardware and software details.

## 9.0 DIGESTION PROCEDURE

### 9.1 Prepare the Calibration Standards

9.1.1 **Initial Calibration standards**: Add the following volumes of a 0.100 µg/mL mercury working standard to a series of clean, empty digestion tubes. These standards expire after 24 hours.

| ICAL Level | Final Concentration (µg/L) | Using Vol (mL) of 0.100 µg/mL Working Std | Final Volume in DI Water (mL) |
|---|---|---|---|
| Cal Zero | 0.0 | 0.00 | 50. |
| Std #1 | 0.2 | 0.10 | 50. |
| Std #2 | 0.5 | 0.25 | 50. |
| Std #3 | 2.0 | 1.00 | 50. |
| Std #4 | 5.0 | 2.50 | 50. |
| Std #5 | 10. | 5.00 | 50. |

Document the LIMS ID of the 0.100 µg/mL working standard in the digestion benchbook.

9.1.2 **ICV and CCV**s: Using the 0.100 µg/mL second source standard, prepare the standards below in clean, empty digestion tubes. These standards expire after 24 hours..

| ICAL Level | Final Concentration (µg/L) | Using Vol (mL) of 0.100 µg/mL Second Source Working Std | Final Volume in DI Water (mL) |
|---|---|---|---|
| ICV | 5.00 | 2.50 | 50. |
| CCV | 5.00 | 2.50 | 50. |

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Document the LIMS ID of the 0.100 µg/mL working standard in the digestion benchbook.

9.1.3 Starting at Step 9.3 below, add the reagents so the volume of the standards matches that of the samples and digest the standards along with the samples

9.2 **Prepare the Batch QC and Samples**

9.2.1 Turn on the digestion block and allow it to come to temperature (95°C ± 3°C).

9.2.2 Check samples out of the cold room and allow them to come to room temperature.

9.2.3 Calibrate the auto-pipette at each of the volumes needed to spike the batch QC samples and create the initial calibration standards. Document this calibration in the pipette benchbook.

9.2.4 Verify that the balance has been calibrated for the day; if it has not, calibrate it and document the calibration in the balance benchbook.

9.2.5 Verify the calibration of the reagent dispensors.

9.2.6 Select sufficient digestion tubes for all of the samples in the batch plus a method blank, blank spike, blank spike duplicate, and two matrix QC samples. Label each tube with a sample number or QC-type.

9.2.7 Homogenize a 5-10g subsample then weigh out triplicate 0.2g portions (for a total weight of 0.6g ± 0.05g) of the homogenized subsample into the bottom of the tared digestion tube labeled with that sample number.

9.2.8 Record the sample number, container *letter*, and sample weight (to 0.01g), in the benchbook. Place the digestion tube in the rack.

9.2.9 For the method blank (MB), label an empty digestion tube and weigh 0.6g ± 0.05g of Teflon boiling chips into the tube.

9.2.9 Laboratory Control Samples (LCS = Blank Spike) should be spiked between the low and middle level of the calibration standards.

Prepare the blank spike (BS) and blank spike duplicate (BSD) by adding 1.25 mL of the 0.100 µg/L Hg working standard to each of two digestion tubes containing 0.6g ± 0.05g of Teflon boiling chips.

9.2.10 Review the job sheets to determine if any of the clients in the batch requested that matrix QC be done on their sample. If so, use that sample for the MS/ MSD, otherwise choose a sample for batch QC so that matrix QC is rotated throughout the laboratory's clients and so that no one client's samples predominate over a period of time.

9.2.12 Prepare the matrix spike (MS) by adding 1.25 mL of the 0.100 µg/mL Hg working standard to a second 0.6 g aliquot of the sample being used for QC.

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*Note:* TCLP MS/MSD extracts should have been spiked immediately following filtration; do not add additional spiking solution.

9.2.13 Prepare the matrix spike duplicate (MSD) by adding 1.25 mL of the 0.100 µg/mL Hg working standard to a third 0.6 g aliquot of the sample being used for QC.

*Note:* TCLP MS/MSD extracts should have been spiked immediately following filtration; do not add additional spiking solution.

9.2.14 To each sample and batch QC sample, add 5 mL DI water and swirl to mix.

**9.3 Add the remaining Digestion Reagents and Complete the Digestion**

9.3.1 To each sample, batch QC sample, and calibration standard, add 5 mL of concentrated Aqua Regia and swirl to mix.

Document the C&T ID of the Aqua Regia in the digestion benchbook.

9.3.2 Place the rack containing the samples, batch QC, and calibration standards in the 95°C digestion block for 2 minutes then remove from the block and cool to about room temperature.

9.3.3 Add 25 mL deionized water to each sample and batch QC sample. Swirl to mix.

9.3.4 Add 7.5 mL of 5% $KMnO_4$ solution to each tube, including calibration standards, then mix thoroughly.

9.3.5 Put the tubes in the digestion block. Set the block to maintain a temperature of 95°C $\pm$ 3°C for 30 minutes. In the digestion log, record the time and temperature when the samples are placed into the digestion block and then both again when they are removed from the block.

9.3.6 After about 5 minutes, verify that the digests are still purple in color. If they are not, slowly add just enough granular $KMnO_4$ to make the color persist. Use the same $KMnO_4$ as used to make the liquid solution.

If additional $KMnO_4$ is added, make a note of it in the 'comments' section of the benchbook, next to that sample, as it will likely require a dilution.

*Note:* C&T uses granular $KMnO_4$ because samples high in organics often require too much additional permanganate to make addition of the liquid solution feasible.

9.3.7 Cool to about room temperature then add 3.0 mL of sodium chloride-hydroxylamine-HCL to reduce the excess permanganate.

9.3.8 Add enough DI water to each tube to bring all sample, batch QC and calibration standards to 50mL final volume, then cap and invert to mix.

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9.3.9 Filter each sample, including batch QC samples, through a clean 0.45µm syringe filter and place the filtrate into a labeled autosampler tube.

9.3.10 Document the LIMS standard number of the spikes and all C&T reagent ID's of the reagent in the digestion benchbook, and complete the digestion record.

9.3.11 Enter the prep data into LIMS and scan the prep benchbook page into LIMS.

## 10.0 INSTRUMENT PROCEDURE

### 10.1 Calibrate the Instrument

10.1.1 While the samples are digesting, refill the stannous chloride reservoir and make sure the inlet tube is submerged in the reagent; document the reagent ID in the digestion log. Turn on the lamp and allow the instrument to equilibrate for about an hour.

10.1.2 After digestion, load the calibration standards onto the autosampler tray in order of increasing concentration.

10.1.3 Add instrument blanks before the ICAL standards and immediately following the ICV, to demonstrate that the low-level standard was not influenced by instrument contamination and that analytes at the high-level concentration will not carry-over into real-world samples.

10.1.4 Analyze the calibration standards and blanks.

10.1.5 Use the Calibration Data section found in the WinLab results window, below ICAL 5, to verify that the correlation coefficient (r) for the resulting calibration curve is ≥ 0.995.

If one level of the initial calibration curve has a particularly deviant response factor, that level may be rerun so long as it immediately follows the last calibration standard and no sample digests have been injected.

10.1.6 Before loading samples, verify that the ICV recovery is within acceptance limits and that no mercury was detected in the instrument blank. If the ICV is outside the acceptance limits, it may be reanalyzed once, so long as no sample digests have been injected (which could change the instrument response).

Table 10-1: Calibration Verification Limits

| Standard | Concentration (µg/L) | Acceptance Limit | Acceptance Range ((µg/L) |
|---|---|---|---|
| ICV | 5.00 | %D ≤ 10 | 4.50 – 5.50 |
| CCV | 5.00 | %D ≤ 20 | 4.00 – 6.00 |
| CCV (QSMv5.0) | 5.00 | %D ≤ 10 | 4.50 – 5.50 |

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10.1.7 An acceptable ICV must be analyzed before any samples are loaded. If the ICV does not pass acceptance criteria and samples were analyzed immediately following it, the entire calibration must be reanalyzed, as there is no way of determining what affect the sample matrix would have on any subsequent ICV analysis.

Create a sequence calibration in LIMS after an acceptable ICV run by first selecting the appropriate fill boxes (to the left of the ICAL and ICALBLK runs) then select 'Tasks>' at the bottom of the page and click the Create calibration button.

10.2 **Analyze the Samples**

10.2.1 After the instrument has been calibrated, set up the remainder of the sequence as outlined below, with a CCV/CCB pair analyzed after every ten samples (excluding the method blank and blank spikes), and at the end of the sequence.

Initial Calibration Standards (run lowest to highest)
Initial Calibration Verification (ICV, second source) standard
Initial Calibration Blank (ICB)
Method (Prep) Blank
Blank Spike
Blank Spike Duplicate
QC Sample (MSS)
Matrix Spike
Matrix Spike Duplicate
7 more samples
Continuing Calibration Verification (CCV) standard
Continuing Calibration Blank (CCB)
10 samples
CCV
CCB
etc……

Instrument blanks should not precede CCVs; if CCVs routinely fail due to instrument carryover, the instrument rinse/bake times must be adjusted instead. Instrument Blanks may be analyzed after high concentration samples regardless of whether a sample or CCV follows.

10.2.2 Place the samples in an autosampler rack and program the autosampler appropriately. The instrument will automatically add the 10% stannous chloride reagent and analyze the samples.

10.2.3 **Dilutions:** If the sample concentration is greater than the highest point in the calibration curve, and the digest was completed within the past 24 hours, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range, using Appendix_3 as a guide.

If the digestion was completed more than 24 hours previously, redigest the sample in a new batch and dilute the digestate prior to analysis.

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If the sample was initially run at a dilution but was ND ("not detected"), it must be rerun undiluted, or at a dilution that brings the instrument reading into the middle of the calibration range. If the digestion was completed more than 24 hours previously, the sample will need to be redigested before being reanalyzed.

10.2.4 If a **Serial Dilution (SER)** is needed, measure 2.0 mL of the MSS (unspiked aliquot of the "matrix spike sample") into a digestion tube. Add 8.0mL of the 5% digested blank (to match the reagent concentrations) and swirl to mix. Decant into an autosampler tube and add to the sequence.

10.2.5 If a **Post-Digestion Spike (PDS)** is needed, pipette 9.5 mL of the MSS into a digestion tube. Add 0.5 mL of a 0.1 mg/L standard. Cap and swirl to mix. Decant into an autosampler tube and add to the sequence.

## 11.0 DATA REVIEW & REPORTING

Quantitation is based on comparison of the measured absorbance for the sample against the initial calibration curve, with adjustments for the sample preparation concentration factor and instrument dilution factor. See Appendix 4 for example calculations. Concentrations in soil samples are expressed as milligrams per kilogram (mg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

11.1 After the sequence is complete, review the data to determine if the data may be reported or if reanalysis or redigestion is necessary. Any corrective action involving reanalysis of the digests must occur within 24 hours of the digestion, or the digestion must be repeated.

11.2 Evaluate the **Continuing Calibration Verification (CCV)** data. If the %D for any element is outside this acceptance window, LIMS will use the following to determine if the associated results are reportable:

a. If a low recovery is observed, any samples bracketed by the low standard must be reanalyzed.

b. If a high recovery is observed and no mercury was detected in the samples, those samples may be reported without reanalysis.

c. If a high recovery was observed and mercury was detected in the samples, those samples must be reanalyzed.

11.3 Evaluate the **Continuing Calibration Blank (CCB)** data. Mercury should not be detected in the calibration blanks at any level greater than the method detection limit however, if it is detected, use the following to determine the course of action:

a. If mercury is detected above the MDL, but below ½ RL, in the CCBs, narrate the problem on the data review checklist and report the data without reanalysis.

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b. If mercury is detected above ½ the RL but mercury was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c. If mercury was detected above ½ the RL and mercury was also detected in the sample(s), but the level in the sample(s) is greater than 10x that in the blank, the results may be reported without reanalysis, as the contamination level is considered insignificant in relation to the sample levels.

d. If mercury was detected above ½ the RL and mercury was also detected in the sample(s) at levels < 10x that in the blank, the samples must be reanalyzed.

11.4 Evaluate the **Method Blank (MB)**. The results for the method (preparation) blank should be < 1/2 RL. If reanalysis confirms the contamination, initiate a Corrective Action Report (CAR) and use the following steps to determine if the sample results may be reported:

a. If the concentration in the blank is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

b. If mercury was found in the blank but was not detected in the associated samples, the data may be reported and the problem narrated.

c. If mercury was found in the method blank and was also detected in the associated samples, but the level in the samples is greater than 10x the level in the blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If mercury was detected in the blank and was also detected in any of the associated samples, but at a level less than 10x the level in the blank, and reanalysis confirms the problem, those samples must be redigested and reanalyzed.

11.5 Evaluate the **Blank Spike (BS) and Blank Spike Duplicate (BSD):** Both recoveries and the RPD should fall within acceptance. If either recovery or the RPD fails, immediately reanalyze the failing batch QC sample. If the failure is repeated, initiate a Corrective Action Report (CAR) and use the following steps to determine the appropriate corrective action:

a. If a low recovery is observed, all samples in the batch must be redigested and reanalyzed.

b. If a high recovery is observed and no mercury was detected in the samples, those samples may be reported without reanalysis.

c. If a high recovery was observed and mercury was detected in the samples, those samples must be redigested.

d. If a high RPD was observed but both recoveries are within limits and no mercury was detected in the associated samples, the samples may be reported without reanalysis.

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e. If a high RPD was observed and mercury was detected in the associated samples, the samples must be redigested.

*Note:* Project-specific limits may be different than C&T acceptance limits. For samples submitted for that project or client, the project-specific limits supersede C&T limits.

11.6 Evaluate the **Matrix QC (SSPIKE/SDUP or MS/MSD):** The recoveries and RPD should fall within limits, or the QC sample (MSS) and matrix spikes may need to be redigested and reanalyzed. Use the following steps to determine any necessary corrective action:

a. If the concentration in the sample is greater than the linear range and the sample needs to be rerun for quantitation, report the MS/MSD with a LIMS-flag of ">LR" on those recoveries, without reanalysis.

b. If the concentration in the sample is within linear range but the concentration in the matrix spikes is greater than the linear range, LIMS will apply a ">LR" flag to those recoveries. Report the data without reanalysis.

c. If the concentration is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

*Note:* If the concentration is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis, unless project notes (on job sheet) specifically require reanalysis of failed MS/MSD.

e. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, report the non-MSS samples with just the BS/BSD. Redigest and reanalyze the QC sample (MSS) and matrix spikes.

f. If one or both recoveries fail and the RPD is outside limits, examine the data to determine if a matrix interference should be suspected. If no interference or isolated problem can be identified, report the non-MSS samples with just the BS/BSD; redigest and reanalyze the QC sample (MSS) and matrix spikes.

11.7 Examine the **Sample Results**. If the concentration in any extract exceeds that of the most concentrated standard, the results will be flagged '>LR' by LIMS; the extract must be diluted and reanalyzed to bring the response into the calibration range. Diluted sample concentrations should be in the mid- to upper-half of the calibration range.

Any dilutions must be analyzed within 24 hours after completion of the digestion. If analysis is delayed, the sample must be re-digested in a new batch and run at a dilution.

If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

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## 11.8 LIMS Review & Reporting

11.8.1 Open a web browser and go to the main METALS page.

11.8.2 Under Recent Sequences, choose the instrument and then choose the sequence you want to review.

11.8.3 Check the sequence for any errors. LIMS takes the run information directly from what is typed in the sequence. Typing errors can affect how the data is processed by LIMS.

If you need to make any changes to the sequence, click FIX. Make any needed changes, noting the reason in the space provided at the top of the form, then click UPDATE.

11.8.4 Once everything is correctly processed in the sequence, review the sequence to see that all samples, STDs, and QC meet acceptance criteria. Samples and QC should have an “OK” flag in the QC column of the sequence, otherwise reanalysis may be required. Once the sequence has been reviewed, it must be signed in the REVIEW APP.

11.8.5 Open REVIEW APP for the sequence by choosing the sequence (e.g. MET14 / 05/08/07) under the Review pull down list on the top right corner of the sequence page.

Two windows should open. One window shows the main Review App page. One window shows the documents (e.g. reports, scanned documents, Form 1s, etc).

*IMPORTANT!* Make sure you are logged in with your own user initials. The current user's initials are displayed on the bottom left corner of the review app window. To logout, click the user initials and Review App will ask you to login.

11.8.6 The sequence is listed on the left frame of the Review App page with the calibration displayed at the top. Clicking on an item on that list will display the LIMS report and any associated documents.

11.8.7 The analyst must review, sign and send the calibration for secondary review before the sequence can be verified/reviewed (for either all files in the sequence or those files the analyst has reviewed if the sequence is still running).

11.8.8 From another open web browser, go back to the main METALS page and click on run chooser.

11.8.9 Enter the login number, analysis or rgroup (metals), and matrix for the job to be reported. Choose the result(s) to be reported for each sample and QC for the job.

11.8.10 Select reports from the top left side of the screen. From the views section in the upper right select possible. This will list the possible reports that can be printed.

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Select the reports to be printed and print to the printer designated "Nowhere". This will select and lock the final results in the LIMS database.

11.8.11 From the REVIEW APP page you left open previously, enter the batch number in the box in front of the search button and click the button.

11.8.12 Review the results and sign the main page of the batch, then send the batch to the review queue for second party review.

11.8.13 From a different open web browser, go back to the main METALS page and enter the job number you wish to report in the search box and select search. Open REVIEW APP for the job by choosing the job (e.g. 214373 METALS Filtrate) under the Review pull down list on the top right corner of the page.

11.8.14 Review the results and sign the main page for the job.

11.8.15 Verify that the prep sheets are complete and signed, that all necessary sample information is present (see section below for details). Complete any narrative comments, review the checklist and sign the 'Data Review Checklist'.

11.8.16 Submit the data to the Group Leader, qualified analyst, or QC Chemist for second-party review.

Any changes made by the second-party reviewer must be individually signed by the reviewer. The second party reviewer must sign each user report, make any additional comments on the case narrative and sign the completed checklist. If a Level III or IV data package is required, the second-party reviewer will generate it after signing the reviewed data and reports.

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## 12.0 DOCUMENTATION REQUIREMENTS

### 12.1 Sample Prep Documentation

A copy of the digestion benchbook page for the sample digestion must be scanned so prep info can be included with reported data. The digestion benchbook entries should include:

- C&T sample ID's and unique container identifier,
- date of sample digestion, initial volume or weight of sample, and final digestate volume,
- time the samples were placed in the water bath and time they were removed,
- identity of QC samples (spikes, duplicates & LCS),
- amount of spikes added and LIMS identification numbers of all spiking solutions,
- a list of all reagents used (C&T ID or manufacturer and lot number),
- identification of all support equipment (digestion tubes, pipettes, thermometer, digestion block, balance) used in preparation of the batch
- indication of whether or not the digests were filtered after digestion,
- any unusual occurrences observed during the digestion procedure

### 12.2 "Paperless" Package

12.2.1 Scan the digestion benchbook page into the scanner. Select Lab > Batch Prep Sheet > Enter Batch Number. Scan.

12.2.2 Sign the Batch using the Review App. Confirm the batch prep sheet appears correctly.

12.2.3 Sign the Calibration for all instruments and runs for that batch. This is located under the ICAL section on the left side of the Review App beneath the batch number.

12.2.4 Sign the sequences in which the jobs were run. This is verifying that the samples were loaded in the correct order and at the correct dilutions as indicated by the sample files in the software.

12.2.5 Sign all the jobs within the batch. Verify that the reports have been printed and that all the runs are correctly chosen. Insure that there are no samples marked as "NA" due to missing Mercury runs. Samples may be marked "NA", however, due to other runs needed within the department.

12.2.6. Have the data package reviewed by the Department Manager or QC Chemist.

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## 13.0 POLLUTION PREVENTION

Vent the mercury analyzers into the fume hood. Purchase only sufficient standard and reagent volume to use within the expiration date, to reduce the volume of laboratory waste.

## 14.0 WASTE DISPOSAL

All containers holding chemical waste (liquid or solid) must be kept closed except when necessary to add or remove waste.

Waste containers must be compatible with the type of material being stored and must be in good condition (ie: not rusted or cracked).

Each container must be clearly labeled “Hazardous Waste”, with a description of the composition, physical state of the waste (‘Liquid/Solid’), the type of hazard (Corrosive, Flammable Solvent, etc. ), and the date that the first contents are added to the container

14.1 Soil samples must be returned to the coldroom for later inclusion in the lab’s soil waste stream.

14.2 Excess digest and standard volume may be accumulated in the Mercury lab for up to 9 months after which they must be transferred to the 55 gallon tight-head plastic drum labeled “Acid Waste”, located in the garage.

## 15.0 REVISION HISTORY

The previous document (revision 18) was changed as follows:

- Section 2: Removed Leeman equipment manuals from references, added Perkin-Elmer documents
- Section 5: Updated support details to match DoD QSMv5.0 requirements. Added 40CFR136.7 items that are not applicable to Mercury, with notes to that effect

- Section 8: Added Teflon boiling chips to equipment list. Updated digestion block temperature range to match 7471B (95°C ± 3°C). Updated syringe filter details and instruments (from Leeman to Perkin-Elmer)
- Section 9.2: Added requirement to use Teflon boiling chips for blank and blank spikes. Added note that TCLP MS/MSD will already have been spiked.
- Section 12: Added requirement to document ID's of support equipment
- Appendix_2: Updated with current standards details
- Removed Appendix_7 which previously contained instructions for using Leeman software

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**APPENDIX_1:** **MERCURY SOIL**
**Digestion Summary**

| | |
|---|---|
| Sample Weight: | 0.6g (± 0.05 g) |
| Spike: | 1.25 mL of HG 0.1 STD |
| 1st Digestion: | + 5 mL DI water<br>+ 5 mL Aqua Regia<br>95 °C<br>2 minutes |
| 2nd Digestion: | + 25 mL DI water<br>7.5 mL of 5% $KMnO_4$ solution<br>90 - 95 °C<br>30 minutes (add $KMnO_4$ crystals if color fades) |
| Final: | + 3.0 mL NaCl-Hydroxylamine HCl<br>repeat until appearance is stable & colorless<br>+ DI water to bring final volume to 50mL |
| Filtration: | 0.45 µm Syringe Filter, including all batch QC |

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## APPENDIX _ 2: REAGENTS & STANDARDS

The standards and reagents listed below are those in use at the time this procedure was written. Alternative Reagents and Supplies may be substituted provided the substitution is equivalent and meets all calibration, quality control, and traceability requirements.

### REAGENTS

Document the preparation of all reagents, including dilutions into DI water, in the reagent prep benchbook; include the date and analyst initials, the name of the reagent, and the manufacturer and lot# in the benchbook entry. Assign a unique ID, based on the type of reagent and the date prepared; record this ID in the digestion benchbook each time the reagent is used.

Label each bottle of reagent obtained from a vendor with the date received, date opened, and the expiration date.

Label each bottle of reagent prepared by C&T with the name (and concentration where applicable) and the preparation date. The expiration date must be clearly listed either on the bottle or in the preparation record.

Deionized Water, ASTM Type II (ASTM D1193)

Hydroxylamine Hydrochloride ($H_2NOH.HCl$), reagent grade
Fisher Scientific catalog # H330-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Nitric Acid ($HNO_3$), concentrated, InstraAnalyzed grade
JT Baker catalog # 7697-37-2
Store in a Corrosives cabinet for up to 2 years

Hydrochloric Acid (HCl), concentrated, InstraAnalyzed grade
JT Baker catalog # 7647-01-0
Store unopened bottles in the corrosives cabinet.
Store open bottles under the fume hood for up to five years.

Potassium permanganate ($KMnO_4$), reagent grade, mercury free
Fisher Scientific catalog # P279-212
Store in an amber glass bottle at room temperature for up to 5 years

Potassium persulfate ($K_2S_2O_8$), reagent grade
Fisher Scientific catalog # P281-500
Store at room temperature for up to 5 years

Sodium Chloride (NaCl), reagent grade
Fisher Scientific catalog # S671-500
Store in a tightly sealed plastic bottle at room temperature for up to 5 years

Sulfuric Acid Concentrated ($H_2SO_4$), concentrated, InstraAnalyzed grade

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JT Baker, VWR catalog # JT9691-3
Store in a Corrosives cabinet for up to 2 years

Aqua Regia: *prepare daily*

Prepare immediately before use in a glass bottle

1.) Measure 96 mL concentrated nitric acid ($HNO_3$) into a glass bottle
2.) Add 32 mL concentrated hydrochloric acid (HCl).
3.) Swirl to mix.

5% Nitric Acid ($HNO_3$)

1.) Add 50mL concentrated $HNO_3$ to 950mL deionized water.
2.) Swirl to mix.
3.) Store at room temperature for up to 1 year

5% Potassium permanganate ($KMnO_4$, saturated) by weight: *expires after 3 months*

1.) Dissolve 50 g of potassium permanganate in about 400mL DI water in a 1L volumetric flask.
2.) Agitate to dissolve the solids.
3.) Bring to 1,000 mL with DI water.
4.) Cap and invert at least 3 times to homogenize.
5.) Store in an amber glass bottle at room temperature, under a fume hood, for up to three months.

5% Potassium persulfate ($K_2S_2O_8$), by weight: *expires after 1 year*

1.) Dissolve 50 g of potassium persulfate in 1000 mL DI water.
2.) Store in an amber glass bottle at room temperature in the hood for up to one year.

Sodium chloride-hydoxylamine hydrochloride solution: *expires after 3 months*

1.) Add 120g of sodium chloride to about 200mL of DI water in a 1L volumetric flask.
2.) Add 120g of hydroxylamine hydrochloride.
3.) Agitate to dissolve the solids.
4.) Dilute to 1,000 mL with DI water.
5.) Cap & invert at least 3 times to homogenize.
6.) Store in a glass bottle at room temperature, under a fume hood, for up to three months.

10% Stannous Chloride ($SnCl_2$) “flow reagent” solution: *expires after 5 days*

1.) Add 40g stannous chloride to 200 mL of DI water.
2.) Add 30 mL HCl and swirl to mix.
3.) Add 170 mL of DI water to bring the total volume to 400 mL.
4.) Agitate the mixture to dissolve any residual tin chloride crystals.
5.) Store under a hood, in an amber glass bottle at room temperature for up to 5 days; prepare fresh solution more frequently if it turns slightly yellowish.

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## SOURCE STANDARDS

Source standards are those purchased from a chemical manufacturer or vendor, and the source standard should be traceable to NIST. For source standards, the LIMS S-name is unique to both the composition (compound list) of the standard *and* to the vendor of that standard. A new S-name must be assigned whenever the composition is changed or when the standard is obtained from a different vendor; the information must then be entered in the "Standard Definitions" table before the new standard is assigned an S#.

For calibration standards, at least one of the manufacturers (either primary or secondary) must be ISO Guide 34 accredited. This is an A2LA requirement associated with C&T's DoD accreditation.

Certificates of Analysis should be obtained from the vendor of each source standard; each standard should be traceable to NIST. If the supplier did not provide a certificate, call and request that a copy be emailed. Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory".

Label the Certificate of Analysis with the LIMS S#, date received and expiration date (if not already listed on vial). Scan the certificate into LIMS, then query it to verify that the scanned copy is complete and legible.

Label each vial with the contents, LIMS S#, and expiration date. Store each standard in the vendor's bottle at room temperature, until the assigned expiration date.

| Analytes | Concentration | Supplier & Catalog# | LIMS S- Name |
|---|---|---|---|
| Mercury, primary source std. (Guide 34 traceable) | 1,000 µg/mL | CPI # Z-G34-060080-02-01 | HG STD |
| | | | |
| Mercury, second source std. | 1,000 µg/mL | Inorganic Ventures # AAHG1 | IV HG |

## WORKING STANDARDS

Working standards are those prepared at C&T. For working standards, the LIMS S-name is not unique to the source standard vendor but *is* unique to the compound list and concentrations contained in the working standard; if the concentration or compounds in the working standard changes, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard is logged in and assigned an S#. It is very important to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Verify that the LIMS expiration date does not exceed the Source Standard expiration date. If it does, change the working standard expiration date to that of the source standard. **The**

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**expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

In the Standards Benchbook, enter:

- prep date,
- LIMS S#, concentration and volume of each source standard used,
- LIMS S-name, final volume and concentration of the working standard,
- Lot# of any acid used to acidify the standard,
- expiration date of the new working standard, and
- prep chemist's initials

In LIMS, enter the prep chemist's initials, prep date, and S# of all source standards used to make the working standard; LIMS will then assign a working standard number (S#).

Prepare working standards by diluting source standards to volume in a Class-A volumetric flask. Label the standard vial with the contents, the LIMS WS#, the expiration date of the standard, and the prep chemist's initials.

**Procedure:**

1.)Prepare all working standards in a **100 mL Class-A volumetric flask**, initially adding about 50mL DI water.
2.)Add 5mL concentrated Nitric Acid to the DI water (so the final acid concentration in 100mL final volume will be 5%). Swirl to mix.
3.)Add the volumes and standards listed below to make each standard.
4.)Bring to volume with DI water.
5.)Cap and invert three times to mix. Allow sufficient time, during each inversion, to mix thoroughly.

All volumes given below may be adjusted proportionally to yield more or less of the solution required. All spiking standards should be analyzed to verify their concentration **PRIOR TO USE**. The data from these screens should be stored in the appropriate binder.

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## MERCURY STANDARDS PREP

| Working Standard | WS Conc. (ug/mL) | Using "Source" Std | Add Vol (mL) Source Std | Final Vol (mL), in 5% HNO3 | LIMS S-Name | Shelf Life |
|---|---|---|---|---|---|---|
| Primary Source Intermediate | 10.0 | HG STD | 1.0 | 100 mL | HG 10 STD | 3 mo |
| Primary Source Working Std | 0.100 | HG 10 STD | 1.0 | 100 mL | HG 0.1 STD | ** |
| | | | | | | |
| 2nd Source Intermediate | 10.0 | IV HG | 1.0 | 100 mL | HG 10 REF | 3 mo |
| 2nd Source Working Std. | 0.100 | HG 10 REF | 1.0 | 100 mL | HG 0.1 REF | ** |

** The SW-846 methods (7470A, 7471A, 7471B) require the 0.1 µg/mL standard to be prepared "daily". Sequences that carry overnight into the next day may continue with the previous day's standards, but the sequence may not be continued through a second night.

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## DAILY CALIBRATION STANDARDS

| CAL Standard & Concentration | Final Volume (mL) | Using Source Std | Add Vol (mL) Source Std | LIMS S-Name |
|---|---|---|---|---|
| CAL-Zero 0.0 µg/L | 50 | DI water | 0.00 | |
| CAL-1 0.2 µg/L | 50 | HG 0.1 STD | 0.10 | |
| CAL-2 0.5 µg/L | 50 | HG 0.1 STD | 0.25 | |
| CAL-3 2.0 µg/L | 50 | HG 0.1 STD | 1.00 | |
| CAL-4 5.0 µg/L | 50 | HG 0.1 STD | 2.50 | |
| CAL-5 10.0 µg/L | 50 | HG 0.1 STD | 5.00 | |
| | | | | |
| ICV 5.0 µg/L | 50 | HG 0.1 REF | 2.50 | HG ICV |
| | | | | |
| CCV-1 0.5 µg/L | 50 | HG 0.1 STD or REF | 0.25 | CCV1 HG |
| CCV-1 2.0 µg/L | 50 | HG 0.1 STD or REF | 1.00 | CCV2 HG |
| CCV-1 5.0 µg/L | 50 | HG 0.1 STD of REF | 2.50 | CCV3 HG |

**APPENDIX _ 3: SAMPLE DILUTIONS**

If the sample concentration is greater than the highest point in the calibration curve, prepare a dilution at a level that will bring the absorbance into the middle of the calibration range. This dilution must be injected onto the instrument within 24 hours of completion of the digestion; if the digestion was completed more than 24 hours previously, redigest the sample in a new batch and dilute the digestate prior to analysis.

Examine the original sample data to determine what dilution factor will be required to bring the absorbance down to the middle of the calibration range.

Using calibrated auto-pipettes, add the following volumes sample followed by $HNO_3$ acidified (5% vol:vol) deionized water to a 15mL disposable centrifuge tube. Cap and invert three times to mix, allowing sufficient time for complete mixing with each inversion.

**DILUTIONS**

| Instrument Dilution Factor (IDF) | Digestate Volume | Add Volume (mL) Acidified DI Water | Final Volume (mL) |
|---|---|---|---|
| 2 | 5.0 mL | 5.0 | 10 |
| 5 | 2.0 mL | 8.0 | 10 |
| 10 | 1.0 mL | 9.0 | 10 |
| 20 | 0.50 mL | 9.5 | 10 |
| 50 | 0.20 mL | 9.8 | 10 |
| 100 | 0.10 mL | 9.9 | 10 |

If a sample should need a dilution of more than 100x, prepare a 100x dilution first, then use that to make subsequent dilutions.

**SERIAL DILUTIONS**

| Instrument Dilution Factor | Using Primary Dilution | Volume of Primary (100x) Dilution | Add Volume (mL) Acidified DI Water | Final Volume (mL) |
|---|---|---|---|---|
| 500 | 100x | 2.0 mL | 8.0 | 10 |
| 1,000 | 100x | 1.0 mL | 9.0 | 10 |
| 2,000 | 100x | 0.5 mL | 9.5 | 10 |
| 5,000 | 100x | 0.2 mL | 9.8 | 10 |
| 10,000 | 100x | 0.1 mL | 9.9 | 10 |

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## APPENDIX _4: CALCULATIONS

### SAMPLE CONCENTRATION

Raw Amt (ug/L) = (m * intensity) + b

Where: m = first-order coefficient (slope) from initial calibration (LIMS a1)
b = intercept (LIMS a0)

LIMS then applies the sample prep information to calculate the mercury concentration in the sample by dividing the total ug by the prep dilution factor.

Conc (ug/L or mg/Kg) = Raw Amt * Vf / Si * IDF

Where: Vf = Final Digestate Volume (mL)
Si = Sample Initial Volume (mL) or Weight (g)
IDF = Instrument Dilution Factor

Moisture Corrected Results:
Dry Weight Concentration (mg/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

### BATCH QC CALCULATIONS

**Percent Recovery (%R):**
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws/ S) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard
S = Sample weight or volume

**Relative Percent Difference (RPD):**
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup )| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

For soil MS/MSD's where the sample weights are not weight-targetted, the expected concentrations will vary with sample weight (because the same volume of spike standard is being added to different weights of sample) and must be accounted for when calculating RPD:

%RPD = |( (Wms/Wmsd)*Cms - Cmsd )| / (( (Wms/Wmsd) * Cms + Cmsd)/2) * 100

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## CALIBRATION EQUATIONS

**%Drift used calibration verification**
Used for calibration verification standards, to determine if the instrument is still in control:

% Difference (% Drift) = %D = ((C1 - C2) / C1) * 100

Where: C1 = Concentration of the Calibration Verification Standard
C2 = Measured concentration

**Linear Regression Equations**

$$Y = mx + b$$

Where: y = response (Ax for external standard, or Ax/Ais for internal standard)
Where Ax = Area of compound
Ax / Ais = Area of compound divided by area of internal std
x = concentration (Cx for external standard, or Cx/Cis for internal standard)
m = slope
b = intercept

Slope (m) = $[ (\sum wx_i y_i * \sum w) - (\sum wx_i * \sum wy_i) ] / [ (\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i) ]$

Intercept (b) = $y_{avg} - (m * x_{avg})$

Correlation Coefficient (r)

$$r = \frac{[ (\sum w * \sum wx_i y_i) - (\sum wx_i * \sum wy_i) ]}{\sqrt{\{[ (\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [ (\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i) ]\}}}$$

Coefficient of Determination ($r^2$) = r * r

Where: $x_i$ = individual values for the independent variable (concentration)
$y_i$ = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
$x_{avg}$ = average of the x-values
$y_{avg}$ = average of the x-values

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**STANDARDS PREP & OTHER QC**

Spiking Batch QC for Waters
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/L)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Volume of Sample (mL)

Spiking Batch QC for Soil
Calculate the volume of working standard to add to make the LCS/ MS/ MSD using the following:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where: Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/Kg)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Mass of Sample (g)

Make a Working Standard from a Source (Stock) Standard:
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make new standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

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**APPENDIX_5: INSTRUMENT CONDITIONS**

| | | |
|---|---|---|
| Instrument: | MET-44, MET-54 Perkin-Elmer FIMS-100 | |
| Argon | Purity | 99.99% |
| | Max Pressure | 75 psi |
| | Flow Rate | 0.6 L/min |
| Oven Radiator Temperature: | 125 °C (to maintain 50°C gas temperature) | |
| Lamp Current | 6 – 10 mAmps | |
| Auto Sampler | 30s sip duration<br>75s rinse time | |
| Peristaltic Pump Rate | 6.0 mL/min Sample<br>0.8 mL/min Reagent (Stannous Chloride) | |
| Auto-Integration | 4 replicates | |
| Baseline Correction | 1 point | |

*Note:* The conditions listed above were those in use at the time this document was written and may be modified at the analyst's discretion, so long as those changes are documented in the instrument maintenance log.

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## APPENDIX_6: INSTRUMENT MAINTENANCE & TROUBLE-SHOOTING

All parts & supplies can be ordered from Perkin Elmer; see instrument manual for part numbers. Alternate suppliers and part numbers may be used at the analyst's discretion.

Document all maintenance events must be documented in the instrument maintenance benchbook. Include each of the following in the benchbook:

- reason for the maintenance,
- the initials of the person performing the maintenance and the date performed,
- description of maintenance performed,
- resolution (If the maintenance was due to poor instrument performance, did the maintenance solve the problem?)

**Pump windings** must be replaced whenever cracked or leaking.

The **drying tube** (gas-liquid separator) should be cleaned by sonicating in 1:1 nitric acid/ DI water for 30 minutes whenever the glass begins to turn yellow from tin chloride precipitation.

The **gas-liquid separator** should be cleaned with spectrophotometric grade isopropyl alcohol if the cell appears smudged or dirty.

The **lamp** should be ordered when the voltage is at 10mV and replaced when the voltage reaches 15mV.

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### TROUBLE-SHOOTING

See the appropriate Perkin Elmer Operation Manual for guidance on trouble-shooting.

- Contamination/ Carryover problems:
  Try running an acidic (1:1 nitric/DI water, or 1:1 Nitric/HCl) instrument rinse cycle for ~15 minutes

- Clogged tubing:
  For a largely clogged tube, switch out the lines for new lines. If only a minor clog, try back-flushing first with a 5% Nitric rinse.

- Autosampler arm squeaks or sticks:
  For MET-14, the autosampler arm track may need to be re-greased.

### DIGESTION BLOCK INSTRUCTIONS

1. Turn on the block.
2. On the keypad, press ENTER/ START.
3. Select AUTO.
4. Set the temperature to 95°C.
5. Allow block to warm up for 20 - 30 minutes.

6. Use a thermometer, in a water-filled digestion tube, to verify that the temperature is 95°C.
7. Adjust the temperature setting as needed so that the digestate temperature will be maintained at 95°C.
8. Place the digestion tubes in the bath.
9. Set the timer for 30 minutes for soil samples (2 hours for water).
10. Document the time and temperature in the digestion log.

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# MOISTURE (% Solids)

## IN SOIL & SEDIMENT SAMPLES

## US-EPA CLP Method ILM05.3

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Justin Buehner<br>Metals Group Leader | | 6-23-15 |
| Teresa Morrison<br>QA Director | | 6/23/15 |
| **Reapproved:** | | |
| | | |
| | | |
| | | |
| | | |
| | | |

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# MOISTURE (% Solids)
# In Soil & Sediment Samples
CLP Method ILM05.3

## 1.0 SCOPE

The moisture analysis is normally used to "correct" sample concentrations, obtained from other analyses, for the water content; the pollutants are assumed to all be originating in the dry soil. Results that have not been corrected are reported on an "as received" basis, while those being corrected are reported on a "dry weight" basis. Because the results from this analysis are used to "correct" the results from other analyses so that those results can be reported on a "dry-weight" basis, therefore it is very important to take a representative aliquot from the sample.

"Percent solids" is defined as the residue left after drying a 5-10 gram aliquot of the sample at 103-105 °C. Samples are weighed to the nearest 0.0001 g so that variations in moisture can be detected to 0.1% level or better.

## 2.0 REFERENCES

US-EPA CLP Inorganics Statement of work ILM05.3, Exhibit D, Section 1.6,
See Appendix 1 for CLP method text.
C&T SOP QA 1.4, Balance Calibratoin
TNI Standard, Volume 1, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2, October 2010
DoD QSM, Version 5.0, July 2013

## 3.0 PRESERVATION & HOLDING TIME

Preservation: Store at 0 – 6.0°C
Holding Time: None stated in the regulations

## 4.0 SAFETY

Assume that all samples contain hazardous and/ or toxic material and should be handled with care. Safety glasses, gloves, and a lab coat should be worn whenever handling samples, reagents, or standards.

## 5.0 QC REQUIREMENTS & DEFINITIONS

### 5.1 QC Requirements

| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Initial Calibration (ICAL) | Balance calibration must be verified daily before use, using weights that bracket the mass range used | See Balance Calibration SOP | See Balance Calibration SOP. |
| Method Blank | 1 for every batch of 20 or fewer samples | Final weight within ± 0.01g of initial tare weight | Re-prepare and reanalyze all samples in batch |

| | | | |
|---|---|---|---|
| Sample Duplicate (SDUP) | 1 for every batch of 20 or fewer samples | If Solids is <50%, %Solids RPD ≤ 15<br>If Solids is >50%, %Moisture RPD≤ 15 | Re-prepare and reanalyze all samples in batch |
| Laboratory Control Sample (LCS) | Not applicable to this analysis | | |
| Initial Calibration Verification (ICV) | Not applicable to this analysis | | |
| Continuing Calibration Verification (CCV) | Not applicable to this analysis | | |
| Calibration Blanks (ICB/CCB) | Not applicable to this analysis | | |
| Surrogate(s) | Not applicable to this analysis | | |
| Internal Standard(s) | Not applicable to this analysis | | |
| Method Detection Limit (MDL) Study | Not applicable; limited by 'constant weight' definition | | |
| Limit of Detection (LOD) | Not applicable; limited by 'constant weight' definition | | |
| Limit of Quantitation (LOQ) | Not applicable; limited by 'constant weight' definition | | |
| Demonstration of Capability (DOC) | Each new analyst; annually thereafter | 4 consecutive SDUP %RPD within C&T limits | Retrain analyst and reanalyze DOC. |

## 5.2 Definitions

a. **Batch**: A batch consists of 20 or fewer samples that are prepared and analyzed together.

b. **Method Blank (MB)**: A method blank is analyzed in parallel with the samples, to demonstrate that the laboratory process (and balance) is in control.

c. **Sample Duplicate (SDUP):** A second aliquot of the sample selected for batch QC is prepared and analyzed alongside the other samples in the batch, to demonstrate that the reproducibility (%RPD) of the results is within expected limits.

d. **Drying Time:** Drying time is defined as the elapsed time in the oven; analysts must record time in and out of the oven to document the 12-hour drying time minimum.

e. **Constant Weight:** To demonstrate that a constant weight has been achieved (and that there is no remaining moisture in the sample), data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with a minimum

of 1-hour drying time in each cycle. Constant weight is defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

## 6.0 INTERFERENCES & TROUBLE-SHOOTING

If the sample is an oily matrix, a constant weight may not be achievable; document the problem in the benchbook and initiate a Corrective Action Record.

Always use gloves whenever handling the drying pans to avoid getting oils from your skin on the weighing pans and lids. Skin oils will increase the pan weight and bias the moisture results.

## 7.0 EQUIPMENT & MAINTENANCE

Balance, capable of weighing to at least 0.01g
Drying Oven, maintained at 103 - 105ºC

Desiccator
Keep lid well greased or don't close desiccator lid until the filters have cooled (timing is important); otherwise it creates a vacuum and you may not be able to open it.

Desiccant, 10-20 mesh, VWR Catalog # 22891-083
Desiccant should not be used if it appears more pink than blue (indicating that it has become saturated with water). Regenerate the desiccant by placing a thin layer of it in a shallow pan in an oven at exactly 230°C for 1 ½ to 2 hours. The regenerated desiccant will be a softer blue than when received from the vendor.

## 8.0 PROCEDURE

8.1 Verify that oven temperature is within acceptance criteria (103 - 105 °C) and document the temperature in the Moisture benchbook.

8.2 Each aluminum weighing dish has a number on the lip, written with a sharpie marker. Verify that there sufficient clean, dry dishes with readable numbers and that there are no duplicate numbers in the set you are going to use. If you need to renumber a dish do it now.

8.3 Place the dishes in the oven on the bottom for at least 1 hour before going to the next step.

8.4 Check the samples out of the cold room and allow them to come to room temperature. Arrange samples in numerical order to speed the weighing and recording process. Record the sample numbers and container letters in the Moisture benchbook.

8.5 Verify that the balance has been calibrated for the day. If it has not, verify that the weights bracketing the intended range of use (usually 5g and 10g weights) read within the tolerance limits listed in the front of the balance benchbook (from the Balance Calibration SOP). Record the readings in the balance calibration benchbook.

8.6 Carefully remove the dishes from the oven using gloves, so that no oil transfers from your skin, and place in a desiccator. Allow the dishes to cool for about 5 minutes.

8.7 Transport the desiccator and the pans to the balance area where the samples are lined up.

8.8 Tare the balance to read “Zero” then remove an aluminum dish from the desiccator and place it on the balance pan. Weigh the dish and the lid and record the ***tare*** weight (weight of the pan) with the corresponding pan number in your sample data benchbook.

8.9 Write the sample number and container letter in the moisture benchbook, then open the sample container and use a clean wooden spatula to place between 5 and 10 grams of samples into the pan. Record the exact ***initial*** weight (to at least 0.01g) of the pan plus the sample in the benchbook.

8.10 Take the pan with sample off the balance and place on a ***clean*** countertop. Continue to weigh out all samples in this manner, repeating Steps 8.8 and 8.9.

8.11 Select a sample for duplicate analysis and prepare a second aliquot of this sample, recording “SDUP” and its sample number clearly in the Moisture benchbook.

8.12 After all samples have been weighed out, place samples in pans into the oven. Record the date and time in the Moisture benchbook.

8.13 Leave samples in oven overnight, not less than 12 or more than 24 hours. Record the date and time samples were removed from the oven and the oven temperature at time of removal.

8.14 Carefully remove the samples from the oven and place them in the desiccator. Put the pan lids on..Let the weighing pans and lids sit in the desiccator for about 1 minute to cool down.

8.15 Tare the balance to read ‘Zero’ then carefully place the first dry pan on the balance. Record the ***final*** weight of pan and dried sample in the Moisture benchbook. Repeat until all samples have been weighed.

8.16 Enter data (tare weight, initial weight, and final weight) into LIMS; LIMS will then calculate the %Solids, %Moisture and RPD of duplicate.

8.17 Print the results from LIMS and review that data against the benchbook entries to ensure there are no data entry errors & that the data makes sense (ie: if the soil was fairly dry, does the %moisture reflect that or does it imply that the sample was mostly water).

8.18 Review the SDUP %Solids results. If the sample is relatively dry (Moisture <50%), the %Solids RPD should be ≤ 15%. If the sample is very wet (Moisture >50%), the %Moisture RPD should be ≤ 15%.

If the RPD is >15%, inform the Department Manager and start a Corrective Action Record; the entire batch will need to be re-prepared and reanalyzed.

8.19 Scan the LIMS moisture entry report, benchbook, and balance calibration log into LIMS for review and signature. Scan in under 'Wetchem Packages', and the batch number, then turn the data in to the Department Manager or QC Chemisty.

## 9.0 QUICK ANALYSIS

If you can't wait overnight for moisture data use the following quick technique:

9.1 Perform Steps 8.1 – 8.13 above, but remove samples after they have been in the oven for 4 hours.

9.2 Place the pans with the dry samples in a desiccator until they've cooled.

9.3 Weigh, record the first ***final*** weight and place the sample & pan back in the oven for at least one hour.

9.4 Remove from the oven, place in desiccator to cool, the weigh the sample again.

9.4.1 If the second ***final*** weight for the same sample is within ± 0.01g of the first ***final*** weight, then the longer drying time is not required and the data may be used from either weighing event.

9.4.2 If the second *final* weight differs from the first *final* weight, by more than 0.01g, the samples must be placed back in the oven and the cycle repeated until the weights are in agreement.

## 10.0 DOCUMENTATION REQUIREMENTS

A copy of the benchbook page documenting when and how the samples were prepared may accompany the samples. The completed benchbook pages must be scanned into the LIMS using the workflow menu lab sample numbers and batch numbers may need to be entered into the scanner.

Each benchbook page should include the following information:

- Identification of analyst
- C&T sample ID's
- Initial and final sample weights
- Tare weight of pan
- Date & Time the determination started and finished
- Calculation of total time in oven
- First and final weights as needed to demonstrate attainment of constant weight
- Oven temperature verification 103-105 C

Any unusual observations

## 11.0 WASTE DISPOSAL

All containers holding chemical waste (liquid or solid) must be kept closed except when necessary to add or remove waste. Waste container must be compatible with the type of material being stored and must be in good condition (ie: not rusted or cracked).

Each container must be clearly labeled “Hazardous Waste”, with a description of the composition, physical state of the waste (‘Solid’), the type of hazard (‘Toxic’), and the date that the first contents are added to the container.

The dried sample waste may be accumulated in the leachates lab for up to 9 months, after which it must be transferred to the 55-gallon open-head steel drum containing ‘Residual Soil’, located in the 90-day accumulation area of the garage

## 12.0 POLLUTION PREVENTION

No pollution prevention or waste reduction/minimization procedures have been developed for this method at this time.

## 13.0 REVISION HISTORY

The previous document (revision 6) was changed as follows:

- Cover page and numbering were reformatted
- Section 2: Updated references
- Section 5: Added definitions. Added items required in 40CFR136.7 (even though waste water requirements are not applicable to this particular analysis) to standardize SOP format.
- Section 7: Added equipment
- Section 8: Expanded discussion of SDUP results. Added requirement to scan data into LIMS
- Section 11: Expanded waste disposal details
- Appendix_1: Added %RPD calculation

**APPENDIX_1: CALCULATIONS**

LIMS calculates the %solids and %moisture, using the following equations:

%Moisture = (Ww – Wd) / Ww * 100

Where: Wd = weight of dry sample = ($W_3$ – $W_1$)
Ww = weight of original "wet" sample = ($W_2$ – $W_1$)

$W_1$ = initial "tare" weight of pan
$W_2$ = initial (wet) weight of sample plus pan
$W_3$ = final (dry) weight of sample plus pan

%Solids = (100 - %moisture)/ 100

%RPD = |(SA - DU) / ((SA + DU)/2)| * 100

Where: SA = the sample concentration
DU = the duplicate concentration.

**APPENDIX_2: CLP PERCENT SOLIDS DETERMINATION (ILMO5.3D-SEC 1.6)**

1.6.1 Immediately following the weighing of the sample to be processed for analysis, add 5-10 g of sample to a tared weighing dish. Weigh and record the weight to the nearest 0.01 g.

1.6.2 Place weighing dish plus sample, with the cover tipped to allow for moisture escape, in a drying oven maintained at 103-105°C. Sample handling and drying should be conducted in a well-ventilated area.

1.6.3 Dry the sample overnight (12-24 hours) but no longer than 24 hours. If dried less than 12 hours, it must be documented that constant weight was attained.1 Remove the sample from the oven and cool in a desiccator with the weighing dish cover in place before weighing. Weigh and record weight to nearest 0.01 g. Do not analyze the dried sample.

1.6.4 Duplicate percent solids determinations are required at the same frequency as other analytical determinations. Duplicate results are to be recorded on Form VI-IN.

1.6.5 For the duplicate percent solids determination, designate one sample aliquot as the “original” sample and the other aliquot as the “duplicate” sample. Calculate dry weight using the results of the “original” sample aliquot.

1.6.6 Calculate percent solids by the formula below. The value thus obtained will be reported on the appropriate Forms I and, where applicable, Forms VA-IN and VI-IN. This value will be used for calculating analytical concentration on a dry weight basis.

EQ. 1 Percent Solids

$$\%\text{Solids} = \frac{\text{Dry weight of sample}}{\text{Wet weight of sample}} \times 100$$

1.6.7 If the sample contains less than 50% solids, the Contractor shall notify SMO immediately of the samples impacted. After notification to SMO, the Contractor shall proceed with sample analysis and document the issue in the SDG Narrative.

Drying time is defined as the elapsed time in the oven; thus raw data must record time in and out of the oven to document the 12-hour drying time minimum. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with a minimum of 1-hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

ENTHALPY ANALYTICAL
Berkeley

Sonication (EPA 3550) Extraction of Soil Samples for

# POLYCHLORINATED BIPHENYLS (PCB)

For GC-ECD Method EPA 8082

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Aaron Grenier<br>Group Leader | | 7/16/17 |
| Teresa Morrison<br>QA Director | Teresa Morrison | 7/16/17 |
| **Reapproved:** | | |
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Sonication (EPA 3550) Extraction of Soil Samples for

# POLYCHLORINATED BIPHENYLS (PCB)

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Sonication (EPA 3550) Extraction of Soil Samples for

# POLYCHLORINATED BIPHENYLS (PCB)

## 1.0 SCOPE

This document describes the extraction of Polychlorinated Biphenyls (PCBs) from soil samples by sonication extraction for later analysis by GC method EPA 8082, which uses an Electron Capture detector. See Appendix 1 for a summary of this extraction procedure.

Using this procedure, with an initial weight of 30g and a final volume of 25mL, the reporting limits for this procedure are 12-24 µg/Kg for soil samples; see the PCB analytical SOP for details.

## 2.0 REFERENCES

*Extraction & Cleanup:*
EPA 3500C, Organic Extraction & Sample Preparation, SW-846, Feb 2007
EPA 3500B, Organic Extraction & Sample Preparation, SW-846, Dec 1996
EPA 3550B, Utrasonic Extraction, SW-846, Dec.1996
EPA 3550C, Ultrasonic Extraction, SW-846, Feb 2007
EA SOP# CS 2.3, Subsampling & Compositing (ASTM D6323-98)
EA SOP# CS 2.4, Multi-Incremental Sub-sampling (MIS)

*Cleanup Methods:*
EPA 3665A, Sulfuric Acid/Permanganate Cleanup, Rev 1, Dec. 1996

*Analysis:*
EPA 8082A, PCB's by GC-ECD, SW-846, Feb 2007
EPA 8082, PCB's by GC-ECD, SW-846, Dec 1996

*Related QA SOPs and Guidance Documents:*
XLAB 1.2, Chemical Handling in the Extraction Lab
EA SOP# SVOC 3.2, Polychlorinated Biphenyls by EPA 8082/8082A/608
EA SOP# QA 1.4, Balance Calibration Procedures
EA SOP# QA 1.6, Pipette and Auto-Dispensor Calibration Verification
EA SOP# QA 4.1, Establishing Control Limits
EA SOP# QA 4.4, Determining Method Detection Limits (MDL) + Limit of Detection (LOD)
EA SOP# QA 4.6, Limit of Quantitation (LOQ)
EA SOP# XLAB 1.6, Calibrating Volumetric Glassware
EA SOP# XLAB 1.11, Screening of Solvents & Reagents
EA SOP# XLAB 1.12, Sonicator Tuning
EA SOP# XLAB 1.3, Spill Control & Cleanup
TNI Standard, EL-V1-2009, September 2009
Consolidated DoD Quality Systems Manual (QSM), DoD v5.1/ DoE v3.1, Jan 2017

## 3.0 SAMPLE PRESERVATION & HOLDING TIME

Preservation: Store at >0.0 to 6°C.

Holding Time: California: 14 days from sample collection until extraction

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(SW-846 Update III, Chapter 4, 1996)
40 days from extraction until analysis

Arizona: 1 year from sample collection until extraction
40 days from extraction until analysis
(Information Update # 144, Nov.06, 2012)

Other states: No holding time specified for either extraction
Or analysis.
(SW-846 Update IV, Chapter 4, Table 4-1, 2007)

## 4.0 SAFETY

The finals steps of this procedure involve the use of concentrated sulfuric acid, a very corrosive substance that will cause chemical burns & injury if allowed to contact skin or eyes. Take particular care to always wear lab gloves and safety glasses whenever handling strong acids.

This procedure also includes the use of flammable (Hexane) and potentially carcinogenic (Dichloromethane) solvents; see '*Chemical Handling in the Extraction Lab'* for more details.

Assume that all samples contain hazardous and/ or potentially toxic chemicals and should be handled with care. Safety glasses, gloves, and a lab coat should be worn whenever handling samples, standards, or reagents.

## 5.0 QC REQUIREMENTS & DEFINITIONS

Client-supplied quality assurance project plans (QAPP) may have different requirements than those listed below. For those projects, the client's QAPP supersedes those listed below.

### 5.1 QA/QC Requirements

| QC Element | Frequency | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Surrogates | Every sample and batch QC sample | %Recovery within acceptance limits | Low recovery: Re-extract and reanalyze affected sample<br><br>High recovery: Re-extract and reanalyze if results ≥LOQ. Report results <LOQ |
| Method Blank | 1 for every batch of 20 or fewer samples | <LOQ<br><br>QSMv5.1: <1/2 LOQ | Report samples that are ND or >10x contamination; rerun all others |
| Laboratory Control Sample (LCS)<br><br>- or – | 1 for every batch of 20 or fewer samples<br><br>(if insufficient sample | %Recovery & %RPD within acceptance limits | Low recovery: reprep and reanalyze all samples. |

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|---|---|---|---|
| Blank Spike (BS)/ Blank Spike Duplicate (BSD) | volume was submitted for an MS/MSD, extract two LCS, assigning acronyms of BS & BSD in LIMS) | | High recovery: Reprep and reanalyze samples with results ≥LOQ. Report results <LOQ.<br><br>High RPD: Report ND samples, rerun all others |
| Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) | 1 pair for every batch of 20 or fewer samples (if insufficient sample volume was submitted, the LCS/MS/MSD may be replaced by a BS/BSD) | %Recovery & %RPD within acceptance limits | Flag results as possible matrix interference |
| Internal Standards | Not applicable to this procedure | | |
| Instrument Calibration | Not applicable to this procedure | | |
| Standards & Reagents | Each new lot number (or cycle-tainer for MeCl2) – prior to use | Reagents & standards must be approved *prior to use.*<br><br>See XLAB SOP 1.11 for screening reagents | See Reagents SOP |
| Balance Calibration | Verify daily prior to use | Within tolerances posted at the balance; see Balance Calibration SOP for details | Clean & recheck. If still out of limits, initiate a CAR and use a different balance |
| Reagent Dispensors Calibration | Verify before being put into use and quarterly thereafter | Within tolerances listed in Pipette & Dispensors SOP | See Pipette & Dispensors SOP |
| Volumetric Glassware | Upon receipt (Class A are exempt from initial verification), then upon evidence of deterioration | Within tolerances listed in Pipette & Dispensors SOP | See Pipette & Dispensors SOP |
| Method Detection Limit (MDL) Study | When new instrument installed, or significant change made to process | <1/3 LOQ; see MDL SOP for additional details | See MDL SOP |
| Limit of Detection (LOD) verification | Analyze on each instrument, quarterly | Spike at 1-4x MDL and <LOQ; analytes | See MDL SOP |

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|---|---|---|---|
| | (DoD QSM requirement) | detected | |
| Limit of Quantitation (LOQ) verification | Analyze on one instrument, quarterly (DoD QSM requirement) | Spike at or above (up to 2x) the lowest ICAL standard. Recovery within 50-150% | See LOQ SOP |
| Demonstration of Capability (DOC) | Each analyst; upon completion of training & annually thereafter | *Initial:* 4 consecutive LCS within recovery and RPD limits<br>*Continuing:* Another 4 consecutive LCS, or acceptable PT results | Retrain analyst and reanalyze DOC. |

## 5.2 QA/QC Definitions

5.2.1 Method Blank (MB):
A method blank is an aliquot of a clean reagent that is extracted and analyzed with every batch, to demonstrate that the extraction and analysis procedures are free of contamination and have not contributed to any reported sample results.

5.2.2 Blank Spike (BS) / Blank Spike Duplicate (BSD) – or Laboratory Control Sample (LCS): A Blank Spike is an aliquot of clean matrix to which a known amount of the target compound is added prior to extraction. The extract is then analyzed and the "%recovery" calculated, to demonstrate that the extraction and analysis procedures are effective in the absence of matrix interferences. The Blank Spike Duplicate is a second blank spike that is analyzed to demonstrate the reproducibility of the process

If a client requests matrix spikes on their sample, and supplies sufficient sample volume, a single Blank Spike (designated as an LCS in LIMS) should be extracted along with the matrix spike and matrix spike duplicate.

5.2.3 Matrix Spike (MS) / Matrix Spike Duplicate (MSD) are used to monitor the performance of the analytical process on an environmental sample. An aliquot of a sample is supplemented ("spiked") with known concentrations of the compounds of interest and then carried through the sample preparation and analysis process. The concentrations of the spiked compounds are then compared to the concentration that was added, and is expressed as a "percent recovery". Matrix spikes are often prepared in duplicate; the difference between the recovered concentrations is reported as the "relative percent difference" (RPD).

5.2.4 Surrogate:
A surrogate is a compound that is added to every sample prior to extraction in order to monitor the accuracy of the extraction and analysis. These are compounds that are not normally found in environmental samples but are chemically related to the compounds of interest, and so behave in a similar fashion. Surrogate recovery failure indicates a problem with the process or a matrix interference.

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5.2.5 Limit of Detection (LOD) samples are aliquots of deionized water that are spiked at or just above the calculated Method Detection Limit (MDL), to verify that that the analyte can be detected at those levels. The LOD must be verified quarterly on every instrument for DoD (or annually for NELAP compliance). See the QA SOP 4.4 'Method Detection Limits' for details.

5.2.6 Limit of Quantitation (LOQ) samples are aliquots of deionized water that are spiked at 1-2x the lowest calibration standard and used to verify the accuracy of the procedure at the reporting limit. The LOQ must be determined quarterly on a single instrument

## 6.0 EQUIPMENT & MAINTENANCE

Top-loading (2 decimal) balance
Ultrasonic extractor
Boiling flasks, 500mL, flat bottom
Funnels
K-D concentrators & receivers
Water bath

Broken glassware should be given to the Group Leader for disposal or repair.

Operator Manuals for the sonicators can be found in LIMS, under the SOPs > Equipment Manuals > Sonicator link. See the 'Sonicator Tuning' SOP for details.

## 7.0 INTERFERENCES & TROUBLE-SHOOTING

7.1 If the extracts are allowed to go dry during concentration, the lightest analytes may be lost or thermally labile analytes may be degraded, introducing a low bias.

7.2 If the extract stops refluxing during concentration and no volume change is observed over about 10 minutes, the extract should be removed from the water bath and brought up to a higher final volume. Once the extract volume has been reduced as far as it will go, additional heating will only lead to loss of light weight analytes or degradation of thermally labile analytes.

7.3 If a sample contains a large portion of clay, charcoal or other adsorbant matrix, low surrogate recoveries may be observed.

7.4 Storing the extracts at 4°C helps reduce solvent loss due to evaporation and minimizes the resulting high biases on sample results and surrogate recoveries.

7.5 If, upon the addition of sulfuric acid, the extracts turn a dark, brick-red and exhibit a much more exothermic reaction than normal (ie: the extract explodes out of the scintillation vial), the samples should be re-extracted as this effect has been observed when acetonitrile was inadvertently used in place of hexane.

7.6 If, upon the addition of sulfuric acid, the acid layer becomes large (approximately 2-3 times larger than the normal 10mL) and turns a bright cherry-red, the samples should be re-extracted to avoid interference or degradation of the analytical column. This effect has

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been observed when the sample is exchanged into hexane before the sample has concentrated below 15mL, causing acetone to remain in the sample post-exchange.

## 8.0 PROCEDURE

### 8.1 SAMPLE PREPARATION

8.1.1 Verify that the balance has been calibrated earlier in the day, using weights that encompass the entire mass that will be placed on the balance (total sample weight plus the container weight). If it has not, calibrate it before proceeding. If it has not, calibrate it before proceeding.

8.1.2 Select enough pre-cleaned certified 8oz jars for the samples (up to twenty) plus a Blank, LCS, MS, and MSD.

8.1.3 Label the jars with the laboratory sample number for each sample and QC sample.

8.1.4 Place the first jar on the scale and tare the scale.

8.1.5 Verify the label on the jar against the label on the container submitted by the client.

8.1.6 In the appropriate analysis or Soil Aliquot benchbook, write the laboratory sample number and bottle letter, and analysis.

8.1.7 If the sample was submitted in a brass or steel core/sleeve, use a disposable wooden spatula (for sand or light soils) or single-use metal spatula (for heavier, claylike soil) to remove and discard the top ~1cm of sample.

Discard any leaves, twigs, large stones, etc and take a visually representative aliquot of each sample. Document your observations and actions (ie: “discarded leaves & twigs”) in the prep log.

**Composites**: If a client requests a ‘composite’, determine what weight of each discrete sample will be needed to make somewhat more composite than needed. Following the SOP “Subsampling & Compositing” which is posted near the balance, weigh out exactly the same amount of each discrete, and document these weights in the soil aliquot log.

**Concrete**: If the sample is known to be concrete or containing concrete, DO NOT use 1:1 Dichloromethane:Acetone. Just use Dichloromethane. Concrete contains Limestone, which forms diacetone alcohol in a base-catalyzed aldol condensation. Diacetone alcohol shows up as a large peak after the solvent peak and can cause interferences with target analytes.

8.1.8 Using the same spatula, thoroughly homogenize the next several cm of sample then weigh out 30 ± 0.5g of this homogenized fraction.

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8.1.9 Record the weight to the nearest 0.01g in the benchbook.

8.1.10 Discard the wooden spatula or place the metal spatula in the bin to return to dishwashing.

8.1.11 Repeat Steps 8.1.4 through 8.1.10 for each sample.

8.1.12 For the method blank and laboratory control sample (or BS/BSD), weigh out 30 ± 0.5g of kiln-fired, solvent-rinsed granular sodium sulfate into a pre-cleaned certified 8oz glass jar.

8.1.13 For the matrix spike and matrix spike duplicate, select a sample so that matrix QC is rotated throughout the laboratory's clients, so that no one client's samples predominate over a period of time.

8.1.13.1 Weigh out just over 90g of sample into a pre-cleaned 16 oz wide mouth glass jar.

8.1.13.2 *Thoroughly* homogenize the sample, then weigh out three 30g (± 0.5g) portions into the appropriate pre-cleaned 8oz jars labeled with the sample number and "MSS", "MS", and "MSD" respectively. Record the sample weights to 0.1g.

8.1.14 To the LCS, MS, and MSD, add 1.0mL of the 16/60 SPIKE Spike Solution. See Appendix 2 for instructions on preparing the Matrix Spiking Solution.

Document the LIMS ID of the spike and the volume added in the benchbook.

8.1.15 To every sample, including each batch QC sample, add 1.0mL of the PCB/PESTSURR Surrogate Solution. See Appendix 2 for instructions on preparing Surrogate Solution.

Document the LIMS ID of the surrogate and the volume added in the benchbook.

8.1.16 Mix each sample, including the MS and MSD, with enough (20 to 100 grams) granular anhydrous sodium sulfate to make the soil free flowing.

*Method Modification:* EA-Berkeley follows the more recent method (3550C) which requires that the surrogate and spike solutions be added to the samples prior to drying.

8.1.17 Add at least 100mL of 1:1 Dichloromethane/Acetone to each jar, completely covering the sample.

**Concrete**: If the sample is known to be, or contain concrete, DO NOT use 1:1 Dichloromethane:Acetone. Just use Dichloromethane. Concrete contains Limestone, which forms diacetone alcohol in a base-catalyzed aldol condensation. Diacetone alcohol shows up as a

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large peak after the solvent peak and can cause interferences with target analytes.

**8.2 FILTER FUNNELS & K-D CONCENTRATOR ASSEMBLY**

8.2.1 Using solvent-rinsed, baked glass wool, place a glass wool plug in the bottom of each funnel.

8.2.2 Rinse the walls of the funnel and the wool with dichloromethane, collecting the rinsate in a waste jar.

8.2.3 Add approximately 40g of baked granular or powdered sodium sulfate to each funnel (powder is preferred for soil with fine sediments that would easily pass through the granular version).

8.2.4 Place the funnels in the bottom rack and rinse the funnel with dichloromethane, again collecting the rinsate in the waste jar.

8.2.5 Rinse each K-D concentrator with three aliquots of dichloromethane, discarding the rinsate into a waste-solvent jar, and then hang the K-D upside-down in the top rack to drain.

8.2.6 Rinse each receiver with three aliquots of dichloromethane, discarding the rinsate into a waste-solvent jar. Immediately connect each receiver to a K-D.

8.2.7 After receivers have been added to each K-D, use plastic clips to secure the two pieces together.

8.2.8 Label each K-D *and* receiver with the sample ID and analysis, then replace in the top rack right-side up.

8.2.9 After all concentrators have been assembled and labeled, add a solvent-rinsed boiling chip to each.

8.2.10 Discard the waste dichloromethane in the jars into the waste solvent jug.

8.2.11 Reverse the filters and concentrators so that the filters are on the top rack and the concentrators are on the bottom rack.

8.2.12 If the water baths have not been turned on yet, make sure each is filled with DI water and turn the dial to setting 10. Allow baths to heat while you filter the extracts.

**8.3 EXTRACTION PROCEDURE**

8.3.1 Verify that the receiver is securely attached to the K-D concentrator.

8.3.2 Place the sample into the sonicator so that the probe tip lies below the surface of the solvent just above the solid layer.

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8.3.3 Sonicate for 3 minutes, with the sonicator set at a duty cycle of 50% and the output control set to full power.

8.3.4 Filter the extract through the appropriate filter funnel into the K-D concentrator.

8.3.5 Add a second 100mL portion of the solvent to the sample; repeat the extraction and filter as above, combining the 2nd portion with the original extract.

*Note:* Rotate the jar between solvent aliquots to make sure that ultrasonic energy is breaking up the solid matter and no clumps remain (typically at the union between the bottom of the jar and the walls of the jar).

8.3.6 Perform a third extraction in the same manner and filter, combining the 3rd portion with the previous extracts.

8.3.7 Pour the entire sample into the filter funnel and rinse the jar with a 25mL aliquot of Dichloromethane into the filter funnel. Collect the rinsate with the extract.

8.3.8 Allow the funnel to drain completely, then use dichloromethane to rinse around the top of the funnel (~25mL) 2 more times, washing any remaining extract through the funnel into the concentrator and again allow the filter to drain completely.

8.3.9 While the filters are draining, rinse each three-ball Snyder column at least 3 times with dichloromethane, then allow to drain by placing at edge of hood, with tip pointing outward to indicate that it has been rinsed.

8.3.10 After the filters have completely drained, remove the funnels from the rack and dump the sodium sulfate into the drying tray under the hood.

**8.4 CONCENTRATION PROCEDURE**

8.4.1 Add water to any hot water bath that is not completely full.

8.4.2 Verify that the dial setting is at '10' and that the water bath is boiling.

8.4.3 Verify that each K-D concentrator contains 1 to 2 clean boiling chips. Attach a rinsed 3 ball Snyder column to each K-D concentrator.

8.4.5 Place each K-D concentrator on a boiling water bath so that the receiver is immersed in the hot water to just below the joint of the receiver and K-D. If the receiver is not immersed far enough, the extract will take longer to concentrate. If the receiver is immersed past the joint, any water leaking into the joint will contaminate the extract..

8.4.6 The solvent should immediately begin boiling. If it does not, the concentrator should immediately be removed from the water bath and additional boiling chips should be added to the receiver. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood.

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8.4.7 Concentrate the extract to approximately 15 mL then exchange solvents

*Note:* Reducing the volume to only 25 mL or so prior to solvent exchange may result in incomplete elimination of the acetone and cause instrument problems.

**8.5 SOLVENT EXCHANGE**

Because the Electron Capture Detector used in the PCBs analysis is sensitive to chlorine (and therefore Dichloromethane), the Dichloromethane must be exchanged for hexane.

8.5.1 After the extract volume has been reduced to 10-15mL, but while the extract is still boiling, add 15 - 20 mL of Hexane through the top of the Snyder column.

8.5.2 Concentrate to about 25 mL and again add 15 - 20 mL Hexane.

8.5.3 Concentrate to between 5 - 10 mL, allowing the extract to cook for a minimum of 5 minutes after the second exchange to ensure a complete exchange has taken place.

8.5.4 When the extract volume in the receiver is about 5 mL, remove the K-D apparatus from the water bath and let it drain. This should result in a final volume of 6 to 8 mL.

*Note:* The MS and MSD should be concentrated to the same final volume as the Matrix Spike Sample.

8.5.5 Let the extract cool to room temperature.

8.5.6 Select enough pre-cleaned 40mL VOA vials for the entire batch and label them (both vial and cap) with the sample number. Line them up in numerical order.

8.5.7 Remove the Snyder column, remove the plastic clip and dry the joint with a ChemWipe.

8.5.8 Carefully loosen the K-D/ receiver joint, remove the K-D concentrator and place the receiver in the metal rack *in numeric order.*

8.5.9 Adjust the volume to 25 mL with Hexane.

8.5.10 Record the final volume of extract in the benchbook.

8.5.11 Bring the metal rack over to the vortex mixer and, beginning with the batch QC, use the vortex mixer to homogenize the extracts.

8.5.12 Immediately transfer each extract to the 40mL VOA vial labeled (both cap and vial) with that sample ID, verifying that the sample numbers match before each transfer.

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**8.6 ACID CLEAN UP**

8.6.1 Add 10 mL of concentrated Sulfuric Acid and agitate for 60 seconds with a Vortex mixer. As an alternative to using the Vortex mixer, samples may be placed inside of the VOA box labeled as “PCB Shaker Box”. Close and secure the lid, then vigorously shake the samples for a minimum of 1 minute.

8.6.2 Separate the phases by centrifugation for 2 minutes or by allowing the solution to stand a minimum of 10 minutes. If the top (solvent) phase is not clear, pipette it into a clean 40mL VOA vial labeled with that sample number and repeat the acid cleanup.

8.6.3 Select enough pre-cleaned scintillation vials for the entire batch. Label them (cap and vial) with the sample numbers, batch number, and “PCB”.

8.6.4 Pipette approximately 5 mL of the hexane layer (top layer) of each acid-cleaned extract into the scintillation vial labeled with that sample number, verifying that the sample numbers match before each transfer. Use care to not touch the pipette to the acid layer, and to not accidentally transfer the acid layer by mistake.

8.6.5 Cap the scintillation vials, making sure that the sample number on the cap matches that on the vial. Place the scintillation vials *in order* in a box labeled with batch number, “PCB”, and date.

8.6.6 Place the box in the Delfield refrigerator # 9 - making sure that the temperature of the refrigerator has been verified and recorded for that day. Storing the extracts at 4°C will help reduce solvent loss due to evaporation, which would lead to high biases on sample results and surrogate recoveries.

8.6.7 Cap the 40mL VOA vials, containing the remaining extract and sulfuric acid, and place them in a separate box labeled with the batch number, “PCB”, and date. Store these for 40 days, as a backup to the fraction in the scintillation vials.

8.6.8 Complete the laboratory notebook entry. Make sure all times, reagent manufacturers and lot numbers, working and source standard LIMS ID numbers, as well as responsible chemists' initials have been **legibly** recorded in the benchbook entry and on the batch QC checklist.

8.6.9 Have the entry peer reviewed and documented on the appropriate checklist. After review, copy the entire page of the benchbook then scan the checklist, job sheets and benchbook page into LIMS. “Kill" the batch in LIMS by updating the status of the batch to "DONE".

**9.0 DOCUMENTATION**

9.1 **Benchbooks**: Every extraction must be completely documented in the appropriate benchbook. Any changes must be made with a single line through the incorrect entry and initialed and dated by the chemist making the change. The benchbook entries must include the following:

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Sample number, accompanied by the unique container identifier (A-> Z)
Initial sample weight
Final sample volume
LIMS S# and volume used for all surrogate and spike standards
Manufacturer and lot# for all solvents, reagents, and filters
ID of the balance used to weigh out the samples
Observations concerning unusual sample appearance, odor, behavior
Errors during extraction (spilled, possibly 2x spiked, etc.)

9.2 **LIMS**: All extraction weights, volumes, and S# must be entered into the prep entry database. It is very important that the entries are accurate and complete, as LIMS uses these to calculate sample concentrations and spike results.

9.3 **Peer Review**: The benchbook entries and LIMS prep entry must be reviewed by another extraction chemist, an analyst, or Group Leader; the reviewer must sign and date the benchbook.

## 10.0 WASTE DISPOSAL

Waste container must be compatible with the type of material being stored and must be in good condition (ie: not rusted or cracked).

Each container must be clearly labeled "Hazardous Waste", with a description of the composition, physical state of the waste ('Liquid/Solid'), the type of hazard (Flammable Solvent, Toxic Solvent, etc. ), and the date that the first contents are added to the container

All containers holding chemical waste (liquid or solid) must be kept closed/capped except when necessary to add or remove waste.

After the extraction steps are completed, the spent sample and any waste solvent must be properly discarded by:

a. Sample jars or sleeves must be returned to the coldroom for later disposal by Sample Control.

b. After extraction, the expended soil and sodium sulfate remaining in beakers or filter-funnels should be transferred to a drying tray under a hood and allowed to dry.

c. After drying, the solid waste may be accumulated in the 55 gallon "Residual Soil" open-head steel drum located in the ventilated closet in the extraction lab for up to 1 month, after which the drum is be transferred to the 90-day accumulation area of the garage.

d. Dichloromethane waste may be accumulated in a 1 gallon plastic container, labeled "Hazardous Waste – empty daily". This waste should be transferred daily to the "Methylene Chloride/Chlorinated Solvents" 55 gallon tight-head steel drum in the waste room.

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e. Waste Hexane is collected in the poly "Flammable Mixed Solvents" container located in the Flammables cabinet. This may be accumulated for up to 9 months, after which it must be transferred to the Flammable Solvents waste 55-gallon 'tight head' steel drum, located in the flammables cabinet in the waste room.

f. Sample extracts, including the vials containing sulfuric acid, should be stored at 4°C for at least 40 days, after which they may be transferred intact into the 'PCB+Pesticide' 55 gallon open-head poly drum, located in the waste room.

g. Vials containing expired standards should be placed in the plastic bucket, containing 'PCB/Pesticides' waste, in the PCB lab. When the plastic bucket is full, but no longer than 9 months after initial accumulation date, the vials are transferred to the "90-day" PCB/Pesticide 55-gallon OH (open-head) waste drum, located in the waste room.

## 11.0 POLLUTION PREVENTION

Prepare only sufficient standard and reagent volume that can be used within the expiration date, to reduce the volume of waste generated by the laboratory and to reduce costs.

## 12.0 REVISION HISTORY

The previous document (revision 13) was changed as follows:

- Throughout document, replaced C&T name & logo with EA
- Section 2: Updated QSM to current version
- Section 8: Updated spike and surrogate addition to be completed prior to drying, to match 3550C.
- Section 9: Added balance ID to documentation requirements

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**APPENDIX_1:** **PCB SOIL EXTRACTION SUMMARY**

| | | |
|---|---|---|
| Sample Weight: | 30g (± 0.5g) | |
| Surrogate Solution: | *PEST/PCBSURR*<br>TCMX & DCB @ 1 µg/mL | Add: 1.0 mL |
| PCB Spike Solution: | *PCB 16/60 SPIKE*<br>AR1016 and AR1260 @ 5 µg/mL | Add: 1.0 mL |
| Extraction Solvent: | 1:1 Dichloromethane/ Acetone | |
| Water Bath Temp: | Boiling | |
| Final Solvent: | Hexane | |
| Final Volume: | 25 mL | |
| Extract Cleanup: | Sulfuric Acid (required) | |

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**APPENDIX_2: COMBINED PESTICIDE (8081) / PCB (8082) EXTRACTION SUMMARY**

| | | |
|---|---|---|
| Sample Weight: | 30g (± 0.5g) | |
| QC Requirements: | LCS, MS, MSD (8082 spike)<br>LCS, MS, MSD (8081 spike) | |
| Pesticide (8081) Spike Solution: | *ISOPEST_SPK* @ 1-10 µg/mL | Add: 0.40 mL |
| PCB (8082) Spike Solution: | *PCB 16/60 SPIKE* @ 5 µg/mL | Add: 1.0 mL |
| Surrogate Solution: | *PEST/PCBSURR*<br>TCMX & DCB @ 1 µg/mL | Add: 0.8 mL |
| Extraction Solvent: | 1:1 Dichloromethane/ Acetone | |
| Water Bath Temp: | Boiling | |
| Final Solvent: | Hexane | |
| Final Volume: | 20 mL<br>Split into two 10mL aliquots; one for 8081 and one for PCB | |
| Extract Cleanup: | Sulfuric Acid (required for PCB - not to be used for 8081<br>Florisil (required for 8081, optional for PCB) | |

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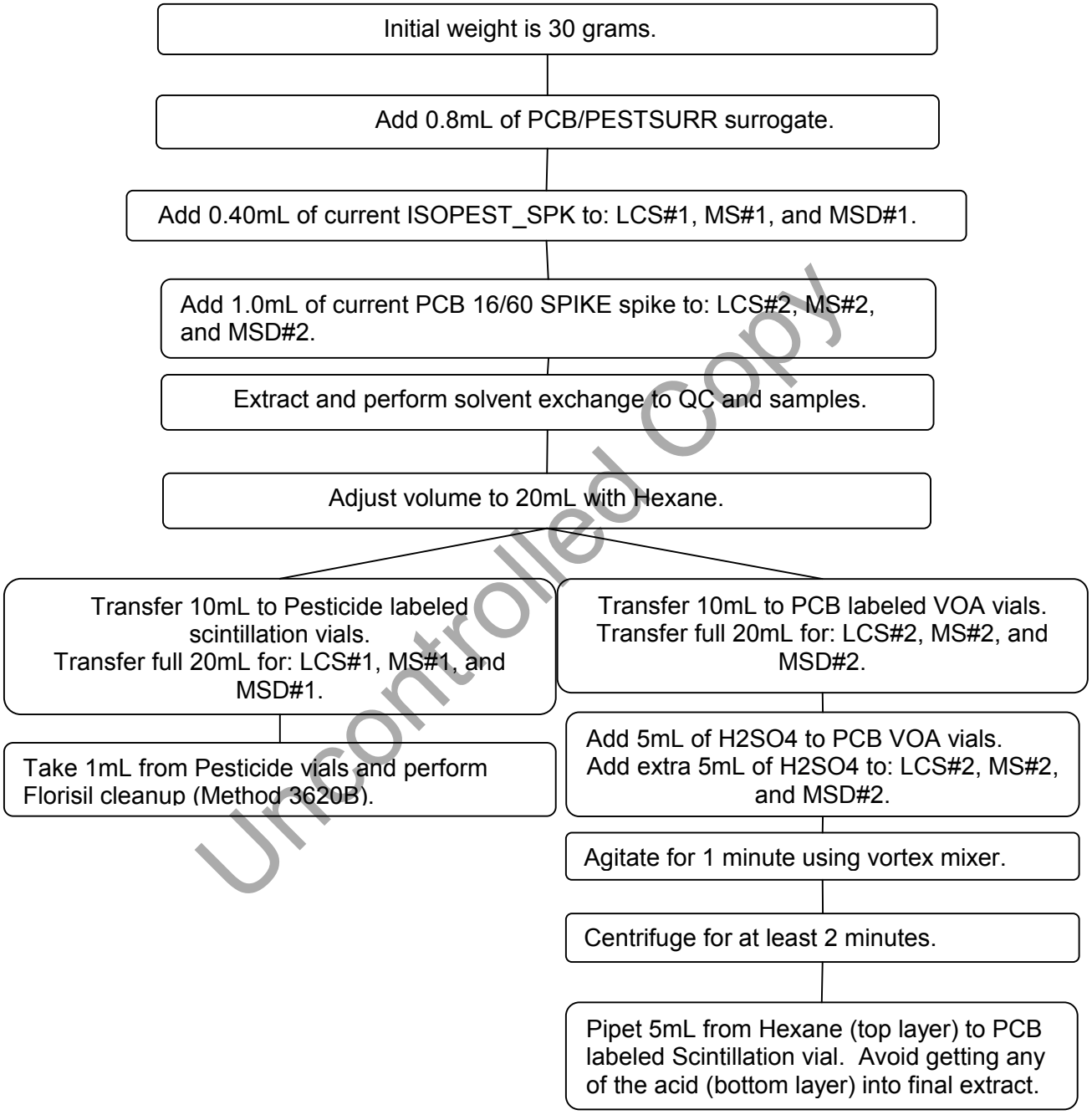
**Flowchart for 8081/8082 combined extraction by method 3550:**

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**APPENDIX_3: STANDARDS & REAGENTS**

**WORKING STANDARDS**

The LIMS name/definition of a working standard is unique to the composition and concentration of the standard but not necessarily unique to the vendor of the source standards. If the concentration of a working standard is changed or a new compound is added to the working standard mix, a new name must be assigned and the information entered in the LIMS standards table before the standard is assigned an S#.

All working standards should be prepared in a water-soluble solvent (acetone or MeOH) so that the spikes and surrogates will be completely miscible in the sample prior to extraction.

Working standards expire 180 days after preparation from the stock standard. *Note:* The expiration date of the working standard *must not* exceed the expiration date of any of the stock standards from which it was made. If any of the stock standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards.

Document the preparation of all working standards in the standards prep bench book and in the LIMS Standards database; LIMS will assign an S#. Include the name and/or description of the standard being prepared, prep date, LIMS S# and concentration of the source standard(s) used, volume of source standard used, final volume, final concentration, expiration date, and prep chemist's initials in the benchbook entry. Scan the prep benchbook entries into LIMS then verify that the scanned copy is legible.

Label the standard bottle with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

Store in the freezer at < -10°C.

**PROCEDURE:**

1. Prepare all working standards in a water-soluble solvent (MeOH, or 1:1 MeOH:DCM) using Class-A volumetric flasks, and the volumes and standards listed below.
2. Bring the source standards to room temperature before using them to make the working standards.
3. Place the ampules in the sonic bath rack and sonicate for about 5 minutes; this step is important to make sure all of the compounds are in solution!
4. Use the vortex mixer to homogenize each source standards then examine the vials to verify that the standards are completely in solution and that there is no visible precipitate.
5. Select an appropriate Class-A volumetric flask for the final volume listed in the table below. Rinse the flask 3 times with DCM.
6. Partially fill the flask with 1:1 MeOH:DCM.
7. Add the required volume of each source standard needed, as listed in the table below.
8. Brink to the mark with 1:1 MeOH:DCM. Cap and invert the flask at least 3 times to mix, allowing sufficient time for complete mixing with each inversion.
9. Transfer the solution to storage bottles, labeled with the new LIMS S# and expiration date.
10. Cap with a Teflon-lined cap.

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11. Have the new standard analyzed and approved **PRIOR TO USE!** Keep the results of these checks on file.

| Working Standard & Conc. | Final Volume (mL) | Using Source Std | Add Vol. (mL) Source | LIMS S-Name |
|---|---|---|---|---|
| PCB Spiking Solution: Ar1016/1260 @ 5 µg/mL | 200 | 1660_U | 1.0 | PCB 16/60 SPIKE |
| Surrogates: TCMX & DCB 1 µg/mL | 1,000 | PSS | 5.0 | PEST/PCBSURR |

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**SOURCE STANDARDS**

All source standards should be NIST-traceable. Each source standards must be documented in LIMS upon receipt, through the Standards Menu. The LIMS S-name for a source standard is unique to the vendor that the source is obtained from; if a source standard is obtained from a different vendor, a new name must be assigned and the information entered in the LIMS Standards table before the standard is assigned an S#.

Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is two years from date received.

Enter the lot#, date received, and expiration date of each source standard into LIMS immediately upon receipt, using the Standards Menu “Standard Inventory”.

Write the S# and the date received on the ‘Certificate of Analysis’ that accompanied the standard; if the supplier did not provide a certificate, call and request that a copy be emailed. Scan the certificate into LIMS then verify that the scanned copy is legible.

Label each container with the LIMS S# and expiration date.

Store the source standards in the freezer section of Refrigerator # 5, at -10 to -20°C.

| Analytes | Concentration | Supplier & Catalog# | LIMS S- Name |
|---|---|---|---|
| Surrogate (TCMX and DCBP) Source Std | 200 µg/mL | Ultra Scientific # ISM-320-1 | PSS |
| Spiking (Ar1016/1260) Source Standard | 1,000 µg/mL | Ultra Scientific # PPM-8082 | 1660_U |

*Note:* Although EPA 608 does not apply to soil samples, EA-Berkeley uses the same spiking standards for both soil and water and EPA 608 requires that the spiking standard be from a second source than the calibration standard.

**REAGENTS**

The preparation of all reagents, including dilutions into Millipore DI water, must be documented in the reagent prep benchbook. Each prepared reagent must be assigned a unique ID, based on the manufacturer and the date prepared. Each reagent received from an outside vendor should be labeled with the receipt date and expiration date.

Acetone, EM Science, Omni-Solv grade, VWR Catalog# TXEMAX01 161 CUT
Store in a Flammables cabinet for no more than 3 months; after 3 months, aldol condensation products will start forming and interfering with the analysis.

Dichloromethane, EM Science, Omni-Solv grade, VWR Cat# TXDX0837-39CUT
Store at room temperature for up to 1 year

1:1 Acetone/Dichloromethane
Prepare by carefully decanting approximately 2L of acetone into a brown solvent jug containing approximately 2L dichloromethane. Label the bottle with the reagent ID. Store in a Flammables cabinet under the fume hoods for no more than 3 months

Hexane, EMD Omnisolv, HR-GC Grade, VWR Catalog# EM-HX0297-1
Alternative: JT Baker, Ultra Resi-Analyzed grade, VWR Catalog# JT9262-3
Store at room temperature in a Flammables cabinet for up to 1 year.

Sodium Sulfate, Granular Anhydrous, EM Science 99.0%,
VWR Cat# EM-SX0760-20
Sodium Sulfate, Powder Anhydrous, EM Science 99.0%,
VWR Cat# EM-SX0763-30
Store at room temperature for up to 1 year
Kiln-bake at 400°C for 4 hours then cool in desiccator.
Rinse with dichloromethane prior to use.

Sulfuric Acid, Concentrated, 90.5-92.7% by volume,
JT Baker, VWR catalog # JT9691-3
Store in a Corrosives cabinet for up to 5 years

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**APPENDIX_4: CALCULATIONS**

**Make a Working Standard from a Source (Stock) Standard:**
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where:
Vss = Volume of Source Std (mL) needed to make Working Std
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (ug/mL) of the Working Standard
Css = Concentration (ug/mL) of the Source Standard

*Note:* If the volume to be added is less than 1ąL, make an intermediate dilution by diluting the source standard 1:10 (1mL + 9 mL), then use 10 times as much of this intermediate. For example, if you need to add 0.3 ąL of the source standard, dilute 1.0mL of the source standard into 9mL of the solvent, then use 3.0 ąL of this dilution to make your final working standard.

**Spiking Batch QC for Waters**
Use the following to determine the volume to be used for spiking MDL's or if a client requests that batch QC be spiked at a different level:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where:
Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/L)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Volume of Sample (mL)

**Spiking Batch QC for Soil**
Use the following to determine the volume to be used for spiking MDL's or if a client requests that batch QC be spiked at a different level:

Vws (mL) = Cf / (Cws * pdf * 1000)

Where:
Vws = Volume (mL) of Working Standard to use for spike
Cf = Final Concentration in sample (ug/Kg)
Cws = Concentration (ug/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Mass of Sample (g)

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Curtis & Tompkins, Ltd

# POLYCHLORINATED BIPHENYLS (PCBs)
## By GC-ECD Analysis Methods
## EPA 8082, EPA 8082A, EPA 608 & TO-10A

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Cheryl Bowden<br>Operations Manager | Cheryl Bowden | 2/18/15 |
| Teresa Morrison<br>QA Director | Teresa Morrison | 2/18/15 |
| **Reapproved:** | | |
| Teresa Morrison<br>QA Director | Teresa Morrison | 3/18/16 |
| Teresa Morrison<br>QA Director | Teresa Morrison | 4/25/17 |
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Curtis & Tompkins, Ltd

# POLYCHLORINATED BIPHENYLS (PCBs)

By GC-ECD Analysis Methods
EPA 8082, EPA 8082A, EPA 608 & TO-10A
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# POLYCHLORINATED BIPHENYLS (PCBs)

## 1.0 SCOPE

This document describes the analysis and quantitation of PCB's that have been extracted from liquid samples, solid matrices, and wipes or other miscellaneous matrices. The analysis is performed by first injecting the hexane extract into a capillary column GC to separate the components into discreet compounds. An Electron Capture Detector detects those components that are halogenated compounds and the analyst uses pattern recognition to identify the Aroclor mixtures. Analysis for the quantitation of the discreet PCB congeners is described in a separate procedure.

Sample concentrations are reported in micrograms per liter (µg/L) for water samples or micrograms per kilogram (µg/Kg) for solid samples. See Appendix 8 for the compound list and reporting limits.

EPA 8082 was written by the EPA's Office of Solid Waste with additional guidance for surface water and ground water, as opposed to EPA 608 which was written by the EPA's Office of Water specifically for wastewater. EPA 608 may also be requested for groundwater samples if the client is planning to discharge the water, with or without additional treatment, into a wastewater stream or into naturally occurring surface waters (bay or river). See Appendix 13 for EPA 608 requirements.

Method TO-10A is designed for measurement of low level PCBs in ambient air, by collection on polyurethane foam (PUF). See Appendix 14 for discussion and reporting limits.

## 2.0 REFERENCES

*Analytical Methods:*

EPA 8082, Polychlorinated Biphenyls (PCB's), SW-846, Dec 1996
EPA 8082A, Polychlorinated Biphenyls (PCB's), SW-846, Feb 2007
EPA 8000B, Determinative Chromatographic Separations, SW-846, Dec 1996
EPA 8000C, Determinative Chromatographic Separations, SW-846, Mar 2003
EPA 608, Organochlorine Pesticides & PCBs, 40CFR136 Appendix A

*Extraction & Cleanup Methods:*

EPA 3500B, Organic Extraction and Sample Preparation, SW-846, Dec 1996
EPA 3500C, Organic Extraction & Sample Preparation, SW-846, Feb 2007
EPA 3510C, Separatory Funnel Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846, Dec 1996
EPA 3540C, Soxhlet Extraction, SW-846, Dec 1996
EPA 3550B, Ultrasonic Extraction, SW-846, Dec 1996
EPA 3550C, Ultrasonic Extraction, SW-846 Feb 2007
EPA 3660B, Sulfur Cleanup, SW-846, Dec 1996
EPA 3665, Sulfuric Acid Cleanup, SW-846, Dec 1996
EPA 3580A, Waste Dilution, SW-846, Dec 1996
EPA 608, Organochlorine Pesticides & PCBs, 40CFR136 Appendix A

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*Related References:*
C&T SOP XLAB 2.2.5, PCBs by Continuous Liquid-Liquid Extraction
C&T SOP XLAB 3.2.3, PCBs by Sonication Extraction
C&T SOP XLAB 4.1, Waste Dilution for PCBs in Oil
C&T SOP XLAB 8.2, TO-10A Extraction of PCBs from PUFs
C&T SOP SVOC 3.0, Electron Capture Detectors
Varian Ni 63 ECD Safety Manual
C&T SOP QA 1.4, Balance Calibration Check & Maintenance
C&T SOP QA 1.5, Calibrating & Maintaining Temperature Controls
C&T SOP QA 1.6, Pipette Calibration Check Procedures
C&T SOP QA 4.1, Establishing Control Limits
C&T SOP QA 4.4, Determining Method Detection Limits (MDL) & Limit of Detection (LOD)
C&T SOP QA 4.6, Limit of Quantitation (LOQ)
C&T SOP QA 9.6, Insuring Compliant Manual Integrations
TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2, October 2010
DoD/DoE Consolidated QSM, Version 5.0, July 2013
40CFR136 Methods Update Rule (MUR), Table II, May 2012
SW-846 Chapter 4, Table 4-1, Update IV, Feb 2007

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## 3.0 BOTTLES, PRESERVATION & HOLDING TIME

Bottles: Water samples should be received in amber glass bottles; plastic bottles are not appropriate for this analysis. Soil samples should be received in brass sleeves or glass jars.

Preservation: No chemical preservation.
Store samples and extracts at > 0.0 to 6.0 $^{o}$C

Holding time:

| | |
|---|---|
| $H_2O$: | 7 days from collection to extraction*. |
| Soil: | 14 days from collection to extraction. |
| Extract: | 40 days after extraction. |

Arizona: 1 year from collection to extraction
40 days from extraction to analysis
(see Information Update #114)

40CFR136 Table II (2012 MUR) for waste water – EPA 608:
1 year from collection to extraction
1 year from extraction to analysis

SW-846 Update IV (EPA 8082A): Holding times for soil and water have been updated to “None”, so long as the water sample has no residual chlorine. California regulators do not yet recognize this SW-846 update but other States and the DoD agencies do.

** EPA 608 Method Note:* EPA 608 requires that if the extraction is not begun within 72 hours of sample collection, the sample pH be adjusted to 5.0 – 9.0 SU with NaOH or $H_2SO_4$ to extend the holding time to 7 days. *However,* the 40CFR136 Methods Update Rule of 2012 extended the holding time to 1 year with no requirement to adjust the pH.

## 4.0 SAFETY

Assume that all samples contain hazardous and/ or potentially toxic chemicals and should be handled with care. Safety glasses, gloves, and a lab coat should be worn whenever handling samples, extracts, reagents, or standards.

## 5.0 QC REQUIREMENTS & DEFINITIONS

For samples associated with site-specific project plans developed by the client, the requirements of those plans supersede the requirements listed in this document.

### 5.1 QC Requirements

| QC Element | Frequency: | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Surrogate | Every sample and batch QC sample<br><br>*EPA 608:*<br>Not discussed; use in-house requirement | %Recovery within statistical limits.<br><br>*QSM 4.2/5.0:*<br>%Recovery within QSM control limits. | Low recovery:<br>Reanalyze affected sample. If still out, re-extract sample.<br><br>High recovery:<br>If due to obvious coelution, flag and narrate without reanalysis. Report 'ND' samples and re-extract any others |
| Method Blank | 1 for every batch of 20 or fewer samples | <1/2 RL<br><br>*QSM 4.2/5.0:*<br><1/2 RL | Report samples that are ND or >10x contamination; rerun all others |
| Laboratory Control Sample (LCS)<br><br>- OR -<br><br>Blank Spike (BS)/ Blank Spike Duplicate (BSD) | 1 for every batch of 20 or fewer samples<br><br>(if insufficient sample volume was submitted for an LCS/MS/MSD, a blank spike and blank spike duplicate may be analyzed instead)<br><br>*EPA 608:*<br>LCS for every 10 or fewer samples | %Recovery & %RPD within statistical limits<br><br>*QSM4.2/5.0:*<br>%Recovery within QSM control limits.<br>%RPD within statistical limits.<br><br>*EPA 608:*<br>%Recovery & %RPD within statistical limits | Low recovery:<br>Reanalyze all samples.<br><br>High recovery:<br>Reanalyze samples with results ≥LOQ. Report results <LOQ.<br><br>High RPD:<br>Report ND samples, rerun all others |

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| QC Element | Frequency: | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) | 1 pair for every batch of 20 or fewer samples (if sufficient sample volume was submitted) | *C&T Requirements:* %Recovery & %RPD within statistical limits<br><br>*QSM4.2:*<br>%Recovery within QSM LCS limits.<br>%RPD within statistical limits.<br><br>*QSM5.0:*<br>%Recovery within QSM LCS limits.<br>%RPD ≤ 30 | Flag results as possible matrix interference. |
| Initial Calibration Curve | Whenever CCVs will not pass, after major instrument maintenance, or when new equipment is installed.<br><br>Low pt ≤ RL<br><br>*EPA 8082/8082A:*<br>AR1016/Ar1260 require minimum of 5pt (6pt for quadratic) ICAL. Single pt for all others.<br><br>*QSM4.2/5.0:*<br>5pt (minimum) required for any Aroclor; no 1pt allowed<br><br>*EPA 608:*<br>3pt (minimum) required for any Aroclor; no 1pt allowed | 5 points minimum (or 6 points for quadratic), with low point at or below the RL.<br><br>Avg RF %RSD ≤ 20,<br><br>If using linear/quad. regression, the correlation coefficient must be r ≥0.995 (or $r^2$ ≥0.99)<br><br>*QSM 4.2/5.0:*<br>Same as above.<br><br>*EPA 608:*<br>Avg RF %RSD ≤ 10<br>Linear/Quad: r ≥0.995 | Perform instrument maintenance and recalibrate |
| Initial Calibration Verification (ICV) | After ICAL curve and before samples | *EPA 608/ 8082:*<br>%D ≤ 15<br><br>*EPA 8082A:*<br>%D ≤ 20 | Perform maintenance and rerun; if still out, reanalyze curve. |

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| QC Element | Frequency: | Acceptance Limits | Corrective Action |
|---|---|---|---|
| | | *QSM 4.2/5.0*:<br>%D ≤ 20 | |
| Continuing Calibration Verification (CCV) | AR1016/1260 @ beginning of sequence, before any samples, then after every 12 hours or 10 field samples (including the MS/MSD), and at end of sequence.<br><br>Other Aroclors must bracket samples known to contain, and being quantitated for, those PCBs.<br><br>*QSM4.2/5.0*:<br>Ar1016/160 at beginning of sequence, after every 10 samples (excluding MB, LCS/ BS/BSD) regardless of time-frame, and at end of sequence. Other Aroclors to bracket samples know to contain those PCBs. | *EPA 608/ 8082*:<br>%D ≤ 15<br><br>*EPA 8082A*:<br>8082A: %D ≤ 20<br><br>*QSM 4.2/5.0*:<br>%D ≤ 20 | Perform maintenance and rerun; if still out, reanalyze curve.<br><br>For reporting:<br>Low recovery: Reanalyze all samples.<br><br>High recovery: Reanalyze samples with results ≥RL. Report results <RL.<br><br>See Section 8.2 below for additional guidance. |
| Internal Standards | Not applicable to this analysis | | |
| Method Detection Limit (MDL) Study | When new instrument installed, or significant change made to process | ≤1/3 LOQ; see MDL SOP for additional details | See MDL SOP |

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| QC Element | Frequency: | Acceptance Limits | Corrective Action |
|---|---|---|---|
| Limit of Detection (LOD) | Analyze on each instrument, quarterly (DoD QSM requirement) | Spike at 2 - 4x MDL and <LOQ;<br><br>Each analyte detected (pattern must be identifiable) | See MDL SOP |
| Limit of Quantitation (LOQ) | Analyze on one instrument, quarterly (DoD QSM requirement) | Spike at 1-2x the RL<br><br>Recovery within 50-150%. | See LOQ SOP |
| Demonstration of Capability (IDOC) | Each new analyst; annually thereafter | 4 consecutive LCS within recovery and RPD limits, or acceptable PT results | Retrain analyst and reanalyze DOC. |

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## 5.2 QA/QC Definitions

5.2.1 **Initial Calibration** curve is a series of standards that establishes the quantitation range of the instrument and the relationship of concentration to instrument response.

5.2.2 **Initial Calibration Verification (ICV)** standard is a “second source” standard that is obtained from a different manufacturer than the standards used for the initial calibration curve. The ICV is used to verify that both the working and source standards used for calibration were prepared correctly.

5.2.3 **Continuing Calibration Verification (CCV)** standards are analyzed throughout the sequence to demonstrate that the curve is still valid.

*Note:* The US Army Corp of Engineers recommends that the CCV’s be prepared from the same source standards as the ICAL curve in order to better differentiate between changing instrument response and simple differences between standards.

5.2.4 **Batch QC:** The following quality control (QC) samples must be prepared in the same manner as the analytical samples at a rate of once per twenty or less samples.

5.2.4.1 **Method Blank (MB):** An aliquot of deionized water is extracted and analyzed with each batch of 20 or fewer samples to demonstrate that the glassware and reagents are free of contamination.

5.2.4.2 **Blank Spike / Blank Spike Duplicate (BS/BSD):** A blank spike and a blank spike duplicate are aliquots of deionized water to which a known amount of analyte is added. These spikes are then extracted and analyzed to

demonstrate that the procedure is accurate (measured by the *recovery*) and precise (measured by the *RPD*) in the *absence* of matrix interferences.

5.2.4.3 **Matrix Spike (MS) / Matrix Spike Duplicate (MSD):** An MS and MSD are aliquots of real world samples to which a known amount of analyte is added prior to sample extraction; the spikes are then extracted and analyzed to demonstrate the accuracy and precision of the process on environmental samples, which may include matrix interferences.

5.2.5 **Method Detection Limit (MDL):** An MDL study is required when new equipment is installed or significant changes have been made to a procedure. For this study, a set of at least 7 low-level blank spikes are prepared and analyzed to demonstrate that the sample preparation and analysis procedures are adequate to meet required reporting limits. See the QA SOP "Determining MDLs" for details.

5.2.6 **Limit of Detection (LOD)** samples are aliquots of deionized water that are spiked at or just above the calculated MDL, to verify that that the analyte can be detected at those levels. The LOD must be verified quarterly on every instrument for DoD (or annually for NELAC compliance). See the QA SOP 4.4 'Method Detection Limits' for details.

5.2.7 **Limit of Quantitation (LOQ)** samples are aliquots of deionized water that are spiked at 1-2x the lowest calibration standard and used to verify the accuracy of the procedure at the reporting limit. The LOQ must be determined quarterly on a single instrument for DoD (or annually for NELAC). See the QA SOP 4.6 'Limit of Quantitation' for details.

**6.0 EQUIPMENT** (see Appendix 4 for instrument specifications & conditions)

Hewlett-Packard Gas Chromatographs:

GC: Hewlett-Packard Model 5890
Detector: Hewlett-Packard Model 19233 or G1223A, Electron Capture Detector
Autosampler: Hewlett-Packard Model 7673A or 767B

Varian Gas Chromatographs:

GC: Varian Model CP-8400
Detector: Varian 02-001972-00, Electron Capture Detectors:
Autosampler: Varian Model CP-8400

Columns:

The following columns are typically used because they demonstrate superior separation of the PCB congeners. Others are available and may be used for the Aroclors analysis, so long as the pertinent information is documented in the maintenance log.

Front (Channel A): Restek Rtx-CLP Pesticides, 30m x 0.32-mm I.D., 0.5 µm df
Back (Channel B): Restek Rtx-5, 30m x 0.32-mm I.D., 0.5 µm df

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Data acquisition and processing software: EZChrom, version 3.1.7

## 7.0 ROUTINE MAINTENANCE

Generally, the septum, injection liner, and blue seal should be changed every couple of days, before analysis of an opening Continuing Calibration Verification (CCV). The injection liner is a 4mm splitless single goose-neck liner. The column may need to be trimmed (usually a minimum of 15cm) if oily or highly contaminated samples were analyzed in the previous sequence; additional trimming of the column may be necessary, depending on the results of the analysis of the CCV standard. See Appendix 5 for additional maintenance procedures & details.

## 8.0 DAILY SEQUENCE

Each sequence should begin with an instrument blank followed by a Continuing Calibration Verification (CCV) standard. Once CCV has passed acceptance criteria (see Appendix 10 for criteria), sample extracts may be added to the instrument sequence. Additional instrument blanks and CCV's must be analyzed after every ten field samples, not including known batch QC samples, and at the end of the sequence. The concentration of the CCV's should be varied within the calibration range, excluding the highest or lowest points. Instrument blanks may be included after each CCV bracket to demonstrate that instrument contamination is not contributing to the reported results.

All solutions are analyzed on both columns in the following sequence:

Hexane Blank
CCV – Ar1016/1260
Hexane Blank (optional)
Method Blank
Blank Spike
Blank Spike Duplicate
10 Samples
CCV – Ar1016/1260
Hexane Blank (optional)
Up to 10 Samples
CCV – Ar1016/1260
Hexane Blank (optional)
Up to 10 Samples
CCV

The sequence must end with CCV regardless of the number of samples analyzed.

Although the current SW-846 methods allow up to twenty runs between CCV's, C&T typically runs CCV's after every ten samples to meet the additional SW-846 requirement that no more than 12 hours should elapse between CCV's and to reduce the number of reanalyses caused by failing CCV's. Additional samples may be analyzed between CCV's so long as the 12-hour window is met.

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If the instrument will be running unattended or overnight, it is a good idea to load two CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error.

DoD QSM: The DoD QSM requires CCVs to be run after every 10 field samples (excludes batch QC and instrument standards/blanks). If the sequence includes DoD QSM samples, the CCVs immediately bracketing those samples must pass the QSM criteria.

EPA 608: EPA 608 only requires that a CCV be analyzed once daily, however C&T standard practice is to follow SW-846 guidelines as described above.

**8.1 File Naming Conventions**

Various user reports are automatically produced after the run is complete. This automation is based on the type of sample being analyzed, the sample number, the LIMS identification of any associated calibration or spiking standards, the batch number, and any applicable dilution factors. This data must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information and should be written into the sequence as follows:

Date files are named using the Julian date, followed by a dash, and then the run number (e.g. 123-002). These files are written to the G:\ezchrom\Projects\GCxx\Data subdirectory, where xx is the GC number.

Examples of EZChrom method (processing) file names include (where ### is the Julian date):

| | |
|---|---|
| Aroclor 1016 / Aroclor 1260: | pcb-run-###.met |
| Aroclor 1221 / Aroclor 1254: | pcb-ar2154-###.met |
| Aroclor 1248: | pcb-ar1248-###.met |

Examples of other types of file names and paths include:

| | |
|---|---|
| Data files: | G:\ezchrom\Projects\GC16\Data\219-015 |
| Sequence files: | G:\ezchrom\Projects\GC16\Sequence\219.seq |
| Report format: | G:\ezchrom\Projects\GC16\Template\Ar1254-REPORT-04-13-14.rep |

See Appendix_11: Using EZChrom for additional instructions for writing the sequence into the software.

**8.2 CCV (Continuing Calibration Verification)** (See Appendix_10 for Acceptance Criteria):

Begin each sequence with an Aroclor 1016/ Aroclor 1260 CCV ("AR1660" Continuing Calibration Verification) standard to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. Use a standard at one of the mid-levels of the calibration curve; do not use either the highest or lowest point. It is also a good idea to run Aroclor 1254 with samples from a new site, since this compound is also fairly common.

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8.2.1 Decide what CCV standards to analyze, based on the spiking compound used in the batches to be analyzed and keeping in mind that the concentrations should be alternated across the mid-levels of the calibration.

8.2.2 Load CCV standards after every ten samples and at the end of the sequence, excluding batch QC, instrument blanks and other standards in the count. Remember to add any other Aroclor CCVs bracketing extracts that are known to contain non-1016/1260 Aroclors or in which these other Aroclors have been identified.

LIMS identifies samples that are associated with the DoD QSM requirements, other DoD requirements or commercial clients. It identifies all project specific criteria that are reviewed by the analyst before sample analysis.

For sequences containing samples that are not associated with requirements from DoD QSM, if the instrument is running unattended or overnight, it is a good idea to load two CCV standards for each bracket, to reduce the number of samples that have to be reanalyzed due to an injection error. Type in the sequence with an “x” stype for the second CCV so that only the first CCV will automatically process.

*DoD QSM:* If the sequence includes DoD QSM v4.2 or QSM v5.0 samples, the CCVs immediately bracketing those samples must pass the QSM criteria.

8.2.3 Analyze the standards using the same data acquisition method as for the samples, typing “CCV,” before the working standard number, so that LIMS will automatically generate and print a CCV summary, which compares the calculated concentrations from this run to the known concentrations of the standard.

8.2.4 Examine the CCV summary against the criteria listed in Appendix_10 to determine whether the CCV is acceptable.

8.2.5 If the acceptance criteria are not met, examine the integration to verify that the peaks were correctly integrated. Manual integrations must be consistently applied to standards and samples. If manual integrations or baseline corrections are performed, resend the file to LIMS and generate a new CCV summary.

a. If associated samples are being quantitated for a hit of one of the non-Ar1016/1260 PCBs based on a single-point calibration, the bracketing Ar16/60 CCVs on that channel must pass, as the linearity of the calibration is demonstrated by the Ar1016/1260.

b. If a non-Ar1016/1260 PCB is being quantitated from a 5-pt calibration and the bracketing CCVs of that analyte pass, the sample(s) may be quantitated for that analyte regardless of the Ar1016/1260 CCVs failure.

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**Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.**

8.2.6 If the acceptance criteria are not met, analyze another CCV standard. If the second analysis of the standard fails to meet the criteria, recalibrate and/or perform other instrument maintenance.

8.2.7 If the CCV's fail acceptance criteria, data may be reportable based on the following criteria:

a. If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b. If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c. If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples must be reanalyzed.

d. If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample must be reanalyzed.

8.2.8 Load additional CCV's after every ten samples and at the end of the sequence. The standard concentration used for the CCV should be alternated over the course of the sequence. See Appendix_1 for calculation of %D.

**8.3 Prepare the Sample and Batch QC Extracts for analysis**

8.3.1 Remove the extracts from the extraction lab refrigerator and let the extracts warm to room temperature then aliquot approximately 1mL of extract into an autosampler vial.

8.3.2 If the extract is dark & oily or opaque, do another acid cleanup on the extract. If the extract is still highly colored, make a dilution that will result in an extract that is light yellow in color. If dark, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and failing CCV's.

8.3.3 Sulfur cleanup, using Copper powder, should be performed for all soil and water samples, along with the associated batch QC.

8.3.4 If dilutions are required, see Appendix_3 for instructions on preparing the dilutions.

8.3.5 Place the samples on the autosampler tray beginning with the lightest colored extracts followed by more highly colored or viscous extracts.

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## 9.0 QUALITATIVE ANALYSIS

Before any PCB can be quantitated, it must be qualitatively identified. Each Aroclor is a mixture of many individual polychlorinated biphenyls (for example: 3,3',4,4'-tetrachlorobiphenyl is a single component "congener" of an Aroclor mixture). Aroclor identification is based on pattern recognition, through comparison of the sample pattern to those of the Aroclors used as calibration standards. Trace overlays on EZChrom are very useful in determining which, if any, Aroclors are present because it can be used to overlay the sample chromatogram with the standards chromatograms.

*Note:* Although second-column confirmation is not required by EPA 8082, as the identity is based on pattern recognition and not on an individual peak, most of C&T's Department of Defense (Army Corp, Navy, AFCEE) clients do require it. C&T standard practice is to work up data from both columns and report the higher of the two results, unless specifically directed otherwise by a client's project plan.

### 9.1 Pattern Recognition

Aroclors are not discrete chemicals but are comprised of many related Polychlorinated Biphenyl (PCB) compounds. For environmental samples, these analytes are identified by comparison of the sample chromatograms to those of the standards. The composition and relative ratios of the target peaks in the sample chromatogram should *resemble* the standard but do not have to exactly match that of the standard, since the composition of these analytes is not completely defined, may vary between manufacturers and from batch to batch, and may have changed as the sample is "weathered" in the environment.

Except for Aroclor 1016 and Aroclor 1260, the components of different Aroclors often overlap, thus requiring separate calibration standards for these multi-component analytes. If the peaks do not overlap the standards may be combined, as in Ar1016/1260 and Ar1221/1254. For each of the Aroclors, five characteristic peaks are chosen as representative of each analyte; these peaks should be the largest peaks present that do not coelute with other target analytes. To positively identify the analyte in the sample, the chromatogram must contain at least 3 of the characteristic peaks (if present at low levels) with increasing resemblance to the standard as the concentration increases. Note, however, that the simple presence of 3 or more peaks within the expected retention window does not necessarily constitute a positive identification; positive identification is based on overall pattern recognition, including the presence or absence and ratios of non-quantitated peaks.

Non-target chlorinated compounds such as DDT and Endrin are typically removed prior to analysis by the sulfuric acid cleanup, however Technical Chlordane is not degraded by the acid and may appear in the latter ½ of the run; the two peaks with the highest response are typically a-Chlordane and g-Chlordane. Toxaphene may also be present and elute in the latter ½ of the run.

Use EZChrom's "trace overlay" feature to compare the sample pattern to each of the Aroclor patterns. If an Aroclor other than Ar1016 or Ar1260 is present and CCV's for that Aroclor do not bracket the sample extract, the extract must be reanalyzed with that Aroclor. See Appendix_7 for example chromatograms.

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**9.2 Interferences**

The ECD detector is a highly selective detector for halogenated compounds, which means that although the ECD can detect very low levels of chlorinated compounds in sample extracts, there may also be matrix effects upon the system that are not apparent in the sample chromatogram.

9.2.1 Most hydrocarbon and many pesticide interferences are removed during the extraction procedure, when the extracts are treated with sulfuric acid, however the sulfuric acid does not reduce interferences from technical chlordane. See Appendix 7 for an example chromatogram of chlordane.

9.2.2 If the extract is dark, oily & viscous, or opaque even after cleanup, repeat the cleanup or make a dilution that will result in an extract that is a very light yellow in color. If black, oily, or viscous extracts are analyzed, a tarry residue will build up in the injection port, causing active sites and CCV failure. See Appendix 3 for preparing various dilutions.

9.2.3 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting peaks. Because of the prevalence of this interferents, C&T normally performs a cleanup procedure on all extracts of soil or water samples, using copper powder (EPA 3550). Sulfur contamination is often found at relatively high levels in sediment samples. If after cleanup, the chromatogram still exhibits interfering levels of sulfur, the extract should be treated with additional copper. See Appendix 7 for an example chromatogram of sulfur interference.

9.2.4 If interferences are causing the retention times to shift slightly but the pattern clearly matches the Aroclor CCV, re-assign the peak RT to match the corresponding peak in the CCV. Note the reason for the adjustment on the user report.

9.2.5 Keep in mind that the ECD is responsive to all halogenated compounds to varying degrees, so samples containing bromine or fluorine (for example) may also be detected. It also responds to nitriles, nitro- compounds, organophosphorus compounds, carbamates, organometallic compounds or other electron-absorbing compounds.

**9.3 Retention Times for Quantitation**

Identification of the discrete components that are used for quantitation of the Aroclors is based on comparison of the peak retention times in the sample to the retention times of the peaks in the mid-level initial calibration standard or, for single-point calibrations, the retention times of the standard used for initial calibration. For the standards, each peak used for quantitation must fall within its retention time window and be automatically identified on both columns by the data system.

**9.4 Integration**

Determine whether manual integration is necessary by examining the sample chromatogram. For samples in which no matrix interferences are present, the sample

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peaks should be integrated from baseline to baseline, in the same fashion as the calibration standards.

For samples in which interferences raise the baseline, integration of the target compounds should be done on a valley-to-valley basis, unless a nearby negative peak would contribute a positive bias to the reported result; if a negative peak is present, use a baseline event to extend the baseline horizontally across the dip.

If there is an obvious inflection point, where an interfering peak is coeluting with the peak of interest, use the 'split peak' function to drop a vertical line from the inflection point to the baseline. In the LIMS Review App, document this event using the drop-down menu and indicate which column and peak or retention time was affected.

If manual integration is necessary there are a number of different baseline events to choose from. See Appendix 11 for a listing of EZChrom integration events.

***Warning:*** Unsubstantiated alteration of peak integrations solely to pass QC criteria (ie: calibration, surrogate) is ***illegal*** and is grounds for immediate termination of employment.

## 10.0 QUANTITATIVE ANALYSIS

Because the components of the various Aroclors overlap and multiple Aroclors may be present in the samples, the entire area of the standard can not be used for quantitation as is done for TPH-diesel (EPA 8015) samples. Instead, five characteristic peaks are identified in each Aroclor; these peaks are used both to assist in pattern recognition and in sample quantitation. A maximum of two of the five peaks may be discarded if it is evident that matrix interferences or "weathering" are distorting the quantitation of the peaks relative to the others.

Analyte quantitation is done using the external standard technique. Quantitation is based on comparison of the area of each target peak to the initial calibration curve for that peak, with adjustments for the sample preparation concentration factor and instrument dilution factor. The concentrations of the selected peaks within each target compound are then averaged to obtain the reported concentration. See Appendix 1 for example calculations. Concentrations are expressed as micrograms per liter or kilogram (µg/L, µg/kg).

All results are reported on a wet-weight ("as received") basis unless otherwise requested by the client. If the client requests 'dry-weight' corrections, the 'wet-weight' results in the results database are corrected for moisture by LIMS when producing the final report forms.

### 9.5 Evaluate the Sample Results

A user report for Ar1016/1260 will be automatically generated once the run is complete. Review any batch QC sample data first to verify that samples from that batch can be reported, then review the sample results to identify any samples that need to be rerun and/ or diluted.

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9.5.1 Verify that the target compounds are correctly integrated and bracketed by the applicable Aroclor standards.

9.5.2 If an Aroclor other than Ar1016 or Ar1260 is present and appropriate CCV's bracket the extract, process the data with the appropriate EZChrom method for that Aroclor; see Appendix 11 for EZChrom instructions.

9.5.3 If EZChrom identifies Aroclor peaks but the pattern does not resemble that standard, check the "FP" (false positive) box in the LIMS user page then click "Update LIMS" to save the change.

**9.5.4** If interferences are causing the quantitated result for one or two of the characteristic five peaks to be mis-quantitated, drop that/those peak(s) from the selected peaks so that the reported result is not artificially biased. Keep in mind that there must be a minimum of three peaks used for quantitation of each Aroclor.

9.5.5 If the concentration of any of the 3 to 5 Aroclor peaks selected for quantitation of the sample exceeds that of the highest concentration standard used in the ICAL curve for that compound (or of the highest Ar1016/Ar1260 calibration standard), LIMS will flag that over-range peak. Dilute the extract and reanalyze. See the "Dilutions" section below for further details.

9.5.6 If the Aroclor identified in the sample (see the 'Qualitative Analysis' section above) was different than the bracketing CCV standards, the extract must be reanalyzed with bracketing CCV's of the identified Aroclor. Quantitation is then calculated by averaging the concentrations of three to five characteristic peaks within the PCB pattern.

9.5.7 Neither EPA 608 nor EPA 8082 require second-column confirmation for PCBs, however EPA 8082A and nearly all DoD clients do require it. C&T's default process is to use second-column confirmation for all samples, unless specifically noted otherwise in the project comments. Using second-column confirmation, determine if a result should be reported by reviewing the data from both columns. To be reported, the analyte must be detected on both channels. A tentatively identified hit is considered a false-positive and reported as 'ND' if:

a. the PCB pattern is present on one column but not on the other,

b. the quantitation peak on the confirmation column falls outside the RT-window,

c. the result on the quantitation column is ≥ 2x the reporting limit but less than the reporting limit on the confirmation column, and the client is not requesting 'J-flagged' (estimated results <RL), or

d. the result on the quantitation column is < 2x the reporting limit and is less than ½ the reporting limit on the confirmation column, and the client is not requesting 'J-flagged' (estimated results <RL).

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If an analyte is detected on both columns with an RPD of ≤ 40% between the two results, report the higher of the two concentrations.

If the RPD > 40% between the two columns, evaluate the chromatograms for any co-eluting contaminants that may be causing the high RPD.

e. If coelution is evident on one chromatogram, report the result from the other column or clean up the extract; narrate the coelution, and the fact that the lower result was reported, on the "Data Review Checklist".

f. If no coelution is evident, report the higher of the two results.

g. LIMS will apply a 'C'-flag to the reported result.

9.5.8 Report the higher of the two columns' results, unless specifically directed to do otherwise by a client's project plan. *Note:* If reporting the lower result, note this and the project name on the Data Review Checklist.

*Note:* J-flagged hits on primary column will be confirmed by J-flag to MDL on confirm column.

9.5.9 If a sample is analyzed at multiple dilutions, compare the sample results across the various dilutions to verify that the dilutions were prepared correctly. Do the results make sense or is there a discrepancy between the runs? If there seems to be a discrepancy, reanalyze the sample to confirm the results.

**9.6 Surrogates**

9.6.1 After each sample is analyzed, LIMS will automatically generate a user report with the surrogate criteria for that sample and flag any failing recoveries. Evaluate the surrogate recoveries for all samples, method blanks, and spikes. If the extract was diluted by a factor of 10 or more, the surrogate is considered diluted out and LIMS will place a "DO" flag on the user report and final forms.

9.6.2 Evaluate the recoveries. If a surrogate recovery is outside QC limits, verify that the prep information (LIMS S#, amount, and concentration of surrogate added, sample weight/ volume, extract volume, and instrument dilution factors) is correct. If any of these are incorrect, fix the entry and reprocess the data. If the prep entry was correct, determine whether reanalysis is required using the following criteria:

a. If a high recovery is observed but no target analytes were detected above the reporting limit in the sample, note the failure on the 'Data Review Checklist' and report the data without reanalysis, since the possible high bias will not affect sample results.

b. If a high recovery is observed, and target analytes were detected, and there is no obvious chromatographic interference, the sample must be reanalyzed. If

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the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. If the same surrogate fails criteria after re-extraction it is deemed to be matrix effect. Check the "required for confirmation" box in the LIMS "Review App" for the second batch so it will be included if a Level 4 report is required (or requested at a later date) and note the situation in the case narrative.

c. If a low recovery is observed for any surrogate and there is no obvious chromatographic interference, or documented historical site matrix interference, the sample must be reanalyzed. If the same surrogate(s) fails criteria upon reanalysis a Corrective Action report must be initiated and the sample must be re-extracted. Check the "required for confirmation" box in the LIMS "Review App" for the second batch so it will be included if a Level 4 report is required (or requested at a later date) and note the situation in the case narrative.

9.6.3 If a sample must be re-extracted and the holding time has expired, the client's Project Manager must log the sample into LIMS as an alias and have the sample re-extracted as the new sample number. If the sample is still within holding time, the sample should be re-extracted under the original sample number.

9.6.4 If upon re-extraction, the surrogate recovery is again outside limits, note the matrix effect as "confirmed matrix interference" on the User Report and the Data Review Checklist, otherwise report the data with passing surrogate recovery.

Project-specific quality assurance project plans (QAPPs) may require batch control based on different compounds and control limits, in which case the project requirements supersede this SOP for all samples related to that project.

**EPA 608:** Method 608 does not discuss use of surrogate compounds, however C&T standard practice is to follow SW-846 guidance and use surrogates as a means of verifying the efficiency of the extraction and analysis.

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## 9.7 BATCH QC RESULTS

Project-specific quality assurance project plans (QAPP's) may contain different requirements than those listed in this SOP. If so, the QAPP requirements supersede this SOP for all samples related to that project.

**EPA 608**: Method 608 requires an LCS for every 10 samples, so any batch with more than 10 water samples must include a BS and BSD. Matrix spikes are also required on 10% of samples submitted for 608 analysis; if the client does not supply sufficient sample volume for the matrix spike, note this on the case narrative.

### 9.7.1 Method blank (MB):

Evaluate the results of the method blank. No compounds should be detected in the method blank, however if a compound(s) is detected, the following steps are used to determine the corrective action required:

a. If the concentration of the contaminant is below the reporting limit but above 1/2 of the reporting limit, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

b. If the target compound(s) found in the method blank was not detected in the associated samples, the data may be reported and the problem narrated.

c. If the target compound(s) found in the method blank was also detected in the associated samples, but the level in the samples is greater than 10x the level in the method blank, document the contamination on the batch sequence summary and the data review checklist and report the data without reanalysis.

d. If the target compounds detected in the method blank were also detected in the associated samples, but at levels less than 10x the level in the method blank, and reanalysis confirms the problem, the samples containing the contaminant must be re-extracted and reanalyzed. Initiate a Corrective Action Report (CAR) immediately so that re-extraction can begin within the extraction holding time, if necessary.

DoD QSM: The DoD requires evaluation and corrective action for method blank contamination down to ½ the RL, so for any project that references the DoD QSM, if the method blank is contaminated <RL but >½ RL, any samples containing <10xMB of that analyte must be re-extracted.

9.7.2 **Laboratory Control Sample (LCS) or Blank Spike/ Blank Spike Duplicate (BS/BSD):** After the QC samples for the batch have run, check the batch QC status to verify that the QC has passed acceptance criteria for all of the client-specified limits associated with the batch. In the 'general' view, the LIMS Review App will display the tightest limits required for jobs in that batch. To view this report:

9.7.2.1 Open the LIMS web browser and go to the Pesticide/PCB Instrument Page.

9.7.2.2 Next to the "View QC status for batch" box, type in the Batch number and click VIEW.

9.7.2.3 Review the recoveries, and RPD if applicable, to determine if the data can be reported. If either recovery or the RPD fail acceptance criteria, reanalyze the QC extracts. If the failure is confirmed upon reanalysis, initiate a Corrective Action Record, review the data in the 'Job' mode to determine if the results can be reported for any of the jobs, using the following guidance to determine the required corrective action:

a. If a high recovery is observed but no PCBs were detected in the associated samples, note the failure on the Data Review Checklist

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and report the data without re-extraction, as the potential high bias does not affect the sample results.

b. If a high recovery is observed and the samples contain PCBs at levels above the reporting limits, the samples must be re-extracted.

c. If a high RPD is observed but the recoveries are within acceptance limits and PCBs were not detected in the samples at levels above the reporting limits, note the failure on the Data Review Checklist and report the data without re-extraction, as the lack of good precision data does not affect ND samples.

d. If a high RPD is observed and PCB's were detected in the samples at levels above the reporting limits, all samples containing PCB's must be re-extracted.

e. If low recoveries are observed, the associated samples must be re-extracted.

9.7.2.4 If a sample must be re-extracted and the holding time has expired, the client's Project Manager should log the sample in as an alias and have the samples re-extracted as the new sample number. If the sample is still within holding time, re-extract and reanalyze the sample under the original sample number.

**9.7.3 Matrix Spike/ Matrix Spike Duplicate (MS/MSD):**
Evaluate the results of the matrix spikes. If any of the recoveries or RPD are flagged as outside limits, use the following to determine the appropriate action:

a. If the concentration of a target analyte is greater than 4x the spiking level, LIMS will apply a "NM" (for "Not Meaningful") flag to those recoveries. Report the data without reanalysis.

*Note:* If the concentration of a target analyte is greater than the spiking level, LIMS will flag and footnote that concentration for the client's attention.

b. If the concentration of a target analyte in the sample is greater than the linear range, but is not greater than 4x the spiking level, dilute the sample, MS, and MSD and reanalyze the extracts.

c. If the concentration of a target analyte in the sample is within linear range but the concentration in the matrix spikes is just greater than the linear range, dilute the sample, MS, and MSD and reanalyze the extracts.

d. If recoveries fail but the RPD is within acceptance limits, matrix interference is usually suspected. Narrate the failure and report the data without reanalysis

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(except for USACE, or other Level 3 or Level 4 projects that always require reanalysis).

e. If the recoveries fail due to obvious chromatographic interference (ie: coelution of other analytes with the spike compounds), narrate the failure on the Data Review Checklist and report the data without reanalysis.

f. If the recoveries are within limits but the RPD fails, and an isolated problem cannot be identified and documented, reanalyze the sample and matrix spikes.

9.7.4 After the batch QC samples have been reviewed and deemed acceptable, sign the LIMS "Review App" files and submit the forms to the Department Manager or a QC Chemist for review and approval.

## 11.0 DOCUMENTATION & PEER REVIEW

11.1 Review the job sheet to make sure that any special client needs are addressed (for example, CCI needs to be on one instrument). Make sure the correct clean-up version of samples and QC has run and are ready to report. Finally, search for any CARs associated with job.

11.2 Once you know what you need to report, use the C&T search tool and type in the Job#, Product ("pcb"), and Matrix. This will bring up sample data in LIMS. In the top right corner, use the "Review" pull down menu and select the job you would like to report. If you are unsure how to use the LIMS "Review App", see Appendix 12 for detailed instructions.

11.3 Log in if necessary.

***Important!*** You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

11.4 Review the LIMS generated user reports, the data reduction quantitation reports, and the chromatograms to ensure that the correct qualifier flags, dilution factors and results are reported. The user report must be initialed and dated by the analyst reviewing and approving the data for that sample.

11.5 Review the checklist and add any additional narrative needed, then sign the job and send it in for review.

11.6 Any changes made by the second-party reviewer must be individually initialed and dated by the reviewer. The second party reviewer must initial and date each user report, make any additional comments on the case narrative and sign the completed job.

## 12.0 POLLUTION PREVENTION

Direct the split vent and septum purge lines through a carbon trap in order to reduce solvent emissions into the laboratory. Prepare only sufficient standard volume to use within the shelf-life of the standard to reduce the volume of waste generated by the laboratory.

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## 13.0 WASTE DISPOSAL

All sample extracts should be stored in the Delfield refrigerator in the extraction lab. The extracts should be retained for a minimum of 40 days after extraction. After 40 days, the vials (autosampler, scintillation, and VOA) containing the extracts may be included in the 'PCB/Pesticides' waste stream; "satellite" waste accumulation in the PCB lab includes placement of the autosampler, scintillation, and VOA vials and their contents in a plastic bucket in the PCB instrument laboratory. When the plastic bucket is full, but no longer than 9 months after initial accumulation date, the vials should be transferred to the "90-day" PCB/Pesticide 55-gallon OH (open-head) waste drum, located in the waste room.

All containers holding chemical waste (liquid or solid) must be kept closed except when necessary to add or remove waste. Waste container must be compatible with the type of material being stored and must be in good condition (ie: not rusted or cracked).

Each container must be clearly labeled "Hazardous Waste", with a description of the composition ('PCB'), physical state of the waste ('Liquid/Solid'), the type of hazard (Flammable+Corrosive+Toxic), and the date that the first contents are added to the container.

## 14.0 REVISION HISTORY

The previous document (revision10) was updated as follows:

- Section 2: Added Varian ECD manual, QSMv5 to references
- Section 3: Added notes about extended holding times based on Arizona, 40CFR, and SW846 Update IV. Added bottle guidance.
- Section 5: Reformatted for ease of use. Added 'Definitions' section.
- Section 8: Added clarification that all soil and water extracts should be subject to a copper cleanup for sulfur. Added clarification regarding non-Ar1016/1260 quantitation.
- Section 9: Clarified QSM comment re: corrective action for blank contamination. Added additional guidance as to pattern recognition and responsiveness of the detector to non-chlorinated compounds.
- Section 10: Moved "Interferences" to Section 9 (Qualitative Identification). Updated to note that since EPA 8082A and the QSM require 2$^{nd}$ column confirmation, C&T defaults to this practice. Updated method blank criteria to allow samples >10x blank to be reported, matching current regulatory guidance
- Section 11: Minor revisions for paperless reviewing
- Section 13: Expanded details regarding waste
- Appendix_2: Added ISO Guide 34 requirement. Updated archiving of source standard Certificate of Analysis and working standard prep logs into LIMS. Clarified CCV prep and naming.
- Appendix_5: Added suggestion for running primer standards
- Appendix_7: Added Ar1262 & Ar1268 example chromatograms
- Appendix_9: Removed requirement to drop high point of all components if one is dropped from an ICAL point, as the linear ranges now all go through LIMS. Updated review process for 'paperless' system.

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- Appendix_10: Added clarification regarding non-Ar1016/1260 quantitation.
- Appendix_13: Updated comments to note that statistically generated limits are also used for EPA 608
- Appendix_14: Added method TO-10A (PUF) discussion

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## APPENDIX_1: CALCULATIONS

### SAMPLE CONCENTRATION

Quantitation is done by the data system using an external standard calculation method. The concentration of an analyte is calculated, by the data system, by comparing its peak area in the sample chromatogram against the initial calibration curve. This concentration is then adjusted for the dilution/concentration factor to obtain the final concentration of the analyte in the sample.

*Note:* If a client requests results reported on a 'Dry Weight' basis, the concentration is divided by the 'solids', where the solids is (100-%moisture)/100.

Dry Weight Concentration (µg/Kg) = "As Received" Conc. / ((100 - %moisture)/100)

**Concentration using Average Calibration Factor (by external standard)**

$$Cx = [ \sum (Ax / CFavg) / P ] * pdf * idf$$

Where:
Ax = Area of peak
CFavg = Average Calibration Factor for that peak, from the curve
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor
P = number of peaks used

**Concentration using Linear Regression (by external standard)**

$$Cx = [ \sum((Ax – b) / m ) / P ] * pdf * idf$$

Where:
Ax = Area of peak
b = intercept, for that peak from the curve
m = slope, for that peak from the curve
P = number of peaks used
pdf = Prep Dilution Factor (Vf/Vi or Vf/Wi)
idf = Instrument Dilution Factor

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### CALIBRATION EQUATIONS

**Calibration Factor** is the ratio of the detector response (area) to the amount (mass or concentration) in the calibration standard.

$$CF = Ax / Cx$$

Where:
Ax = Area of the compound
Cx = Concentration of the compound

Average CF or RRF = CFavg or RRFavg = $\sum(RFi) / n$

Where: RFi = Response or Calibration factor for each level

n = number of calibration points

**Relative Standard Deviation (%RSD)**
RSD is usually expressed in % and is a measure of the "goodness of fit" of a curve that uses the average response factor.

$$RSD = (SD / RFavg) * 100$$

Where: SD = Standard Deviation
RFavg = Average Calibration Factor or Relative Response Factor

Standard Deviation = $SD = \sqrt{\{[\sum (RFi - RFavg)^2] / (n-1)\}}$

Where: RFi = Response or Calibration factor for each level
RFavg = Average Calibration Factor or Relative Response Factor
n = number of calibration points

**Linear Regression Equations**

$$y = mx + b$$

Where: y = response (area of peak
x = concentration
m = slope
b = intercept

Slope (m) = $[ (\sum wx_iy_i * \sum w) - (\sum wx_i * \sum wy_i) ] / [ (\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i) ]$

Intercept (b) = $y_{avg} - (m * x_{avg})$

Correlation Coefficient (r)

$$r = \frac{[ (\sum w * \sum wx_iy_i) - (\sum wx_i * \sum wy_i) ]}{\sqrt{\{[ (\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [ (\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i) ]\}}}$$

Coefficient of Determination ($r^2$) = r * r

Where: $x_i$ = individual values for the independent variable (concentration)
$y_i$ = individual values for the dependent variable (response, area)
w = weighting factor (for no weighting w = 1)
$x_{avg}$ = average of the x-values
$y_{avg}$ = average of the x-values

**Percent Difference (or Percent Drift), %D:**
For calibration verification standards, the %D is the difference between the true concentration of the standard and the calculated concentration of the standard, divided by the true concentration, multiplied by 100:

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%D (Percent Difference) = ((Cws - Cf) / Cws) * 100

Where: Cws = true concentration of the spiking standard
Cf = final measured concentration in the spiked sample

## BATCH QC

**Percent Recovery (%R):**
The recovery is the measured concentration divided by the true concentration of the spike.

%Recovery = (Cf – Cs) / (Cws * Vws) *100

Where: Cf = final measured concentration in the spiked sample
Cs = measured concentration in the un-spiked aliquot of sample
Cws = concentration of the spiking standard
Vws = volume used, of the spiking standard

**Relative Percent Difference (RPD):**
The RPD is the absolute value of the difference in concentrations divided by the average of the concentrations.

%RPD = |(Cs - Cdup )| / ((Cs + Cdup)/2) * 100

Where: Cs = measured sample concentration
Cdup = measured concentration in the duplicate

**Make a Working Standard from a Source (Stock) Standard:**
Determine the volume of source standard needed to make a given volume of working standard:

Vss (mL) = Vws * Cws / Css

Where: Vss = Volume of Source Standard (mL) needed to make Working Standard
Vws = Final Volume (mL) of Working Standard
Cws = Final Concentration (µg/mL) of the Working Standard
Css = Concentration (µg/mL) of the Source Standard

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**APPENDIX_2: SOURCE & WORKING STANDARDS**

## A. STANDARDS DOCUMENTATION

The standards listed below were those in use at the time this document was written, however standards may be purchased from different vendors so long as they are traceable through LIMS and the Standards Prep Logs.

**Source Standards**

Source standards are those purchased directly from a chemical manufacturer or vendor. The LIMS S-name is unique to the manufacturer of the source standard; if a source standard is obtained from a different manufacturer, a new S-name must be assigned and the information entered in the "Standard Definitions" table before the standard can be assigned an S#.

For calibration standards, at least one of the manufacturers (either primary or secondary) must be ISO Guide 34 accredited. This is an A2LA requirement associated with C&T's DoD accreditation.

Source standards usually have an expiration date set by the manufacturer. If no expiration date is listed, the expiration date is one year from date received. Certificates of Analysis should be obtained from the vendor of each source standard; if no certificate was provided by the vendor, download a copy from their website or call and request that one be emailed.

Enter all source standards into LIMS immediately upon receipt, using the Standards Menu "Standard Inventory", listing the date received, lot number, and expiration date.

Write the LIMS S# and expiration date on the vials and the certificate of analysis. Scan the certificate of analysis into LIMS, then verify that the scanned copy was legible.

**Working Standards**

Working standards are those prepared by C&T. The LIMS S-name is unique to the source standard(s) used to create the working standard, the compound list, and the concentrations contained in the working standard; if any of these are changed, a new S-name, compound list and concentrations must be entered in the "Standard Definitions" table before the standard can be logged in and assigned an S#. It is *very important* to enter this information correctly, as LIMS uses this information to calculate spike and surrogate recoveries.

Working standards expire 180 days after preparation from the source standards unless any of the source standards expire before the 180 days. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard *must not* exceed the expiration date of any of the source standards from which it was made**.

The initial calibration verification (ICV) standard is prepared using a source standard obtained from a different manufacturer from the initial calibration curve standards. Continuing

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calibration verification (CCV) standards are prepared from either the same manufacturer as the initial calibration curve or from a different manufacturer.

In the Standards Benchbook, enter the prep date, LIMS S#, concentration, and volume of each source standard used, solvent name and lot#, the LIMS S-name and concentration of the working standard, expiration date, and prep chemist's initials.

In LIMS, use the "Standards Inventory" screen to enter the prep chemist's initials, prep date, and S# of the source or intermediate standards used to make the working standard; LIMS will then assign a standard number (S#).

After the working standards have been prepared and the benchbook entry completed, scan the benchbook page(s) into LIMS then verify that the scanned copy is legible.

## B. SOURCE STANDARDS

Label each vial with the contents, LIMS S#, and expiration date. Store standards at 4°C (± 2°C) in Refrigerator #21 in the pesticides lab; standards may not be stored in a refrigerator containing samples or extracts.

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**Primary Source Standards**

| Analytes | Concentration (µg/mL) | Supplier & Catalog# | LIMS S-Name |
|---|---|---|---|
| Surrogates (TXCM/DCB) | 200 | Ultra Scientific ISM-320 | PSS |
| Aroclor 1016/1260 Mix | 1,000 | Restek 32039 | AR_16/60 |
| Aroclor 1221 | 1,000 | Restek 32007 | AR21_R |
| Aroclor 1232 | 1,000 | Restek 32008 | AR32_R |
| Aroclor 1242 | 1,000 | Restek 32009 | AR42_R |
| Aroclor 1248 | 1,000 | Restek 32010 | AR48_R |
| Aroclor 1254 | 1,000 | Restek 32011 | AR54_R |
| Aroclor 1262 | 1,000 | Restek 32409 | AR62_R |
| Aroclor 1268 | 1,000 | Restek 32410 | Ar68_R |

**Second Source (ICV) Standards**

| Analytes | Concentration, in Hexane (µg/mL) | Supplier & Catalog# | LIMS S-Name |
|---|---|---|---|
| Aroclor 1016/1260 Mix | 100 | Ultra Scientific PPM-8082-1 | 1660_U |
| Aroclor 1221 | 100 | Ultra Scientific PP-292-1 | 1221_U |
| Aroclor 1232 | 100 | Ultra Scientific PP-302-1 | 1232_U |
| Aroclor 1242 | 100 | Ultra Scientific PP-312-1 | 1242_U |
| Aroclor 1248 | 100 | Ultra Scientific PP-342-1 | 1248_U |
| Aroclor 1254 | 100 | Ultra Scientific PP-352-1 | 1254_U |
| Aroclor 1262 | 100 | Ultra Scientific PP-372-1 | 1262_U |
| Aroclor 1268 | 100 | Ultra Scientific PP-382-1 | 1268_U |

## C. WORKING STANDARDS PREPARATION

Because the solvent may dissipate over time, these solutions must be discarded after six months or sooner if there is a discrepancy when compared with other standards. Verify that the LIMS expiration date of the working standard does not exceed that of any of the source or intermediate standards used to make it. If any of the source standards expire before the 180 days, change the expiration date of the working standard to match the earliest expiration date of the stock standards. **The expiration date of the working standard must not exceed the expiration date of any of the source standards from which it was made**.

Prepare the working standards in hexane, by diluting source standards to volume in a Class-A volumetric flask.

Label the standards vials with the name & concentration (or calibration level) of the standard, LIMS S# and the expiration date.

Complete the standards prep benchbook entry then scan the pages into LIMS. Verify that the scanned copy is legible.

Store standards at 4°C (± 2°C) in the extraction lab refrigerator; do not store in a refrigerator containing samples or extracts.

Make up a 10,000 µg/L intermediate standard then dilute this standard in hexane to make the calibration standards.

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| Level | Add Vol. (mL) Std | Using Std Name | Using Std Conc (µg/L) | Final Vol. (mL) in Hexane | Final Conc (µg/L) of Aroclors / Surrogates | LIMS S-Name |
|---|---|---|---|---|---|---|
| ICAL Intermediate | 1.00<br>1.00 | AR_16/60<br>PSS | 1,000,000<br>200,000 | 100 | 10,000 / 2,000 | RES16/60_S |
| | | | | | | |
| ICAL 1 | 0.100 | RES16/60_S | 10,000 / 2,000 | 100 | 10 / 2 | PCB10_2 |
| ICAL 2 | 0.250 | RES16/60_S | 10,000 / 2,000 | 100 | 25 / 5 | PCB25_5 |
| ICAL 3 | 1.00 | RES16/60_S | 10,000 / 2,000 | 100 | 100 / 20 | PCB100_20 |
| ICAL 4 | 2.50 | RES16/60_S | 10,000 / 2,000 | 100 | 250 / 50 | PCB250_50 |
| ICAL 5 | 5.00 | RES16/60_S | 10,000 / 2,000 | 100 | 500 / 100 | PCB500_100 |
| ICAL 6 | 7.50 | RES16/60_S | 10,000 / 2,000 | 100 | 750 / 150 | PCB750_150 |
| ICAL 7 | 10.0 | RES16/60_S | 10,000 / 2,000 | 100 | 1,000 / 200 | PCB1K_200 |
| | | | | | | |
| ICV Intermediate | 1.00<br>1.00 | 1660_U<br>PSS | 1,000,000 /<br>200,000 | 100 | 10,000 / 2,000 | PCBULTRA_S |
| | | | | | | |
| ICV | 2.50 | PCBULTRA_S | 10,000 / 2,000 | 100 | 250 / 50 | ULTRA_1660 |

Initial calibration curves for the remaining analytes are diluted in the same pattern as shown above and named in reference to the Aroclor (ie: Ar1232 would be 32_750, 32_500, etc). Aroclor

1232, Aroclor 1242, Aroclor 1248, Aroclor 1262, and Aroclor 1268 are analyzed as discrete standards. Aroclor 1221 and 1254 source standards may also be combined into a single standard (2154_750, 2154_500, etc).

*EPA 608 Method Modification:* EPA 608 calls for the "stock standards" to be prepared in isooctane; C&T uses hexane because sample extracts end up in hexane and use of the same solvent improves matrix-matching between standards and samples.

*Note:* The USACE recommends that CCV's be either the same standards as the initial calibration standards or at least be prepared from standards obtained from the primary vendor, to more easily differentiate between instrument problems and variability of the standards.

**Continuing Calibration Verification Standards**
CCVs are made from the 10,000 µg/L intermediate calibration standards, with final concentrations in the middle of the calibration range (100 µg/L, 250 µg/L, 500 µg/L) and following the same naming conventions as the ICAL standards. The Ar1016/1260 CCV standards are named PCB##_$$, where ## is the concentration of the Aroclor and $$ is the concentration of the surrogate. For other Aroclors, the CCVs are named as <PCB>_##, where <PCB> is the Aroclor number(s) and ## is the concentration of the Aroclor.

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**APPENDIX_3: PESTICIDE & PCB DILUTIONS**

Let the extracts warm to room temperature then prepare the dilution in either an autosampler vial or an insert. See table below for appropriate volumes. Shake the dilution and invert 3 times to mix.

| Dilution Factor | Made In | Extract Volume (µL) | Hexane Volume (µL) |
|---|---|---|---|
| 2x | Insert | 100 | 100 |
| | GC vial | 500 | 500 |
| 3x | Insert | 50 | 100 |
| | GC vial | 250 | 500 |
| 4x | Insert | 50 | 150 |
| | GC vial | 250 | 750 |
| 5x | Insert | 40 | 160 |
| | GC vial | 200 | 800 |
| 10x | Insert | 20 | 180 |
| | GC vial | 100 | 900 |
| 20x | Insert | 10 | 190 |
| | GC vial | 50 | 950 |
| 50x | GC vial | 20 | 980 |
| 100x | GC vial | 10 | 990 |

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**SERIAL DILUTIONS**

If you need to make a >100x dilution, first make the 100x dilution listed above, then make further dilutions, in hexane, using that as an intermediate.

| Dilution Factor | Using Primary Dil'n | Made In | Extract Volume (µL) | Hexane Volume (µL) |
|---|---|---|---|---|
| 200 | 100x | GC vial | 100 | 100 |
| 500 | 100x | GC vial | 40 | 160 |
| 1,000 | 100x | GC vial | 20 | 180 |

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## APPENDIX_4: INSTRUMENT CONDITIONS

The settings listed below were those in use at the time this document was written. Parameters may be changed at the analyst's discretion in order to optimize instrument performance. Changes are documented in the instrument's maintenance log.

**GC-06 / GC-16** Hewlett Packard 5890

| | | |
|---|---|---|
| Columns: | Front (Channel A): | Restek RTx-CLPesticides |
| | Back (Channel B): | Restek RTx-5 |
| Column Insertion: | Injector side: | 5 mm from the end of the ferrule |
| | Detector side: | 70 mm from the bottom of the nut |
| Gases: | Carrier: | Hydrogen |
| | Makeup: | 5% Methane in Argon (P5) at 50 mL/min |

**Oven**

| | |
|---|---|
| Oven On: | Yes |
| Equilibration Time(min): | 0.2 |
| Maximum Temperature (°C) | 325 |

| Ramp | Rate (mL/min) | Final Temp (°C) | Hold Time (min) |
|---|---|---|---|
| Initial | - - | 140 | 0 |
| 1 | 7 | 270 | 0 |
| 2 | 25 | 325 | 3 |

**Zone Temperature (°C)**

| | |
|---|---|
| Inj A: | 205 |
| Inj B: | 205 |
| Det A: | 300 |
| Det B: | 300 |
| Constant Flow (mL/min): | 2.5 |

**Front Injector**

| | |
|---|---|
| Sample Washes | 3 |
| Sample Pumps | 2 |
| Viscosity Delay | 1 |
| Solvent A washes | 3 |
| Solvent B washes | 3 |

**Valves**

| | |
|---|---|
| Valve 3: | On |
| Valve 4: | On |

**Inlet Purge Valves**

| Init State | On Time (min) | Off Time (min) |
|---|---|---|
| A | 0.2 | 0 |
| B | 0 | 0 |

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## GC-22 Varian CP-3800 w/ CP-8400 Autosampler

| | | |
|---|---|---|
| Columns: | Front (Channel A): | Restek RTx-CLPesticides |
| | Back (Channel B): | Restek RTx-5 |
| Column Insertion: | Injector side: | 3.7 cm from the bottom of the nut |
| | Detector side: | 10.5 cm from the bottom of the nut |
| Gases: | Carrier: | Helium |
| | Makeup: | Nitrogen |

### Front Injector

Temperature (°C): 210

Split:

| Time (min) | State | Ratio |
|---|---|---|
| Initial | Off | 0 |
| 0.5 | On | 100 |
| 2.0 | On | 50 |
| 2.5 | On | 5 |

| | |
|---|---|
| Constant Flow Rate (mL/min): | 2.2 |
| Pressure Pulse Enable: | On |
| Pressure Pulse Pressure (psi): | 30 |
| Pressure Pulse Duration (min): | 0.25 |

### Middle Injector

Temperature (°C): 210

Split:

| Time (min) | State | Ratio |
|---|---|---|
| Initial | Off | 0 |
| 1.2 | On | 100 |
| 2.7 | On | 50 |
| 25 | On | 5 |

| | |
|---|---|
| Constant Flow Rate (mL/min): | 2.2 |
| Pressure Pulse Enable: | On |
| Pressure Pulse Pressure (psi): | 30 |
| Pressure Pulse Duration (min): | 0.95 |

### CP-8400 Autosampler

| | |
|---|---|
| Injector Position: | 1 & 2 |
| Use Injector Delay (min): | 0.7 |
| Sample Penetration Depth %: | 90 |
| Solvent Penetration Depth %: | 90 |
| Number of Fill Strokes: | 3 |
| Fill Volume for Fill Strokes (µL): | 5 |
| Sample Flushes: | 0 |
| Pre-Inject Flushes: | 3 |
| Post-Inject Flushes: | 1 |
| Clean Solvent Source Vial: | 1 |

### Oven

Stabilization Time (min): 0.2

| Step | Temp(°C) | Rate (mL/min) | Hold (min) |
|---|---|---|---|
| Initial | 140 | - - | 0 |
| 1 | 280 | 7.5 | 0 |
| 2 | 310 | 25 | 4 |

### Detector Settings:

| | | |
|---|---|---|
| Front: | Temperature (°C): | 325 |
| | Range: | 1 |
| | Range Auto-zero: | No |
| | Electronics: | On |
| | Time Constant: | Fast |
| | Cell Current: | N2 High |
| | Contact Potential: | 300 |
| Back: | Temperature (°C): | 325 |
| | Range: | 1 |
| | Range Auto-zero: | No |
| | Electronics: | On |
| | Time Constant: | Fast |
| | Cell Current: | N2 High |
| | Contact Potential: | 300 |

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## GC-25 Varian CP-3800 w/ CTC Analytics Combipal Autosampler

| | | |
|---|---|---|
| Columns: | Front (Channel A): | Restek RTx-CLPesticides |
| | Back (Channel B): | Restek RTx-5 |
| Column Insertion: | Injector side: | 3.7 cm from the bottom of the nut |
| | Detector side: | 10.5 cm from the bottom of the nut |
| Gases: | Carrier: | Helium |
| | Makeup: | Nitrogen |

### Front Injector

Temperature (°C): 200

| Split: Time (min) | State | Ratio |
|---|---|---|
| Initial | On | 10 |
| 0 | Off | 0 |
| 0.3 | On | 50 |
| 2.5 | On | 10 |
| 25 | On | 2 |

| | |
|---|---|
| Constant Flow Rate (mL/min): | 2.5 |
| Pressure Pulse Enable: | On |
| Pressure Pulse Pressure (psi): | 45 |
| Pressure Pulse Duration (min): | 0.23 |

### Middle Injector

Temperature (°C): 210

| Split: Time (min) | State | Ratio |
|---|---|---|
| Initial | On | 10 |
| 0.94 | Off | 0 |
| 1.23 | On | 50 |
| 3.43 | On | 10 |
| 25 | On | 2 |

| | |
|---|---|
| Constant Flow Rate (mL/min): | 2.2 |
| Pressure Pulse Enable: | On |
| Pressure Pulse Pressure (psi): | 30 |
| Pressure Pulse Duration (min): | 0.95 |

### Oven

Stabilization Time (min): 0.2

| Step | Temp (°C) | Rate (mL/Min) | Hold (min) |
|---|---|---|---|
| Initial | 110 | - - | 0 |
| 7 | 280 | 1.72 | 26.01 |

### Detector Settings:

| | | |
|---|---|---|
| Front: | Temperature (°C): | 325 |
| | Range: | 10 |
| | Range Auto-zero: | No |
| | Electronics: | On |
| | Time Constant: | Fast |
| | Cell Current: | N2 High |
| | Contact Potential: | -400 |
| Back: | Temperature (°C): | 325 |
| | Range: | 1 |
| | Range Auto-zero: | No |
| | Electronics: | On |
| | Time Constant: | Fast |
| | Cell Current: | N2 High |
| | Contact Potential: | 330 |

### CombiPal Autosampler Settings

Cycle: GC-DUAL
Syringe: 10 µL

| | | | | | |
|---|---|---|---|---|---|
| 1.Sample Vol: | 1.0 µL | Pre Cln Slv1: | 1 | Fill Volume: | 5.0 µL |
| 1.Air Vol: | 0 nL | Pre Cln Slv2: | 0 | Fill Speed: | 2 µL/s |
| 1.Inject to: | FRONT | Pre Cln Spl: | 1 | Fill Strokes: | 3 |
| 1. Inj Time Diff: | 0s | Int Cln Slv1: | 0 | Pull up Del: | 1s |
| 2.Sample Offs: | 0 | Int Cln Slv2: | 0 | Inject Speed: | 100 µL/s |
| 2.Sample Vol: | 1.0 µL | Pst Cln Slv1: | 0 | Prep Inj Del: | 200 ms |
| 2. Air Vol: | 0 nL | Pst Cln Slv2: | 3 | Pst Inj Del: | 200 ms |
| 2. Inject to: | MIDDLE | | | | |

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**APPENDIX_5: MAINTENANCE & TROUBLE-SHOOTING**

Any maintenance performed on the instrument must be documented in the Maintenance logbook. This notation must include the reason the maintenance was necessary ('CCV failing', etc.), the date, analyst initials, maintenance steps performed, and resolution ('ICAL passed', etc.) of the maintenance.

**"SAFETY SHUT-DOWN":**
A "safety shut-down" will occur when the EPC (electronic pressure control) can not reach the pressure set-points due to problems with the gas supply. The problem may be due to a cored septum, a faulty valve, someone disconnecting a tank incorrectly, a loose injection port screw, a broken or plugged column, or an empty tank. Investigate and correct the problem before restarting the sequence.

The GC will begin beeping up to 2 minutes before the "safety shut-down" happens. If the problem is immediately corrected, the shut-down may be averted. If this happens, watch the sequence to ensure that it does continue correctly.

To restart GC-06 after a safety shut-down, turn off the GC, wait about 15 seconds, then turn the GC back on.

**TROUBLE-SHOOTING**

Symptom: **Dirty instrument blank**

Possible causes:

- Semivolatile or nonvolatile sample residues in injector. Remove injector liner and stainless steel seal and swab out injection port with a Q-Tip soaked in Hexane. Change septum, injector liner and gold seal. Reanalyze instrument blank.

- Semivolatile or nonvolatile sample residues in column. Remove guard column from injector and cut off about 1 loop. Reinstall. Reanalyze instrument blank. If instrument blank is still dirty, rinse column and reanalyze instrument blank. See RESTEK literature for column rinsing instructions.

Symptom: **Low instrument response**

Possible causes:

- Oxygen or water in the gas lines may oxidize the radioactive Nickel source and reduce the detector response.
- Low P5 make-up gas flow. The P5 is needed to provide free electrons that are not contained in the Helium carrier gas.

Symptom: **Peak splitting** (usually TCMX)

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Possible causes:

· Semivolatile or nonvolatile sample residues in injector. Remove injector liner and stainless steel seal and swab out injection port with a Q-Tip soaked in Hexane. Change septum, injector liner and gold seal.

**PREVENTATIVE MAINTENANCE**

1. Septa should be replaced after no more than 100 injections.

2. Injector liners should be replaced whenever a CCV is out of limits or you cannot get a clean instrument blank.

3. Check the tightness of the capillary column detector adapter nut weekly. Replace the ferrule in the adapter when you can no longer tighten the fitting and the adapter is still loose.

4. Check tightness of capillary column ferrule nuts in detector and inlet fittings; they should be snug - do not over-tighten.

5. Autosampler syringes should be removed from the autosampler and cleaned weekly. Clean by drawing and expelling a 1:1 vol:vol Hexane/Acetone mixture. After 3 rinses, withdraw the plunger and wipe it with a Kimwipe. Repeat; continue until no more residue is being wiped from the plunger.

6. A “primer”, usually consisting of one of the 10,000 µg/L intermediate standards, may be needed after the column is changed or when low CCVs are observed. This primer may be any of the Aroclor standards and expired standards may be used.

7. Bake out detectors (thermal cleaning) after running many “dirty” samples. They should be baked out about every three months or whenever the response is not linear or it is exhibiting high background. Warning – baking out a detector will change its response; the instrument will need to be recalibrated after this maintenance!

   To thermally clean an EC detector:

   6.1 Remove the end of each column from it’s detector and cap the detector.

   6.2 Set DET A TEMP and DET B TEMP to 350 °C for HP GC’s, or 375°C for Varian GC’s.

   6.3 Monitor the progress of the cleaning by observing the detector signals (SIG 1 or SIG 2). Typically the signal will rise as the detector temperature goes to 350 °C. If the detector is dirty the signal will peak and decrease signal may just rise until the detector temperature equilibrates and then stay constant. Thermal cleaning is complete when the detector signal has been constant for about 2 hours.

   6.4 Reset DET A TEMP and DET B TEMP to 325 °C

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## APPENDIX_6: RETENTION TIMES & ELUTION ORDER

**A. Retention Time Windows:** "Retention Time Windows" are necessary because compounds may not elute at *exactly* the same time during each and every injection, due to slight variations in temperature, flow rate, or injection composition (sample viscosity, compound concentrations), etc. The RT-window is the length of time (width, in minutes, on the chromatogram) during which any peak eluting within the window is presumed to be the analyte of interest. "72-hour RTW Study" is a term often used by auditors to describe statistical analysis of the retention times of standards injected over a 72 hour sequence; theoretically, the RT windows determined by this study can be used for routine analysis, however the studies that C&T has conducted in the past result in windows that are too narrow for routine use. C&T therefore uses the default retention time windows of ± 0.03 minutes as specified in EPA 8000B.

If a 72-hour RT study is required by a client or auditor, the RT windows are defined as plus or minus three times the standard deviation of the absolute retention times for each compound in the calibration standard mix as measured over the course of 72 hours. (*Note:* This procedure has historically (and consistently) produced RT-windows too tight for routine use.) In the event that a standard deviation is 0.00, then use the 0.03-minute window (see 8000B). However the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

For multi-component standards, i.e. PCB's, the analyst should use the retention time windows but also rely on pattern recognition.

**B. Absolute Retention Times:** The "absolute" retention time of any compound is the expected time of the compound is the center of the RT window. Use the retention time for each analyte from the mid-level initial calibration standard as the "absolute" retention time. Examine the daily CCV retention times and verify that the peak maximum falls at the center of each retention window. If any of the compounds fall outside their daily or approach the limit of the window, update the retention times based on the first CCV of that sequence. Save the method file with the new date and process the entire sequence with the new RT's.

*Method Modification Note:* EPA 8000B, Section 7.6.5 suggests updating the absolute retention times each time a new sequence is started. Because the retention times for these compounds are relatively stable, C&T has only found it necessary to update the retention times when performing the initial calibration or when an RT-shift is observed in the CCV.s.

**C. Elution Order:** The order in which compounds elute is based on chemical composition of the stationary phase of the column and on the instrument conditions (flow rates, temperature programming, column length). Given a specific set of instrument conditions (flow rates, temperature program) the order in which compounds elute from a column should remain constant but may differ between different types of columns.

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## APPENDIX_7: INTERFERENCES & MULTICOMPONENT CHROMATOGRAMS

Matrix interferences may cause inaccuracies in the determined element concentrations. These interferences may interfere with the integration of the data or with the actual instrumental analysis of the samples.

**A. Sulfur**

The presence of elemental sulfur is generally indicated by the presence of several large peaks that may obscure the target analytes or otherwise interfere with the detection and integration of the target analytes; see the following pages for example chromatograms. This is a common interference for most soil and wastewater samples but is not as prevalent in groundwater samples. If sulfur is present, it should be removed using copper powder and the extracts should then be reanalyzed.

**B. Multicomponent Pesticides & PCB's**

PCB's, technical chlordane, and toxaphene are all multi-component (ie: consist of more than a single compound) analytes that will be observable on the sample chromatograms and may interfere with quantitation of discreet PCB congeners or pesticides; see the following pages for example chromatograms. If these multi-components are present, it may not be possible to reliably report a single, undiluted run; see the Department Manager or QC Chemist for guidance.

**C. Organic Acids**

Organic acids do not appear on the sample chromatograms but are indicated by the disappearance of DDT, Methoxychlor, and their breakdown products from pesticide CCV's. These interferences are common to wastewater treatment facilities and municipal landfills. Any sample extracts suspected, or known through site history, to contain organic acids should be run at a 100x or more dilution. If these extracts are analyzed at a less dilute level, the acids will destroy the column's stationary phase and the entire column will then need to be replaced.

**D. Hydrocarbons**

Hydrocarbons present in dark or oily extracts will cause active sites in the injection port, leading to breakdown problems. See the "Dilutions" section in the main body of this SOP for further discussion.

**E. Other interferences**

Keep in mind that the ECD is responsive to all halogenated compounds to varying degrees, so samples containing bromine or fluorine (for example) may also be detected. It also responds to nitriles, nitro- compounds, organophosphorus compounds, carbamates, organometallic compounds or other electron-absorbing compounds.

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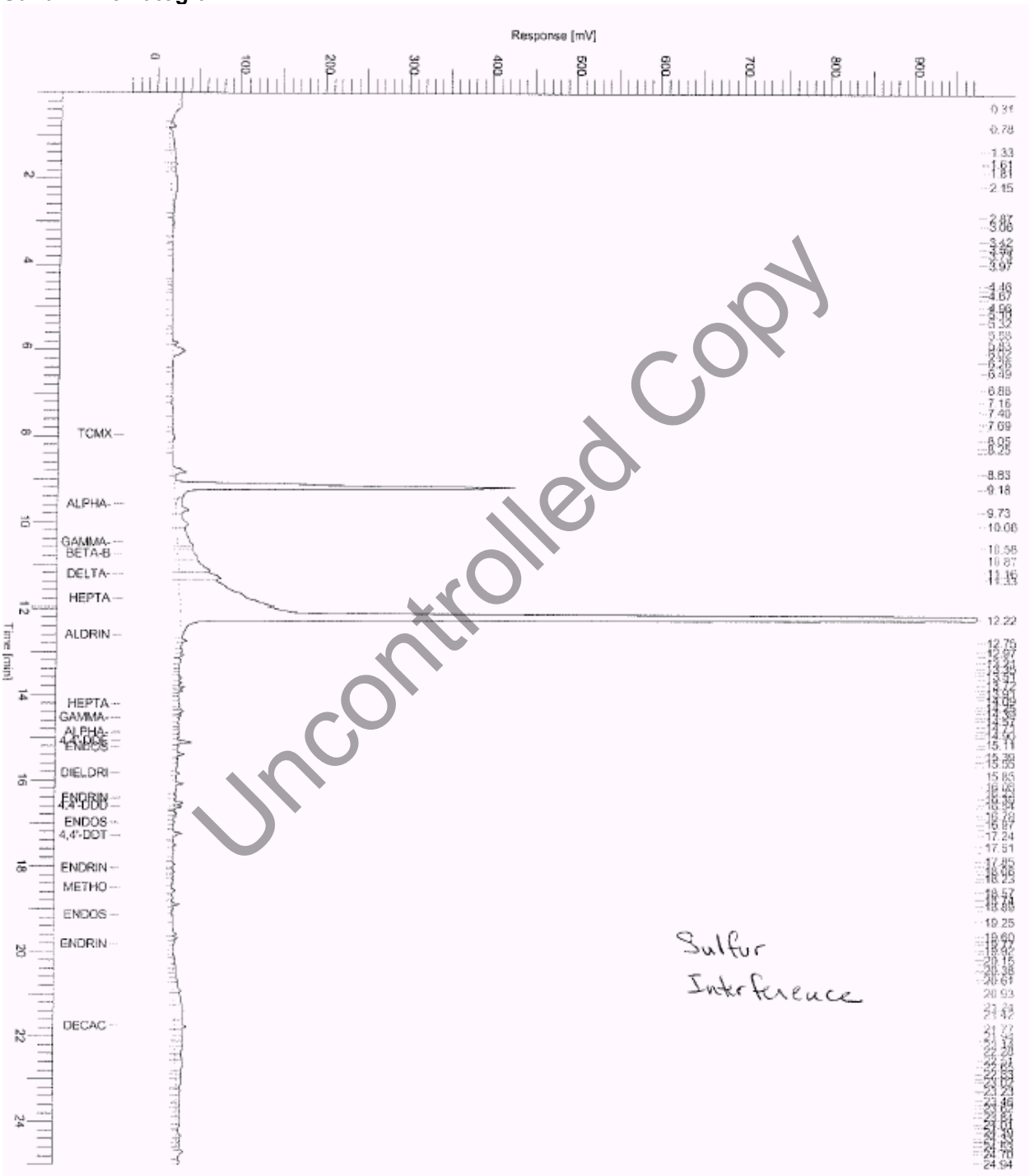
**Sulfur Chromatogram**

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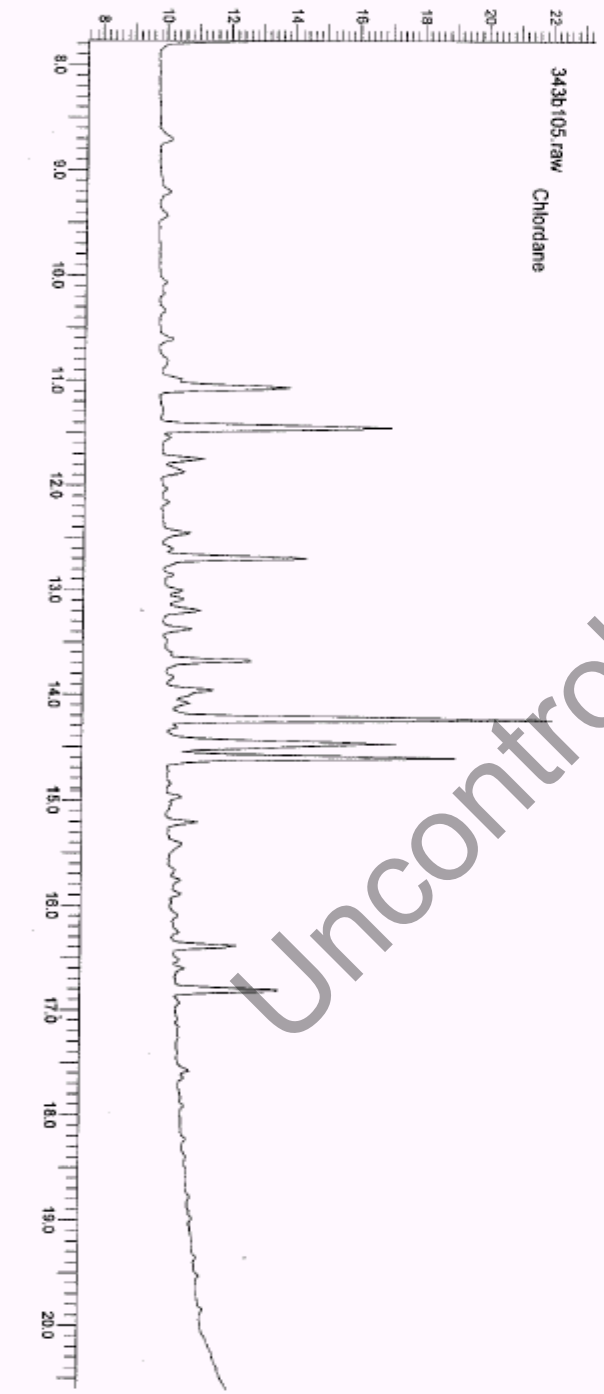
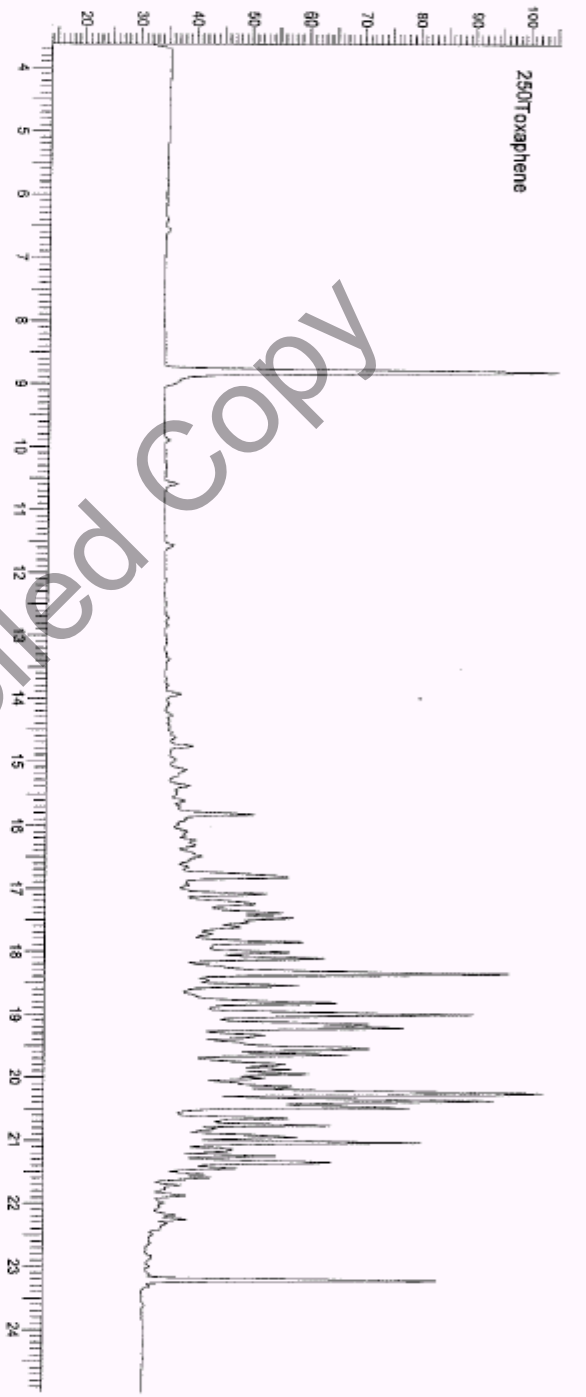
**Technical Chlordane Chromatogram**




**Toxaphene Chromatogram**

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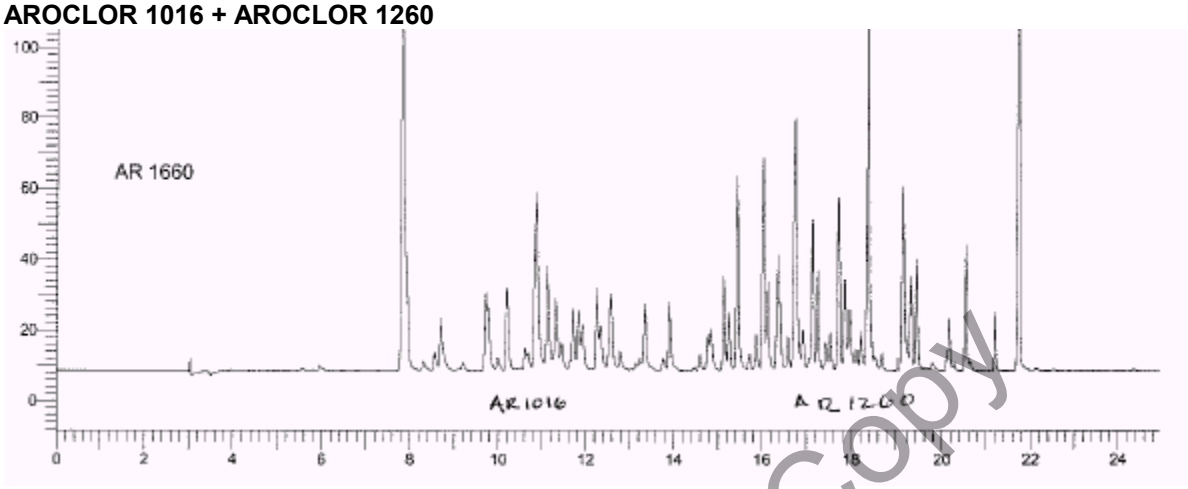
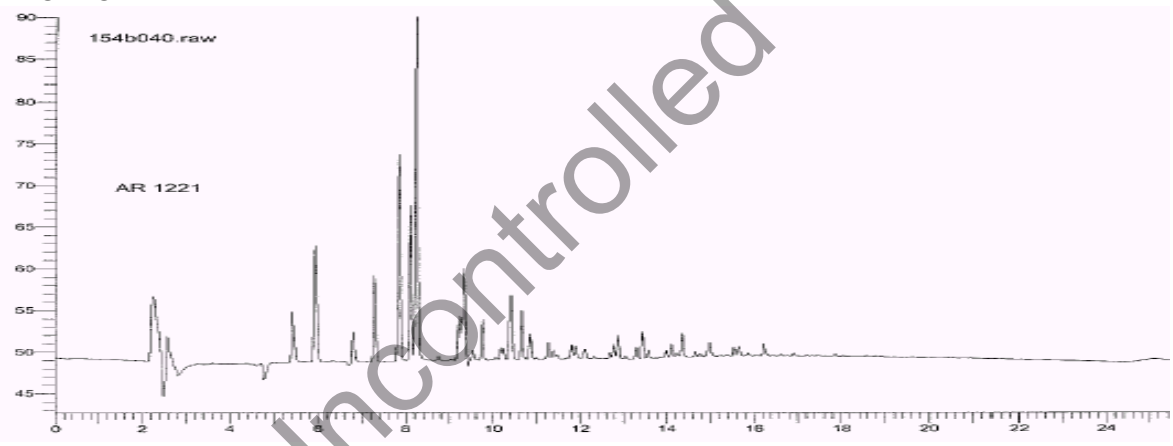
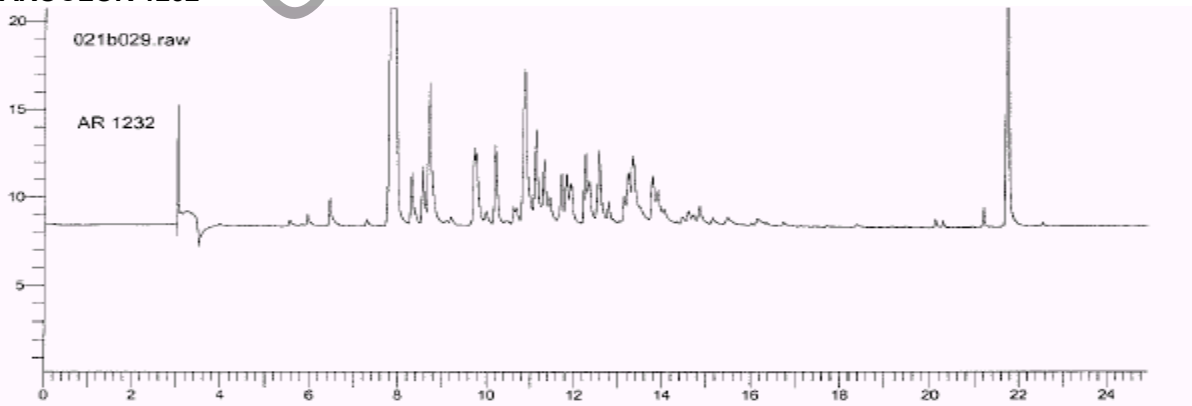
**AROCLOR 1016 + AROCLOR 1260**




**AROCLOR 1221**




**AROCLOR 1232**

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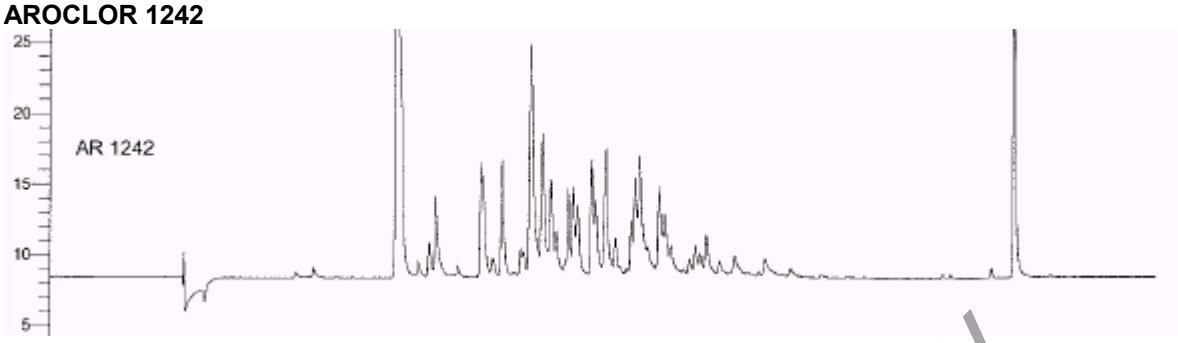
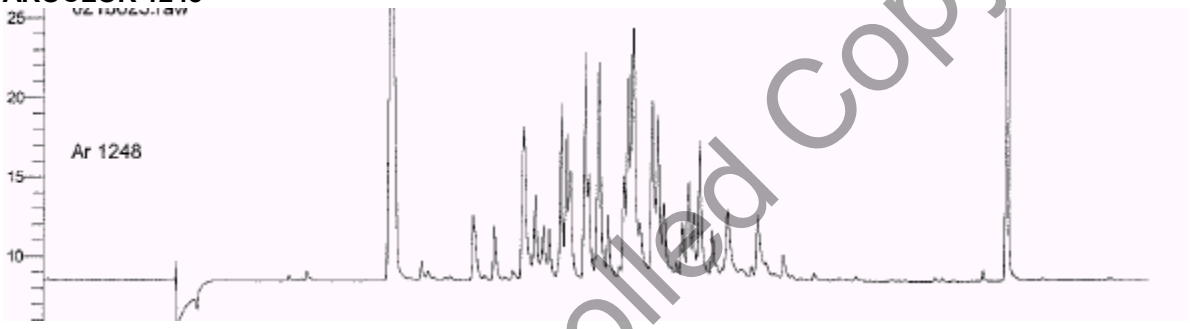
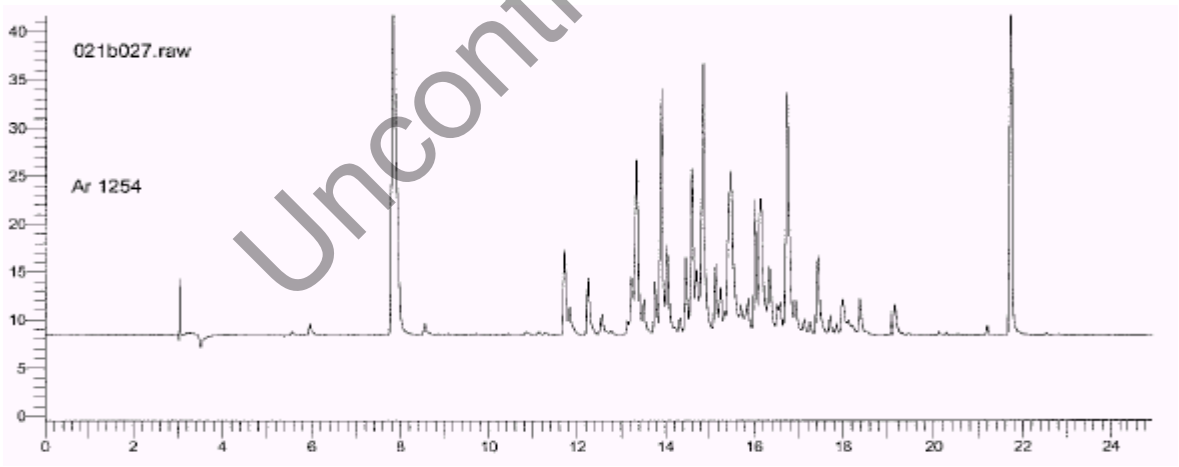
**AROCLOR 1242**




**AROCLOR 1248**




**AROCLOR 1254**

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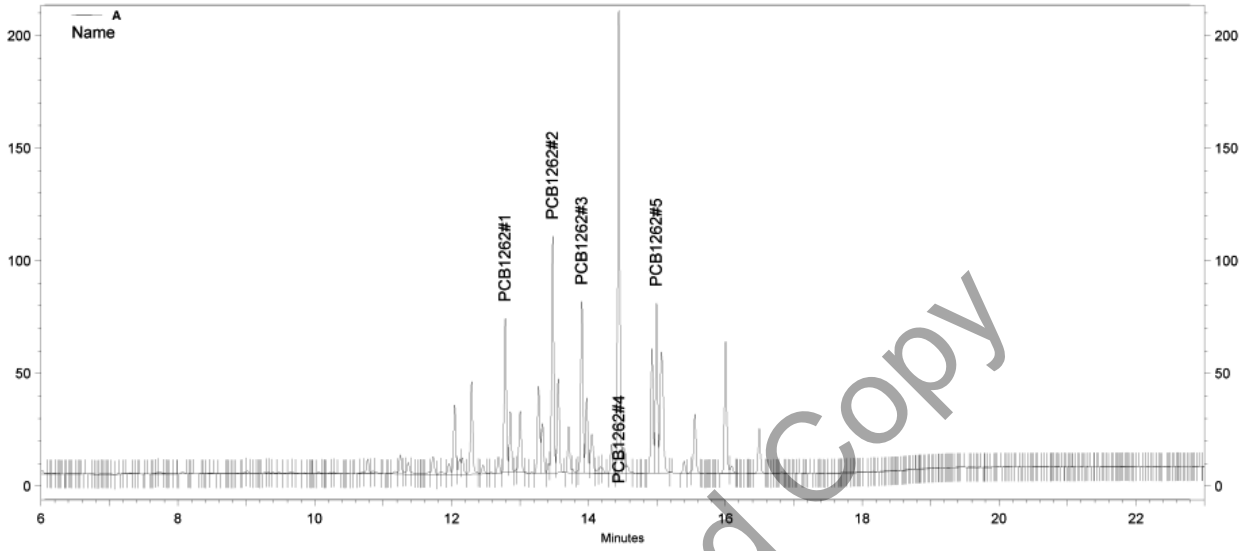
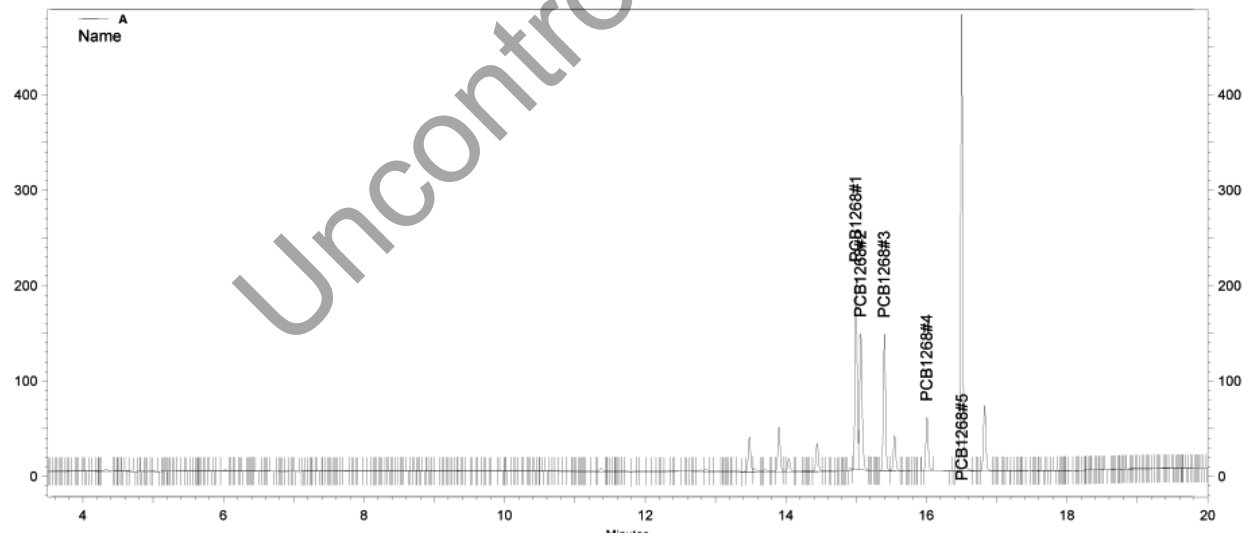
**AROCLOR 1262**




**AROCLOR 1268**

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## APPENDIX_8: TARGET COMPOUNDS & REPORTING LIMITS

| CAS # | Compound | Water RL µg/L | Soil RL µg/Kg | Wipe RL µg/wipe |
|---|---|---|---|---|
| 12674-11-2 | Aroclor-1016 | 0.5 | 12 | 2.5 |
| 11104-28-2 | Aroclor-1221 | 1 | 24 | 5.0 |
| 11141-16-5 | Aroclor-1232 | 0.5 | 12 | 2.5 |
| 53469-21-9 | Aroclor-1242 | 0.5 | 12 | 2.5 |
| 12672-29-6 | Aroclor-1248 | 0.5 | 12 | 2.5 |
| 11097-69-1 | Aroclor-1254 | 0.5 | 12 | 2.5 |
| 11096-82-5 | Aroclor-1260 | 0.5 | 12 | 2.5 |

Additional Compounds (may be added to the compound list):

| | | | | |
|---|---|---|---|---|
| 37324-23-5 | Aroclor-1262 | 0.5 | 12 | 2.5 |
| 11100-14-4 | Aroclor-1268 | 0.5 | 12 | 2.5 |

Surrogates:

| | |
|---|---|
| 2051-24-3 | Decachlorobiphenyl (DCB) |
| 877-35-2 | Tetrachloro-m-xylene (TCMX) |

Reporting Limits may vary, depending on the volume submitted for analysis, preparation method used, and %moisture (if reported on a dry-weight basis). The reporting limits listed above are based on the following prep volumes & weights:

| | | |
|---|---|---|
| Water Samples: | Initial Volume = 1,000 mL | Final Extract Volume = 25 mL |
| 3550 Soil Samples: | Initial Weight = 30 g | Final Extract Volume = 25 mL |

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**APPENDIX_9:** **INITIAL CALIBRATION**

Procedure & Acceptance Criteria

## A. REQUIREMENTS

An initial calibration curve must be analyzed whenever instrument conditions (temperature programs, flow rates, etc.) have been changed, the detector has been cleaned, or when a new column has been installed. In general, a new calibration curve must be made whenever instrument conditions have been altered, or whenever the continuing calibration verification no longer passes acceptance criteria.

The instrument analytical range must be established by running a minimum of 5 calibration standards, containing Aroclor 1016 and Aroclor 1260, at levels that bracket the quantitation range (see Appendix_2 for standard suppliers); the lowest standard must be at or below the reporting limit and the highest standard determines the upper end of the quantitation range. The standards must be analyzed in order of increasing concentration. Points may be dropped so long as the following criteria are met:

- The highest concentration standard may be omitted so as long as there are at least five points remaining and the remaining highest point defines the top of the calibration range (any extracts which exceed this response must be diluted and reanalyzed).

- The lowest concentration standard may be omitted from curve if, *and only if*, the resulting lowest standard is at or below the reporting limit for samples and there are at least five points remaining. For surrogates, the lowest concentration standard must be ≤10x of the level used to spike the samples and QC.

- Mid-point standards may not be omitted simply to improve the RSD or linear correlation coefficient. They may, however, be reanalyzed if a poor injection is suspected. The reanalysis must occur immediately after the curve so long as no sample extracts were analyzed since the last calibration standard and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.

The curve must be verified by analyzing an Initial Calibration Verification (ICV) standard comprised of standards obtained from a different manufacturer than those used to prepare the ICAL standards. See Appendix_1 for calculation of response factors, RSD, and correlation coefficient.

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**Initial Calibration Levels (pg)**

| Compound | Level 1 | Level 2 | Level 3 | Level 4 | Level 5 | Level 6 | Level 7 |
|---|---|---|---|---|---|---|---|
| Aroclor 1016 | 10 | 25 | 100 | 250 | 500 | 750 | 1,000 |
| Aroclor 1260 | 10 | 25 | 100 | 250 | 500 | 750 | 1,000 |
| | | | | | | | |
| Decachlorobiphenyl (DCB) | 2 | 5 | 20 | 50 | 100 | 150 | 200 |
| Tetrachloro-m-Xylene (TCMX) | 2 | 5 | 20 | 50 | 100 | 150 | 200 |

*Note:* EPA 8082 Section 5.6.1 specifically allows the use of an Ar1016/1260 mix to establish the linearity of the detector and a single-point initial calibration to establish the response factor for the remaining Aroclors, however EPA 608 and the DoD QSM require a multi-point calibration if the pattern is detected in any of their samples. Check the project notes to verify the requirement for samples associated with any DoD project.

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## B. PROCEDURE

For each of the Aroclors, select three to five representative peaks as the basis of quantitation and assign a unique identification (i.e.'PCB1260 #1', 'PCB1260 #2', etc.) to each peak. LIMS will calculate the calibration factor for each of these peaks in each of the calibration standards.

If the retention *times* have changed significantly, analyze the 5-point Ar1016/1260 along with a single-point for each of the other Aroclors, for pattern recognition. If samples are being analyzed by EPA 8082 and one of the other Aroclors is identified in the extracts, the calibration verification standard immediately preceding the sample can be used to used to establish the calibration factor for that Aroclor, so long as the 1016/1260 ICAL has demonstrated the linearity of the detector (as per 8082 Section 5.6.2).

***EPA 608** Method Note:* If samples are being analyzed for EPA 608, a multi-point calibration must be run for any of the other analytes if they are detected in the sample.

**Run the Initial Calibration Standards:**

1. Prepare the standards as described in Appendix 2.

2. Prepare an Initial Calibration Verification (ICV) standard from source standards obtained from a different manufacturer than the ICAL standards.

3. Perform any needed instrument maintenance and run a "primer" at about 2,000 ug/L followed by a hexane instrument blank. If any target compound is detected above the reporting limit, run another instrument blank.

4. Load the calibration standards onto the autosampler tray in order of increasing concentration.

5. Add instrument blanks before and after the ICAL standards to demonstrate that the low-level standard was not influenced by instrument contamination and that analytes at the high-level concentrations will not carryover into real-world samples.

6. If the retention times of the analytes in the new calibration curve are expected to be significantly different than the previous calibration curve, due to instrument maintenance such as column maintenance or changed flow or temperature parameters, also include a single calibration standard of each of the other Aroclors in the sequence.

7. Load the ICV after the instrument blank that follows the calibration standards. The ICV, prepared from standards obtained from a second manufacturer, must be analyzed to verify that the standards used to create the initial calibration curve were prepared correctly.

8. In the sequence spreadsheet, make sure the new method is entered. To change the method, select the first box in the METHOD column and click the green diamond button. Slect the new method and click OPEN. Right click on the first box in the METHOD column and select FILL DOWN.

   *Note:* Always make sure the new method is created and entered into the sequence BEFORE processing an ICAL or updating retention times. This ensures that you do not change and overwrite the old method.

9. In the SAMPLE ID column, enter ICAL points below, identifying the type of sample as initial calibration standards, the LIMS identification of the standards, and the applicable dilution factors. The “stype” and S# must be correctly entered into the sequence in a specific order for LIMS to be able to interpret the information.

   primer
   hexane
   ICAL, S#, PCB10_2
   ICAL, S#, PCB25_5
   ICAL, S#, PCB100_20
   ICAL, S#, PCB250_50
   ICAL, S#, PCB500_100
   ICAL, S#, PCB750_150
   ICAL, S#, PCB1000_200
   hexane
   ICV, S#, ULTRA_1660

   Where:
   S# is the LIMS S# of the standard used

10. Save the sequence by going to FILE > SEQUENCE > SAVE.

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11. Run the ICAL, following the steps on "Running a Sequence in EZChrom" as described in Appendix_11.

**Process a Multi-Point ICAL in EZChrom:**

12. Make sure that you have created and entered a new method into the sequence before processing an ICAL. Also make sure the new method is currently open in your EZChrom session.

13. Check the integrations of the Initial Calibration data files and work up the data using the integration events outlined in Appendix_11. Make sure all of the analytes are correctly identified for every calibration level on both channels. If you need to change the retention times, see "Updating the Retention Times" in Appendix_11.

    Peaks should be integrated from baseline to baseline. Manual integrations of any kind must be substantiated and documented on the Initial Calibration Report. Manual integrations must be consistently applied to all ICAL, CCV, and sample integrations. **Unsubstantiated alteration of peak integration solely to pass calibration criteria is illegal and is grounds for immediate termination.** If the reason for the integration is not intuitive and obvious, the analyst must document the reason on the data.

14. Once the chromatograms are properly integrated, process the data. In the sequence spreadsheet, change the run type by selecting the row for the run and clicking the blue triangular button under the RUN TYPE column. The Sample Run Type(s) window should open. Check CLEAR CALIBRATION AT LEVEL and click OK. Change all of the ICAL runs to this run type.

15. In the LEVEL column, enter the appropriate calibration level.

    *Note:* To find the calibration level, open the "Peaks & Groups" table by going to METHOD > PEAKS/GROUPS. There is a column for each level with the spike amount entered for the relevant analytes.

16. Process the ICAL by going to SEQUENCE > PROCESS. Type the run numbers for the ICAL runs in RANGE and make sure the box for PRINT METHOD REPORTS is checked.

    *Caution:* Processing the ICAL will overwrite the old calibration in the method. Make sure that the new method is entered into the sequence before processing!

## DATA REVIEW & APPROVAL

Review the calibration in EZChrom before processing it in LIMS.

17. Go to METHOD > REVIEW CALIBRATION to verify that the calibration is linear and that the upper levels are not saturated. Click the name of the analyte to see the %RSD for that compound. To switch channels, use the drop down list on the main toolbar.

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18. Verify that the calibration is useable by reviewing the results against the following criteria:

    18.1 The Relative Standard Deviation (RSD) of the Response Factors for both columns should be less than or equal to 20%.

    ***EPA 608*** *Method Note:* For EPA 608, the RSD must be ≤10%

    18.2 If the RSD fails for any compound, compute the "grand mean" of the RSDs for all target compounds; if this mean RSD is less than or equal to 20% then the average response calibrations may be validated (see EPA 8000B Section 7.5.1.2 for details).

    *Note:* For Department of Defense (Army Corp, Navy, AFCEE) projects, no individual compound's RSD may be > 30% (DoD QSM, Box 33 and Table B-2).

    ***EPA 608****:* For EPA 608, the RSD must be ≤10% and the method does not allow use of this "Grand Mean" concept; each analyte must meet the 10%RSD criteria.

    18.3 If the average response factors fail to meet the 20% RSD or mean RSD criteria, employ a linear regression or quadratic model. If linear regression is used, a minimum of five points is required with a correlation coefficient $r \geq 0.995$ If a quadratic fit is used, a minimum of six points is required with a coefficient of determination $r^2 \geq 0.990$.

    18.4 For each compound:

    - The low point may only be rejected for those compounds that have reporting limits greater than that level.
    - The high point may be rejected for compounds that tend to saturate at high levels so long as there are at least 5 points remaining for each compound in the ICAL.
    - If a single point in the curve is causing the failure, the standard may be reanalyzed, so long as it immediately follows the original curve and all compounds are calibrated using the second run. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria for a particular compound.
    - The %D for recalculated concentrations should be within 20% of the true concentration of the standard (8000C requirement).

19. Save the newly calibrated method by going to FILE > METHOD > SAVE.

20. Open the Initial Calibration Verification (ICV) data file and verify that all compounds are integrated correctly. Process the ICV with the newly calibrated method.

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The ICV verifies that the calibration standards were prepared correctly and highlights any discrepancies between the primary- and second-source standards.

The ICV should meet the CCV criteria of ≤ 15%D. If the %D for any analyte is outside this limit, calculate the mean %D for all compounds. If this is less than 15% then the CCV is considered acceptable.

*Note:* For DoD QSM projects, no individual compound's %D may be > 20%.

If the first ICV does not meet the acceptance criteria, another ICV standard may be analyzed; "x" out the first ICV and process the data from the second ICV. *Do not* "cherry pick" some compounds from the first ICV and others from the second ICV; if the second ICV is processed and used, all compounds must be taken from the second standard.

**Processing an ICAL in LIMS:**
After you've verified that the calibration passes all acceptance criteria, process the ICAL in LIMS.

21. Open a web browser and go to the main Pesticides/PCB page.

22. Under "Recent Sequences", choose the GC and then choose the sequence you want to process.

23. Check the boxes next to each of your ICAL data files. Click the CREATE CALIBRATION button at the bottom of the screen.

24. When the Calibration Editor opens, unselect any analytes that are not being calibrated (ex: Toxaphene and Technical Chlordane).

25. Type a name for the calibration (ex: gc21pest_051, where 051 was the sequence on gc21).

26. Click SAVE + PUT INTO USE and return to the sequence.

27. At the top of the sequence, the newly created calibration should be listed. Click REVIEW to open the Review App for the calibration.

28. Sign and review each of the runs. See Appendix_12 for guidelines on using the LIMS Review App.

**Packaging the ICAL for Review:**
29. Print the LIMS ICAL summary. Click the calibration name at the top of the sequence. Go to the bottom of the on-screen ICAL summary and click the PRINT button.
30. Print the EZChrom Calibration Report by going to METHOD > REVIEW CALIBRATON.

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31. Right click in the box at the top left with the list of analytes and select PRINT ALL PEAKS/GROUPS to print a Calibration Report for each analyte.

32. Switch to the other EZChrom channel and print the Calibration Report for that channel.

33. Verify that the LIMS and EZChrom calibration factors match.

    *Note:* Any corrections to the ICAL must be done through EZChrom and then printed to ezchrom_capture to send the new data to LIMS. A new ICAL must then be created in LIMS. Any data processed with the incorrect ICAL would then need to be reprocessed against the corrected ICAL.

34. Sign the calibration in the LIMS Review App and send it in for review; the ICAL cannot be used to process final forms through LIMS until it has been reviewed and approved in LIMS.

The calibration must be reviewed and 2nd-party approved in LIMS before final sample Form 1's can be printed; any forms printed before the ICAL is marked 'reviewed' will be flagged as draft results.

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## APPENDIX _10: CONTINUING CALIBRATION VERIFICATION (CCV)

### REQUIREMENTS

Continuing Calibration Verification (CCV) standards are analyzed to verify that the response of the instrument has not changed significantly and that the curve may still be used to quantitate sample results. A CCV must be run at the beginning of each sequence before any samples are analyzed, after every 10 samples (or within 12 hours, whichever comes first), and at the end of the analytical sequence. The concentration of the CCV must be varied within the calibration range but should not be analyzed at either extreme (highest or lowest point) of the ICAL curve.

LIMS identifies samples that are associated with the DoD QSM requirements, other DoD requirements or commercial clients. It identifies all project specific criteria that are reviewed by the analyst before sample analysis.

For sequences containing samples that are not associated with requirements from DoD QSM, if the first CCV does not meet the acceptance criteria, another CCV standard should be analyzed. If two CCV's were analyzed, examine the first one against the acceptance criteria; if it fails, "x" out the first CCV, change the second to stype "CCV" and process the data from the second CCV. *Do not* "cherry pick" some compounds from the first CCV and others from the second CCV; if the second CCV is processed and used, all compounds must be taken from the second standard.

If the second analysis of the standard also fails to meet these criteria and the analyst suspects that the CCV standard has degraded, a different CCV standard may be analyzed once. If this standard passes, discard the standard that has been degraded. If the different CCV standard also fails, instrument maintenance required and recalibration may be required if major instrument maintenance is performed.

For sequences where both DoD QSM samples and non-DoD QSM samples are analyzed, both sets of CCVs will be run and integrated and both CCVs must pass the DoD QSM criteria. If the entire sequence contains DoD QSM samples only, then only one CCV will be analyzed.

See Appendix_9 for the Initial Calibration procedure and acceptance criteria. See Appendix_1 for calculation of %D and the calibration factor (CF).

### ACCEPTANCE CRITERIA

Process the CCV through LIMS then examine the summary form against the following criteria to determine whether the CCV is acceptable:

1.) All compounds must fall within its retention time window and be automatically identified on both columns by the data system.

2.) All compound responses should be within 15% of the initial calibration (%D $\leq$ 15%).

3.) If the %D for an individual compound fails acceptance criteria, data may be reportable based on the following criteria:

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a.) If the failing compound is not a target analyte for the associated samples, sample results should be reported without reanalysis.

b.) If the compound fails the %D criterion due to a high response but was not detected above the reporting limit in the associated samples, the sample results may be reported without reanalysis, as the high bias does not affect the sample results.

c.) If the compound fails the %D criterion due to a high response and *was* detected above the reporting limit in any of the associated samples, the samples should be reanalyzed.

d.) If the compound fails the %D criterion due to a low response and *was* detected (even below the reporting limit), the sample should be reanalyzed.

If any of the above criteria are not met, examine the integration to verify that each peak was correctly integrated. Manual integrations must be consistently applied to ICAL, CCV, and sample integrations. If manual integrations are performed, the file should be resent to LIMS so that a new CCV summary form can be generated.

If associated samples are being quantitated for a hit of one of the non-Ar1016/1260 PCBs based on a single-point calibration, the bracketing Ar16/60 CCVs on that channel must pass, as the linearity of the calibration is demonstrated by the Ar1016/1260.

If a non-Ar1016/1260 PCB is being quantitated from a 5-pt calibration and the bracketing CCVs of that analyte pass, the sample(s) may be quantitated for that analyte regardless of the Ar1016/1260 CCVs failure.

**WARNING:** **Unsubstantiated alteration of peak integration solely to pass calibration or QC criteria is illegal and is grounds for immediate termination.**

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## APPENDIX_11: USING EZCHROM

**Setting up a Sequence on EZChrom:**

1. Open EZChrom Elite.

   1.1 Click PCB/PEST LAB.

   1.2 Right click on the GC that will run and select either OPEN (online) or OPEN OFFLINE.

   1.3 In USER NAME and PASSWORD, type the appropriate information.

   1.4 In PROJECT, find the GC you are for which you are opening the session.

   1.5 Click LOGIN.

   Note: You can write a sequence in either an online or offline session, but you can only run the sequence in an online session.

2. Open the latest sequence.

   2.1 Go to FILE > SEQUENCE > OPEN.

   2.2 Choose the latest sequence filename (###.seq).

   2.3 Click OPEN.

   This sequence will be used as the template to set up the next sequence.

3. Create a copy of that sequence.

   3.1 Go to FILE > SEQUENCE > SAVE AS.

   3.2 In “File Name”, type in the current Julian date.

   3.3 Click SAVE.

4. Change the filenames to use the current Julian date.

   4.1 Right click on the first box in the FILENAME column.

   4.2 Select FILL DOWN.

   4.3 In the DATA FILE box, type the Julian date, underscore, and the code for LINE NUMBER (eg 167_<###>).

   4.4 Click OK.

5. Re-number the vials under the VIAL column by right-clicking the first box and selecting FILL DOWN.

   5.1 Check INCREMENT VIALS, enter the vial placement of the starting vial, and increment by 1.

6. In the VOLUME column, enter the appropriate injection volume in µL (see Appendix_4 for the injection volume) and FILL DOWN.

   *Note:* The CombiPal Autosamplers are not controlled by EZChrom so the sequences run with these autosamplers do not use the VIAL and VOLUME columns. These parameters must be entered in the method on the autosampler itself.

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7. In the sequence spreadsheet, make sure that that current method is being used.

   7.1 To change the method, select the first box in the METHOD column and click the green diamond button.

   7.2 Select the appropriate method (e.g. pest-run-###.met) and click OPEN.

   7.3 Right click on the first box in the METHOD column and select FILL DOWN.

8. In the SAMPLE ID column, enter what will run in the sequence in the format seen in the following example of a sequence table:

| Run Type | Level | Vial | Volume (µL) | Sample ID | Method | Filename |
|---|---|---|---|---|---|---|
| Unknown | 0 | 1 | 0.5 | Primer | pcb-run-<###> | <###>-001 |
| Unknown | 0 | 2 | 0.5 | Hex | pcb-run-<###> | <###>-002 |
| Unknown | 0 | 3 | 0.5 | CCV,S<#>,pcb250_50 | pcb-run-<###> | <###>-003 |
| Unknown | 0 | 4 | 0.5 | CCV,<S#>,ar2154 | pcb-run-<###> | <###>-004 |
| Unknown | 0 | 5 | 0.5 | Hex | pcb-run-<###> | <###>-005 |
| Unknown | 0 | 6 | 0.5 | MB,QC<###>,<batch#> | pcb-run-<###> | <###>-006 |
| Unknown | 0 | 7 | 0.5 | LCS,QC<###>,<batch#> | pcb-run-<###> | <###>-007 |
| Unknown | 0 | 8 | 0.5 | <sample#>,<batch#> | pcb-run-<###> | <###>-008 |
| Unknown | 0 | 9 | 0.5 | MS,QC<###>,<batch#> | pcb-run-<###> | <###>-009 |
| Unknown | 0 | 9 | 0.5 | MSD,QC<###>,<batch#> | pcb-run-<###> | <###>-010 |

   "Run Type" and "Level" should be "Unknown" and "0" respectively, unless an initial calibration curve is being run; see Appendix_9 for instructions on processing calibrations using EZChrom.

   The "Vial" and "Volume" columns are not used for sequences run on a CombiPal Autosampler.

20. Save the sequence by going to the menu and clicking FILE > SEQUENCE > SAVE.

**Running a Sequence on EZChrom:**

1. Open an online session on EZChrom for GC you want to run.

2. Open the sequence you want to run.

   2.1 Go to FILE > SEQUENCE > OPEN.

   2.2 Choose the sequence filename (###.seq).

   2.3 Click OPEN.

3. Start the sequence.

   3.1 Going to CONTROL > SEQUENCE RUN.

   3.2 In the RANGE box, type the range of the sequence you want to run.

   3.3 Click START (or SUBMIT if the GC is already running and acquiring data).

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4. You will need to start the CombiPal Autosampler (GC-21 & GC-25) manually because EZChrom does not control that feature.

**Setting up the Sequence on the CombiPal Autosampler:**

1. The main screen on the CombiPal controller should list the Job Queue. There should already be a job listed from the previous run. Turn the silver knob to outline the job. Press the silver button to select the job for editing.
2. Change the position of the first and last vials to be run. Turn the knob to outline FIRST and press the silver button to select. Turn the knob until the number reaches the first vial number you want to run. Repeat the process with LAST to set the last vial you want to run.
3. Press F4 to select HOME and return to the Job Queue menu.
4. Press F4 to select START to start the autosampler; there may be a slight delay.

*Note:* Remember to start the Sequence Run on EZChrom before starting the autosampler, otherwise the autosampler may inject and start the GC run before EZChrom is ready to acquire the data.

**Processing Data on EZChrom:**

1. Open EZChrom an OFFLINE session for the GC from which you will process data.

   Note: You can process data in an online session, however, it is best to have only one online session open for each GC at a time to ensure that only one user is running a sequence on that GC.
2. Open the sequence you want to run.
   - 2.1 Go to FILE > SEQUENCE > OPEN.
   - 2.2 Choose the sequence filename (###.seq).
   - 2.3 Click OPEN.
3. EZChrom sessions usually opens with a default “untitled” method, so you will need to select the correct method.
   - 3.1 Open the current method by going to FILE > METHOD > OPEN.
   - 3.2 Choose the method filename (pset-run-###.met).
   - 3.3 Click OPEN.
4. Open the data file by right clicking on the row of the run in the sequence. Choose OPEN DATA. The chromatograms for the two channels should appear.
5. Check the integrations for each target analyte.
6. If you need to do any integrations, the following are the common integration events:

| | |
|---|---|
| Valley to Valley | ***Draws the baseline to the minimum point between the peaks.*** |
| *Horizontal Baseline* | ***Draws the baseline horizontally determined by the beginning point in the range.*** |

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| | |
|---|---|
| Valley to Valley | ***Draws the baseline to the minimum point between the peaks.*** |
| *Lowest Point Horizontal Baseline* | *Draws a horizontal baseline determined by the lowest point in the range.* |
| *Manual Baseline* | *Draws a straight baseline from one point to another.* |
| *Split Peak* | *Draws a vertical line from the point to the baseline. It is commonly used to determine the end and beginning of a peak.* |
| *Reset Baseline* | *Draws the baseline up to a point on the chromatogram. It is commonly used to bring the baseline up due to a negative peak.* |

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After performing an event, a box will pop up and ask you to ANALYZE NOW.

*Important!* Before clicking ANALYZE NOW, make sure that you are inserting the event into the MANUAL Integration Fixes table. Inserting an event into the Integration Events table will add the event to the Method.

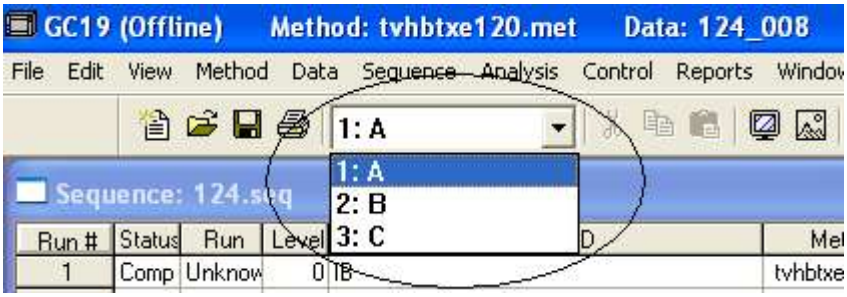
If you want to remove an integration event:

6.1 Open the Manual Integrations Fixes table by going to DATA > MANUAL INTEGRATIONS FIXES.

6.2 Select the integration you want to remove and press DELETE on your keyboard.

6.3 You will then need to analyze the data by going to ANALYSIS > ANALYZE.

To change what channel the table displays, select the channel from the drop down list on the main toolbar, as shown below:




7. Repeat Steps 4 through 6 for the other data files. After you have finished any integration, you need to send the data to LIMS by printing the Method Custom Reports for the data to the ezchrom_capture printer.

   7.1 Go to SEQUENCE > PROCESS.

   7.2 Type the range of data you want to process in RANGE and make sure the box for PRINT METHOD REPORTS is checked.

   Note: Make sure that the printer configured to your EZChrom session is set to ezchrom_capture. You can check the printer by going to FILE > PRINT SETUP.

8. Some runs will need to be reprocessed with a different method (ex: pcb-ar2154-142). To do this, change the method in the sequence table for those runs and save the sequence. Repeat Steps 3-7, now processing with the new method.

9. After EZChrom is finished processing the data, review the data in LIMS.

**Making Compare-Plots ("Trace Overlays") in EZChrom:**
If there is Toxaphene or Technical Chlordane in the samples, create a compare-plot to show the similarities of the sample pattern to that of a standard for that analyte.

1. Open the sample's data file in EZChrom.

2. Right click on the chromatogram from the channel you choose to use for the compare-plot and select PROPERTIES.

3. In the TRACE SETUP tab under Trace 1 Details (Current Data), change SCALE TO: to USER DEFINED. Change the values from Y MIN and Y MAX to leave enough space to add the CCV chromatogram(s).

4. At the top of the Data Graph Properties window, select the box under DATA SOURCE at row # 2. Click the right arrow and select OPEN DATA. Type the filename of the CCV data you will use for the compare-plot and click OPEN.

5. In the new trace's details, click the button to the right of TRACE to change the channel of the new trace to match the sample's chromatogram. Change the Y OFFSET to move the baseline of the new trace so that it does not overlap with the other chromatogram(s). You can also change the Y SCALE to size the chromatogram to better match the size of the sample's hit.

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*Note:* Choose CCV data from the same sequence and use the same channel for each of the chromatograms, so the retention times match.

6. To change the color and line-width of the chromatogram, go to the APPEARANCE tab in the Data Graph Properties window. Under ITEM, choose the chromatogram. With SUBITEM at TRACE, change the SIZE and COLOR.

7. Repeat Steps 3 through 6 for additional chromatograms.

   *Note:* You can click APPLY while you are changing values in the Data Graph Properties Window to see a preview of your changes.

8. Once you are finished adding traces and changing their appearance, click OK.

9. Send the chromatogram to LIMS by printing the compare-plot to ezchrom_capture. Change the print layout to Landscape by going to FILE > PRINT SETUP. Change the Orientation to LANDSCAPE then click OK. Right click the chromatogram and select UTILITIES > PRINT.

10. You can use the same added traces for other sample data chromatograms. When you open another data file in the same EZChrom session, the added traces should remain. You will just need to edit the chromatogram and select CLEAR OVERLAYS.

    *Note:* Remember to change the PRINT SETUP orientation back to PORTRAIT, otherwise the method reports will print in landscape mode.

**Updating the Retention Times:**

1. Create and enter a new method into the sequence before updating the retention times, then make sure the new method is currently open in your EZChrom session.

2. Open the data file you will use to update the retention times.

   *Note:* Retention times should be updated based on the middle calibration point.

3. Add the Retention Time Annotation to the chromatograms by right-clicking on any of the chromatograms and selecting ANNOTATIONS. Under AVAILABLE ANNOTATIONS, select RETENTION TIME and click the green right-arrow. Click APPLY TO ALL so that both chromatograms will display the retention times.

4. Open the peaks and groups table by going to METHOD > PEAKS/GROUPS. Using the retention times from the chromatograms, update the retention times under the column RET TIME. To change which channel is displayed, select the channel from the drop-down list on the main toolbar.

After you have changed the retention times on each channel, save the method by going to FILE > METHOD > SAVE.

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## APPENDIX_12: PROCESSING & REVIEWING DATA IN LIMS

**Work up a sequence in LIMS.**

1. Process sequence in EZChrom, making sure EZChrom print setup is set to \\LIMS\ezchrom_capture.

2. Open a web browser and go to the main Pesticides/PCB page.

3. Under "Recent Sequences", choose the GC and then choose the sequence you want to process.

   *Note:* The data must be processed through EZChrom and printed to ezchrom_capture before you can process it through LIMS. See Appendix_11 above for instructions on processing data through EZChrom.

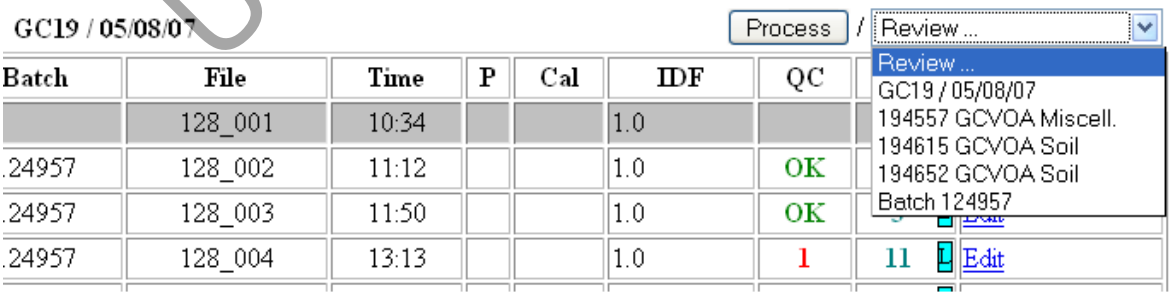
4. Check the sequence for any errors. LIMS takes the run information directly from the EZChrom sequence so any data entry error in the EZChrom sequence will affect the data processed by LIMS.

   If you need to make any changes to the sequence, click FIX at the lower right-hand corner of the LIMS sequence. Make the changes and click Update.

   To regenerate a LIMS user report for a specific run, check the box for the run and click PROCESS.

5. Once the sequence information is correct, use the 'Review App' to review the data, as described below.

6. In the top right, use the "Review" pull down menu and select your Sequence (for example: GC19A/ 05/08/07).




7. Two windows should open. The window on the left shows the main Review App frame (sequence, batch, ICAL) and the other shows the supporting documents (ie: EZChrom raw data reports, scanned prep logs). Clicking on an item in the left-hand frame will bring up the supporting documents for that item.

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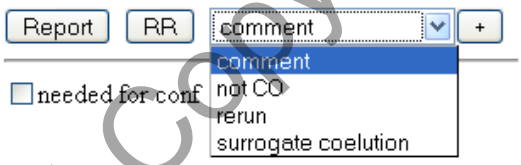
***Important!*** You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

8. Check each run to ensure it is within the acceptable criteria, adding any necessary comments, and signing the data.

   8.1 Scroll down the left screen and make sure the EZChrom TEH raw data and surrogate raw data reports match LIMS, and check for the proper chromatograms.

   8.2 To add a general comment, click the (+) button while the pull down list to the left is set at COMMENT.




   A comment box will appear with your initials.

   Type your comment into the blank box.

   If you want to associate the comment with a certain analyte in the run, you can choose that analyte on the pull down list to the right of the comment box.

   If you want to delete the comment, click the (-) button.

   If you need to edit a comment you made at an earlier time, click the (E) button.

   Other choices under the Comment pull down list will add commonly used comments.

   8.3 Mark data for re-analysis by clicking the 'RR' button. Any re-runs that are not because of linear range will require a brief comment as to why the data needs re-analysis. To undo an RR-flag, click the REPORT button.

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8.4 For over linear range or over diluted samples, use the RR button with the rerun dropdown tab selected and type in the dilution factor needed. Finally, use the RX button for any re-extracts.

8.5 If the run is needed to confirm the results of another run, check the box 'NEED FOR CONF'.

8.6 Note that each analyte can only be 'chosen' once for a sample or QC. To choose analytes for reporting, check the box under the 'U' ("used") column; checking the box at the left of the 'U' header will choose all analytes for that run.

8.7 To change the reporting channel, check the channel for the analyte that needs to be changed or switch the whole list by clicking the CHANNEL pull-down list and choosing 'ALL A' or 'ALL B'. Click REFRESH to apply the changes.

9. Sign the data by clicking the SIGN button at the bottom right. Repeat the process with the other runs in your sequence.

**Assemble a Batch in LIMS**

Batch QC packages can be put together after the extraction lab has scanned in all the sample prep paper work, and all the QC samples (including the MSS and any necessary re-runs) have run and their brackets have been closed off with CCVs.

1. Use the C&T LIMS search function and type in batch number. This will find all data associated with the batch. Use the review apps pull-down menu and select batch number (ie: batch 121060).

2. Log in if necessary.

   ***Important!*** You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

3. In Review Application mode on left screen, the runs for the QC will appear along with associated ICALs, CCVs, and sequences. The associated CCVs should already be signed. If any are not signed, review and sign them.

4. Click the batch number at top of screen. Scanned documents should appear on right screen. Scroll down and check for any Corrective Action Records (CAR's) associated with the batch or with samples in the batch. Review and update the CAR.

5. Make sure that all extraction lab paperwork has been scanned correctly.

6. Review the sample prep log to make sure volumes, spike, and surrogate amounts are entered correctly in LIMS (data on left screen). Also look for any comments in the comment section, such as "2x surrogate" or "sediment".

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7. Click on each QC data file and verify that all QC requirements (clean MB, analyte recovery, surrogate recovery, RPD) are met and that any necessary comments explaining recoveries that are outside of QC limits are present.

8. Check that the LIMS raw data numbers match the EZChrom data.

9. Sign each QC file, signifying that the data has been reviewed and is reportable or has been narrated if it is not being reported.

10. Verify that the run of the MSS that you are reporting is the same as the one that is linked to the MS/MSD. If the MSS was re-run for any reason, the run that we are reporting may not be linked correctly. If necessary, change the MSS run that is linked to the MS/MSD using the "MSS" link from the sequence where the MS/MSD ran. It must be changed for both the MS and the MSD individually. Be sure to make comments about any MS/MSD recovery or RPD failure.

11. Add any necessary comments to the batch as a whole using the comment tool at the bottom of the screen. For example, this would be where you could explain that the MS/MSD were not run if the MSS was run at a dilution >5x.

12. Finally, click the "Sign" button when the batch QC pack is complete and ready for review.

13. Notify a QC reviewer that it is ready (by instant message, telephone, or in person).

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**Assemble a Job in LIMS**

1. Review the job sheet. Check to make sure the correct cleaned-up extract of samples and QC has run and are ready to report. Make sure that any client-specific needs are addressed (for example, CCI needs to be on one instrument). Finally, search for any CARs associated with job and make any comments needed to complete the CAR.

2. Once you know what you need to report, begin by using the C&T search tool and type in job #, product (gcsv), and matrix (ie: 194505 gcsv soil). This will bring up sample data in LIMS. In the top right, use the "Review" pull down menu and select the job you would like to report.

3. Login if necessary.

   ***Important!*** You must be logged in with your own initials to process and review sample data! If another analyst has been using the computer, make sure to login under your name.

4. Go to the 'ALL" mode.

5. Make sure all of the samples in the job have run and the data is reportable. Sign and review all of the samples in the job and their associated batch QC. Make sure that the analytes you want to report have been 'chosen'.

6. Switch to PKG mode. Click the job number at the top left.

7. Click REPORTS. Check all the relevant samples and QC, then click PRINT to print all of the Form 1's on 2nd -page letterhead paper. Click DONE.

8. You may need to click REFRESH to see the Form-1 information on the Review App.

9. Check the Form-1 for any errors, missing analytes, and draft-flags.

10. Click the Job at the top of the list. Check the comments for any issues that you need to address, like missing signatures and documents.

11. Sign the job. Click CHECKLIST > PRINT to print the review checklist.

12. Complete and sign the checklist. Turn in the checklist and Form-1's to the QC Chemist, Group Leader, or Department Manager for review.

13. Remember to log out by clicking on your initials.

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**APPENDIX_13: EPA 608**

The following criteria and discussion apply to any samples submitted for the EPA 608 analysis:

**Initial Calibration:**
Method 608 does not allow the use of single-point calibrations for Ar1221 through Ar1254. It only requires 3 points but requires that the RSD be ≤10% if average response is used; a regression curve may be used if the RSD exceeds 10% or whenever desired. Although 608 only requires 3 calibration points, the same general calibration criteria must be followed (ie: intermediate levels may not be dropped and the low point must be at or below the reporting limit). Any calibrations to be used for both 8081 and 608 must meet the 608 RSD requirement or use linear regression. Additional details regarding the procedure and SW-846 requirements are described in Appendix_9 above.

**Calibration Verification (ICV/CCV):**
Method 608 also only requires that a CCV (Continuing Calibration Verification) standard be analyzed once daily, however C&T policy is to follow the SW-846 guidance and analyze the CCV at the beginning of each sequence, every 12 hours, and at the end of the sequence. The %D for each analyte must be ≤ 15% and EPA 608 does not allow use of the "Grand Mean"; if the CCV does not meet this criterion, use the corrective action guidance described in Appendix_10 to determine the appropriate course of action.

**Surrogates:**
Method 608 does not discuss use of surrogate compounds, however C&T standard practice is to follow SW-846 guidance and use surrogates as a means of verifying the efficiency of the extraction and analysis. See Section 8 in the main body of this document for further guidance.

**Batch QC Requirements:**
Method 608 requires an LCS for every 10 samples, so any batch with more than 10 samples must include a BS and BSD. It also requires a matrix spike on 10% of samples submitted for the 608 analysis; if insufficient sample volume was submitted for matrix spikes, add a comment to this effect in the case narrative. EPA 608 defines initial batch QC acceptance limits in Table 3 of the method, but also states that the accuracy assessment (limits) should be updated regularly; C&T uses statistically generated limits which are routinely tighter than the method listed guidance.

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**EPA 608- Batch QC Recovery Limits**

| Target Compound | Recovery Limits (%) * |
|---|---|
| Aroclor 1016 | 50 – 114 |
| Aroclor 1221 | 15 – 178 |
| Aroclor 1232 | 10 – 215 |
| Aroclor 1242 | 39 – 150 |
| Aroclor 1248 | 38 – 158 |
| Aroclor 1254 | 29 – 131 |
| Aroclor 1260 | 8 – 127 |
| | |
| **Surrogates** | |
| Decachlorobiphenyl (DCB) | |
| Tetrachloro-m-xylene (TCMX) | |

* *Method Note:* EPA 608 only discusses LCS and MS recoveries; it specifies the same limits for the two types of spike but does not discuss duplicates or surrogates and so does not define %RPD or surrogate recovery limits.

**Instrument Conditions:**
Curtis & Tompkins uses current technology, namely capillary columns in place of the packed columns described in the method, so instrument conditions are somewhat different than those listed in the method. See Appendix 4 and instrument maintenance logs for instrument parameters.

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**APPENDIX_14: EPA TO-10A**

This document describes the analysis and quantitation of PCB's that have been collected on low-volume Polyurethane Foam (PUF) ambient air sampling devices.

Reference: *Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling, EPA/625/R-96-010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-10A, 2nd Edition, January 1999*

The PCBs are first solvent extracted using 5% diethyl ether/hexane in a Soxhlet apparatus, following C&T SOP# XLAB 8.2. The resulting extract is then concentrated and injected into a capillary column Gas Chromatograph (GC) to separate the components into discreet compounds. An Electron Capture Detector (ECD) detects those components that are halogenated compounds and the analyst uses pattern recognition to identify the Aroclor mixtures. The method only includes analysis of three Aroclors (Ar1242, Ar1254, Ar1260), however C&T clients will typically request the 7 Aroclors commonly run by EPA 8082. The reporting limits for this procedure are listed below:

| CAS # | Compound | Air RL (µg/s) |
|---|---|---|
| 12674-11-2 | Aroclor-1016 | 0.1 |
| 11104-28-2 | Aroclor-1221 | 0.2 |
| 11141-16-5 | Aroclor-1232 | 0.1 |
| 53469-21-9 | Aroclor-1242 | 0.1 |
| 12672-29-6 | Aroclor-1248 | 0.1 |
| 11097-69-1 | Aroclor-1254 | 0.1 |
| 11096-82-5 | Aroclor-1260 | 0.1 |

Surrogates:

| | |
|---|---|
| 2051-24-3 | Decachlorobiphenyl (DCB) |
| 877-35-2 | Tetrachloro-m-xylene (TCMX) |

These reporting limits listed above are based on the extraction of 1 PUF, with a final extract volume of 10mL.

TO-10A holding time: PUF: 7 days from collection to extraction
Extract: 40 days after extraction

Preservation: No chemical preservation.
Store samples and extracts at > 0.0 to 6.0 °C

**Initial Calibration**
A multi-point (minimum 5pt) calibration is required for Ar1016/1260, with a %RSD ≤ 20.

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Once the linearity of the detector is established by the Ar1016/1260 calibration, a single-point calibration is acceptable for the other Aroclors.

**Initial Calibration Verification (ICV)**
An ICV is required after any multi-point *or single point* calibration, with a %D ≤ 15.

**Continuing Calibration Verification (CCV)**
Ar1016/1260 CCVs should be run at the beginning of each sequence and after every ten samples, with a %D ≤ 15. If a non-Ar1016/1260 PCB pattern is identified in the sample(s), the run reported for that analyte must be bracketed by CCVs containing that analyte, with a %D ≤ 15.

**Sample Analysis**
Method TO-10A requires that all 5 characteristic Aroclor peaks be used in the quantitation; unlike EPA 8082, individual peaks cannot be dropped unless there is significant coelution of non-target analytes. Note, however, that the simple presence of peaks within the expected retention window does not necessarily constitute a positive identification; positive identification is based on overall pattern recognition, including the presence or absence and ratios of non-quantitated peaks.

Dilute and rerun if any of these 5 peaks are greater than the calibration range.

Report higher of two concentrations (quant vs confirm). Include compare-plot for any hits, including not only the quantitation peaks but the entire range used for pattern recognition.

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# SUBSAMPLING & COMPOSITING

| **Approved by:** | Signature: | Date: |
|---|---|---|
| John Goyette<br>Client Services Manager | | 5.19.14 |
| Cheryl Bowden<br>Operations Manager | | 5/19/14 |
| Teresa Morrison<br>QA Director | | 5/19/14 |
| **Reapproved:** | | |
| Teresa Morrison<br>QA Director | | 1/27/15 |
| Teresa Morrison<br>QA Director | | 3/29/17 |
| | | |
| | | |
| | | |

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# SUBSAMPLING & COMPOSITING

## 1.0 SCOPE

This document describes procedures for taking subsamples (aliquots) of liquid and solid samples (aliquots or subsamples) from typical sample bottles, or steel or brass drilling sleeves, to make "composite" samples.

## 2.0 REFERENCES

Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, November 2003

ASTM D-6323, Lab Subsampling of Media Related to Waste Management Activities, 2003

Improving Laboratory Performance Through Scientific Subsampling Techniques, C. Ramsey and J. Suggs, Environmental Testing & Analysis, March/April 2001

*Associated C&T SOPs & Guidance Documents:*
C&T SOP 1.4, Balance Calibration Checks & Maintenance
TNI Standard, Volume 1, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2, October 2010
DoD/DoE Consolidated QSM, Version 5.0, July 2013

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## 3.0 SAMPLE PRESERVATION & HOLDING TIME

| | |
|---|---|
| Preservation: | Store at >0.0 – 6.0ºC (unless only submitted for analyses which do not require refrigerated storage, such as ICP Metals) |
| Holding Time: | This preliminary sample preparation step must be performed in time to allow the preparation steps for the requested analyses to be performed within their respective holding times. |

## 4.0 SAFETY

Assume all samples received contain hazardous chemicals. Wear gloves, lab coats and safety glasses at all times when handling samples and use of ventilation assets ((open doors, turn on fans and blowers and use fume hood hoods as needed to protect yourself from exposure to sample constituents.

Soil samples containing as much as 2% of 2,4,6-TNT have been safely ground and composited, if the samples have been submitted from a firing range or are thought to contain unexploded residues care should be exercises during sample preparation, particularly grinding. Samples containing higher concentrations of unexploded ordinance compounds should not be ground in the mortar and pestle or in the ring and puck mill. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely grayish-white material.

## 5.0 QC REQUIREMENTS

***QSMv5.0, Module 2, Section 5.7.1:***
Sample handling procedures shall address laboratory practices for recording the presence of extraneous materials (e.g., rocks, twigs, vegetation) present in samples in the case of heterogeneous materials. To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± *0.01* g) 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.

## 6.0 INTERFERENCES & TROUBLE-SHOOTING

Any sticks, leaves, rocks, or other objects must be removed as they are not part of the extraction matrix and their presence will interfere with the sample being ground to pass through a 30-mesh sieve.

Water is also a potential interference as its presence decreases the extraction solvent's ability to retain target analytes. Any water layer should be decanted from the sample and the sample must be dry so that is can be ground properly.

## 7.0 EQUIPMENT

Mortar & Pestle
Ring & Puck Mill
Jaw Crusher
Sieves
Sample Pans and Drying Racks

## 8.0 PROCEDURE

### 8.1 Sub-sampling Solid Samples

8.1.1 Verify that the balance has been calibrated earlier in the day. If it has not, calibrate it before proceeding.

8.1.2 Label a pre-cleaned container with the C&T sample number.

8.1.3 Place the container on the balance and tare the balance.

8.1.4 In the appropriate analysis or Soil Aliquot benchbook, write the C&T sample number and bottle letter, and analysis.

8.1.5 Using a clean spatula, disposable spatula or equivalent tool, remove and discard the top ~1cm of sample.

*Note:* Discard any leaves, twigs, large stones, or other material that is clearly not intended for analysis and take a visually representative aliquot of each sample. Document your observations and actions (ie: "discarded leaves & twigs") in the prep log.

8.1.6 Using the same spatula, thoroughly homogenize the next several cm of sample

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then weigh out the mass needed for the requested analysis.

8.1.7 Record the weight of the subsample in the benchbook.

8.1.8 Clean the spatula or tool between samples using deionized water and a clean paper towel to ensure that no contamination is transferred between samples.

**8.2 Compositing Waste or Liquid-Solvent Samples**

Oily, waste, and solvent samples should be composited by weight, following the procedure for compositing solid samples. If DI water does not entirely clean the spatula, use methanol or acetone first, followed by a DI rinse.

**8.3 Compositing Solid Samples**

8.3.1 Verify that the balance has been calibrated earlier in the day. If it has not, calibrate it before proceeding.

8.3.2 Group the samples to be included in the composite and determine what size container will be needed to create more than enough of the composite for all of the analyses needed. Use a minimum of 1g of each sample being added to the composite, as each subsample should representative of the entire contents of each core or bottle.

8.3.3 Label a pre-cleaned container with the C&T sample number of the composite.

8.3.4 Place the container on the scale and tare the scale.

8.3.5 Using a clean spatula, disposable spatula or equivalent tool, remove and discard the top ~1cm from the first sample sleeve.

8.3.6 Discard any leaves, twigs, large stones, or other material that is clearly not intended to be included in the analysis and take a visually representative aliquot of each sample. Document your observations and actions (ie: “discarded leaves & twigs”) in the benchbook.

8.3.7 Using the same spatula, thoroughly homogenize the next several cm of sample, then weigh the necessary aliquot out of this homogenized fraction. Take at least 1 gram of sample for each component of the composite, ensuring each component of the composite is representative of the original sample and equally represented in the composite.

8.3.8 Clean the spatula or tool between samples using deionized water and a clean paper towel, to ensure that there is no contamination between the discrete samples.

8.3.9 Repeat Steps 8.3.5 – 8.3.8 for each of the remaining samples to be included in the composite, using exactly the same weight for each aliquot.

8.3.10 In the appropriate analysis or Soil Aliquot benchbook, write the C&T sample

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number of the composite, along with the sample numbers, bottle letters, and weight used from each of the discrete samples being included in the composite.

Example: 252689-001 comp -001 A-D, 15.0g of each
252014-001 comp -1A, -2A, -3A, 20.0g of each

*Note:* When using composites that have been previously prepared, write "premade comp", "xlab comp", etc. under the Comments/Observations heading.

## 9.0 DOCUMENTATION REQUIREMENTS

The Soil Aliquot or Prep benchbook must be completed each time samples are prepared. Record the following information:

- Date
- Initials of analyst
- Sample number, accompanied by the unique container identifier (A-> Z)
- Total Mass of subsample
- For composites, ID and mass of each aliquot included in the composite
- Observations concerning unusual sample appearance, odor, behavior

## 10.0 WASTE DISPOSAL

Dispose of excess sample in the laboratory soil sample waste stream unless the samples are imported under USDA quarantined from Foreign countries or/and US Protectorates, these materials must remain quarantined and incinerated before disposal.

## 11.0 POLLUTION PREVENTION

Please insure all sample and excess sample materials are disposed of in the appropriate waste stream.

Ask clients to submit only the minimum amounts of materials needed to execute this procedure, or better yet, recommend clients perform this procedure in the field.

## 12.0 REVISION HISTORY

The previous document (revision 5) was changed as follows>

- Cover page and numbering were reformatted. SOP reorganized to match C&T standard format.
- Section 2: References were updated
- Section 3: Added storage & holding time information
- Section 5: Updated to incorporate current regulatory and guidance language
- Section 8: In Section 8.1.6, replaced minimum subsample mass of 10g with instruction to weigh out the amount needed for the requested analysis.
- Section 9: Aligned documentation requirements with this procedure

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# Data Validation Checklist

| | | GC/MS - Level 2 | GC Level "2" Deliverables | General Chemistry Level "2" Deliverables | Trace Metals Level "2" Deliverables | Radiochemistry Level "2" Deliverables | GC/MS - Level 3 | GC Level "3" Deliverables | General Chemistry Level "3" Deliverables | Trace Metals Level "3" Deliverables | Radiochemistry Level "3" Deliverables | GC/MS - Level 4 | GC Level "4" Deliverables | General Chemistry Level "4" Deliverables | Trace Metals Level "4" Deliverables | Radiochemistry Level "4" Deliverables |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Chain of Custody | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 2 | Sample results with analysis and extraction/preparation dates | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 3 | Summary of MS/MSD/Duplicate recoveries and control limits (listing or link with associated samples) | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 4 | Summary of LCS/LCSD recoveries and control limits (listing or link with associated samples) | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 5 | Method blanks (listing or link with associated samples) | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 6 | Summary of surrogate recoveries | x | x | | | | x | x | | | | x | x | x | | |
| 7 | Summary of initial calibration data | | | | | | RRF and %RSD, or r if applicable | %RSD or r if applicable* | correlation coefficient, r | (% recovery - ICP) or (correlation coefficient, r - CVAA/GFAA) | | RRF and %RSD, or r if applicable | RRF and %RSD, r if applicable* | correlation coefficient, r | (% recovery - ICP) or (correlation coefficient, r - CVAA/GFAA) | |
| 8 | Summary of continuing calibration | | | | | | %D and RRF | %D | %D or % recovery | %D or % recovery | | %D and RRF | %D | %D or % recovery | %D or % recovery | |
| 9 | Summary of internal standards | | | | | | area response and retention time | | | x | | area response and retention time | | | x | |
| 10 | Summary of instrument tuning (listing or link with associated samples, must show 12 hour clock for GC/MS) | | | | | | x | | | x | | x | | | | |
| 11 | Injection logs | x | Injection logs or injection log summary | x | Injection logs or injection log summary | | x | Injection logs or injection log summary | x | Injection logs or injection log summary | | x | x | x | x | |
| 12 | Extraction/preparation logs | x | | | | | x | | x | x | | x | x | if applicable | x | |
| 13 | Case narrative to discuss anomalies | x | x | x | x | x | x | x | x | x | x | x | x | x | x | x |
| 14 | Summary of instrument blanks - metals only (listing or link with associated samples) | | | | | | | | | x | | x | | | x | |
| 15 | Summary of ICP interference check (listing or link with associated samples) | | | | | | | | | x | | | | | x | |
| 16 | Summary of ICP post digestion spike, and serial dilution results | | | | x | | | | | x | | | | | x | |
| 16 | Background checks with detectors and date performed identified | | | | | | | | | | x | | | | | x |
| 17 | Efficiency checks with detectors and date performed identified | | | | | | | | | | x | | | | | Efficiency checks with detectors, date performed, counts obtained, count time, NIST traceable standards (or equivalent), geometry (as applicable), calibration range in keV, and laboratory acceptance limits identified |
| 18 | Self absorption curve with detectors, mass used, and date performed identified (as applicable to analysis) | | | | | | | | | | x | | | | | x |
| 19 | Analysis logs or summary | | | | | x | | | | | x | | | | | x |
| 20 | Raw data associated with the summary forms listed above | | | | | | | | | | | x | x | x | x | x |
| 21 | Raw data for item #2 which includes chromatograms, log books, quantitation reports, and spectra. | | | | | | | | | | | x | x | x | x | x |

*OC Pesticides analysis also requires summary results for DDT/Endrin breakdown, RPD between primary and confirmation columns, GPC, and Florisil (if performed)

| | Inorganic | L2 | L3 | L4 |
|---|---|---|---|---|
| 1 | Holding times | | | |
| 2 | Method blanks (listing or link with associated samples) | | | |
| 3 | Surrogate recovery (if applicable) | | | |
| 4 | Matrix spike recovery | | | |
| 5 | Duplicate sample RPD | | | |
| 6 | Laboratory control sample recovery | | | |
| 7 | Field duplicate sample analysis RPD | | | |
| 8 | Overall assessment of data in the SDG | | | |
| 9 | Initial calibration | | | |
| 10 | Continuing calibration | | | |
| 11 | ICP interference check | | | |
| 12 | Serial dilution checks | | | |
| 13 | Internal standard performance (if applicable) | | | |
| 14 | Reporting limits | | | |
| 15 | Analyte identification | | | |
| 16 | Analyte quantitation and detection limits | | | |
| 17 | System performance | | | |

| | Organic | L2 | L3 | L4 |
|---|---|---|---|---|
| 1 | Holding times | | | |
| 2 | Method blanks (listing or link with associated samples) | | | |
| 3 | Surrogate recovery (if applicable) | | | |
| 4 | Matrix spike (and matrix spike duplicate if applicable) recovery | | | |
| 5 | Duplicate sample RPD | | | |
| 6 | Laboratory control sample recovery | | | |
| 7 | Field duplicate sample analysis RPD | | | |
| 8 | Overall assessment of data in the SDG | | | |
| 9 | Initial calibration | | | |
| 10 | Continuing calibration | | | |
| 11 | ICP interference check | | | |
| 12 | Serial dilution checks | | | |
| 13 | Internal standard performance (if applicable) | | | |
| 14 | Reporting limits | | | |
| 15 | Analyte identification | | | |
| 16 | Analyte quantitation and detection limits | | | |
| 17 | System performance | | | |
| 18 | Compound identification | | | |
| 19 | Compound quantitation and detection limits | | | |
| 20 | Tentatively identified compound verification (GC/MS) | | | |

| | | 1 | 2A | lev |
|---|---|---|---|---|
| 1 | Documentation identifies the laboratory receiving and conducting analyses, and includes documentation for all samples submitted by the project or requester for analyses. | | | |
| 2 | Requested analytical methods were performed and the analysis dates are present. | | | |
| 3 | Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result (and the uncertainty of each result and clear indication of the type of uncertainty reported if required, e.g., for radiochemical analyses). | | | |
| 4 | Requested target analyte result units are reported (along with their associated uncertainty units if required, e.g., for radiochemical analyses). | | | |
| 5 | Requested reporting limits for all samples are present and results at and below the requested (required) reporting limits are clearly identified (including sample detection limits if required). | | | |
| 6 | Sampling dates (including times if needed), date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH and temperature) are documented. | | | |
| 7 | For radiochemical analyses, the sample-specific critical values (sometimes called "critical level," "decision level" or "detection threshold") and sample specific minimum detectable value, activity or concentration for all samples are reported and results at and below the requested (required) critical values are clearly identified. | | | |
| 8 | For radiochemical analyses, the chemical yield (if applicable to the method) and reference date and time (especially for short lived isotopes) is reported for all samples (as appropriate). | | | |
| 9 | Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture) to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract. | | | |
| 10 | Requested methods (handling, preparation, cleanup, and analytical) are performed. | | | |
| 11 | Method dates (including dates, times and duration of analysis for radiation counting measurements and other methods, if needed) for handling (e.g., Toxicity Characteristic Leaching Procedure), preparation, cleanup and analysis are present, as appropriate. | | | |
| 12 | Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compounds (DMC) recoveries, laboratory control sample (LCS) recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials) are provided and linked to the reported field samples (including the field quality control samples such as trip and equipment blanks). | | | |
| 13 | Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes, post digestion spikes) have been added, as appropriate. | | | |
| 14 | Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated. | | | |
| 15 | Frequency of QC samples is checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch). | | | |
| 16 | Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract. | | | |
| 17 | Initial calibration data (e.g., initial calibration standards, initial calibration verification [ICV] standards, initial calibration blanks [ICBs]) are provided for all requested analytes and linked to field samples reported. For each initial calibration, the calibration type used is present along with the initial calibration equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the initial calibration curve are present, along with their associated percent recoveries, as appropriate (e.g., if required by the project, method, or contract). For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present. | | | |
| 18 | Appropriate number and concentration of initial calibration standards are present. | | | |
| 19 | Continuing calibration data (e.g., continuing calibration verification [CCV] standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present. | | | |
| 20 | Reported samples are bracketed by CCV standards and CCBs standards as appropriate. | | | |
| 21 | Method specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods, DDT/Endrin breakdown checks for pesticides and aroclors, instrument blanks and interference checks for ICP methods). | | | |
| 22 | Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatography-mass spectroscopy [GC-MS] tunes have been run every 12 hours). | | | |
| 23 | Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract. | | | |
| 24 | Instrument response data (e.g., GC peak areas, ICP corrected intensities) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, matrix spikes, matrix spike duplicates, LCS, and method blanks as well as calibration data and instrument QC checks (e.g., tunes, DDT/Endrin breakdowns, interelement correction factors, and Florisil cartridge checks). | | | |

| | | | | |
|---|---|---|---|---|
| 25 | Reported target analyte instrument responses are associated with appropriate internal standard analyte(s) for each (or selected) analyte(s) (for methods using internal standard for calibration). | | | |
| 26 | Fit and appropriateness of the initial calibration curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the initial calibration curve for each (or selected) analyte(s) from the instrument response. | | | |
| 27 | Comparison of instrument response to the minimum response requirements for each (or selected) analyte(s). | | | |
| 28 | Recalculation of each (or selected) opening and closing CCV (and CCB) response from the peak data reported for each (or selected) analyte(s) from the instrument response, as appropriate | | | |
| 29 | Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated initial calibration response for each (or selected) analyte(s). | | | |
| 30 | Recalculation of percent ratios for each (or selected) tune from the instrument response, as appropriate. | | | |
| 31 | Compliance check of recalculated percent ratio for each (or selected) tune from the instrument response. | | | |
| 32 | Recalculation of each (or selected) instrument performance check (e.g., DDT/Endrin breakdown for pesticide analysis, instrument blanks, interference checks) from the instrument response. | | | |
| 33 | Recalculation and compliance check of retention time windows (for chromatographic methods) for each (or selected) analyte(s) from the laboratory reported retention times. | | | |
| 34 | Recalculation of reported results for each reported (or selected) target analyte(s) from the instrument response. | | | |
| 35 | Recalculation of each (or selected) reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials etc.) from the instrument response. | | | |
| 36 | Each (or selected) sample result(s) and spike recovery(ies) are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract. | | | |
| 37 | All required instrument outputs (e.g., chromatograms, mass spectra, atomic emission spectra, instrument background corrections, and interference corrections) for evaluating sample and instrument performance are present. | | | |
| 38 | Sample results are evaluated by checking each (or selected) instrument output (e.g., chromatograms, mass spectra, atomic emission spectra data, instrument background corrections, interference corrections) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences). | | | |
| 39 | Each (or selected) instrument's output(s) is evaluated for confirmation of non-detected or tentatively identified analytes. | | | |

# APPENDIX B

Environmental Protection Plan

**FINAL**

**APPENDIX B**

# ENVIRONMENTAL PROTECTION PLAN

## Sediment Investigation and Bathymetric Survey for Parcel F

**Hunters Point Naval Shipyard**
**San Francisco, California**

**Contract Number: N62473-12-D-2004, CTO 0014**
**Document Control Number: INEC-2004-0014-0003**

**November 2017**

**Prepared for:**

**Naval Facilities Engineering Command Southwest**
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**Prepared by:**

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**Brea, CA 92821**

**FINAL**

# APPENDIX B

# ENVIRONMENTAL PROTECTION PLAN

## Sediment Investigation and Bathymetric Survey for Parcel F

## Hunters Point Naval Shipyard
## San Francisco, California

**Contract Number: N62473-12-D-2004, CTO 0014**
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**November 2017**

Reviewed and Approved by:

Mitra Fattahipour, PG
Project Manager

Date

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# ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| APP | Accident Prevention Plan |
| BMP | best management practice |
| CSO | Caretaker Site Office |
| ECC-Insight | ECC-Insight LLC |
| EPP | Environmental Protection Plan |
| HPNS | Hunters Point Naval Shipyard |
| IDW | investigation-derived waste |
| NPDES | National Pollutant Discharge Elimination System |
| OSHA | Occupational Safety and Health Administration |
| PEL | permissible exposure limit |
| ROICC | Resident Officer In Charge of Construction |
| SSHP | Site Safety and Health Plan |

# 1.0 INTRODUCTION

This Environmental Protection Plan (EPP) was prepared by ECC-Insight LLC (ECC-Insight) and describes project-specific information for environmental management related to planned sediment investigation and bathymetric survey activities at Parcel F, Hunters Point Naval Shipyard (HPNS) in San Francisco, California (Figures 1-1 and 1-2 of the Work Plan).

ECC-Insight has been contracted to perform this work for the Base Realignment and Closure Program Management Office West, United States Department of the Navy, Naval Facilities Engineering Command Southwest under Contract No. N62473-12-D-2004, Task Order 0014. The work under this task Order will be completed in compliance with the contract plans and specifications, and local, state and federal regulations.

The Work Plan provides a detailed scope of work, including the following:

- A bathymetric survey will be conducted within several subareas of Parcel F (Figure 3-1 of the Work Plan),
- The collection of grab sediment samples from the seafloor surface within six separate site areas where former Parcel B and C piers and wharf structures have been removed (ERS, 2012) (Figure 3-2 of the Work Plan), and
- The data will be used to fill identified data gaps in the Conceptual Site Model, address regulatory comments on the removal action at the former Parcel B and C piers and wharf structures, and refine the remedial alternatives for Parcel F in support of the Proposed Plan, Record of Decision and the Remedial Design.

This EPP provides specific information to provide adequate environmental protection of various endangered/protected species and plants that may inhabit HPNS, San Francisco, California during and following completion of field activities. Based on existing site conditions and location, the following environmental resources were identified as potentially requiring environmental protection and are addressed in this EPP:

- Land and Biological Resources – No disturbance of onshore soil will be conducted during this project,
- Water quality – Water quality must be maintained throughout the phases of the project by implementing proper best management practices,
- Air quality – No activities generating excess fugitive dust will conducted during this project, and
- Spill Prevention and Response – Provisions will be made to respond to spills that could adversely affect the environment.

The Project Manager and Site Superintendent as supported by the Quality Control Manager will be responsible for implementing and monitoring compliance with this EPP.

Field personnel working on the site, including subcontractor personnel, will be instructed on the contents of this EPP. Training will include instruction on detection and prevention of pollution on site, as well as procedures for responding to potential spills or pollution on site. Field personnel also will be instructed on the installation and maintenance of site control features.

## 2.0 LAND RESOURCE MANAGEMENT

Equipment and material storage areas will be indentified and approved by the HPNS Caretaker Site Office (CSO) and Resident Officer In Charge of Construction (ROICC) prior to initiating field activities. Project Team personnel will not use areas on the HPNS property other than those designated for field activities, unless specifically approved by the CSO/ROICC. Site vehicles will remain on designated access roads or hardscapes and will be prohibited from driving on land areas outside of the designated work zones.

# 3.0 WATER RESOURCES PROTECTION

The HPNS is surrounded by the San Francisco Bay (Figure 1-2 of the Work Plan). To reduce the potential for pollution due to field activities, best management practices (BMPs) will be used as described in the California Storm Water Best Management Practice Handbook (Construction Activity Handbook) prepared by the California Stormwater Quality Association (2003).

## 3.1 Storm Water Control

A general National Pollutant Discharge Elimination System (NPDES) permit is typically required for construction activities at sites with construction activity that disturb one acre or more of soil. Since sediment sampling activities will be conducted at discrete locations along the seafloor and the site is regulated under the Comprehensive Environmental Response, Compensation, and Liability Act, a general NPDES permit and site-specific Storm Water Pollution Prevention Plan is not required.

## 3.2 Erosion/Sediment Control

No disturbance of onshore soil or stockpiling will be conducted during this project. Investigation-derived waste (IDW) that is anticipated to be generated will be limited to a small volume of saturated sediment during sample collection and decontamination fluid. IDW will be stored in sealed 55-gallon drums with secondary containment, as appropriate, in accordance with the Waste Management Plan (Appendix C of the Work Plan).

## 3.3 Spill Control

Equipment will be stored in a designated location on site. Marine vessels and trucks will be maintained to prevent oil and/or fuel leaks. Measures will be taken to prevent chemicals, fuels, oils, greases, bituminous materials, and contaminated materials from entering into bay waters and site drainage swales. All vehicle fueling will be conducted off-site to eliminate on-site handling and storage of fuel.

Adsorbents will be made available on site to help clean up leaks or spills that might occur. Absorbent pads and booms will be stored on each survey vessel to collect any discharge onto the water surface. Soil and absorbent materials contaminated by an oil and/or fuel spill will be immediately removed and placed into appropriate containers. The containers will be labeled, dated, and staged for off-site transport and disposal.

## 4.0 WILDLIFE RESOURCES

Precautions will be taken during field activities to minimize disturbance of potential habitat areas and wildlife. Little to no vegetation exists at the project site or laydown area. However, prudent steps will be taken to protect trees and vegetation near the planned field activities. Mitigation measures will be taken to minimize the impact of field activities on existing vegetation, if necessary. Vehicle traffic will be limited to existing roadways and hardscapes.

ECC-Insight will take necessary precautions to minimize disturbances to wildlife and their habitat adjacent to the work areas once work at the site commences. If marine mammals are spotted during offshore activities, work in the vicinity of the wildlife will be postponed, to the extent practicable, until wildlife is a safe distance from the vessel. Safe distances from marine mammals will be maintained in accordance with the Marine Mammal Protection Act as follows: 150 feet for wild dolphins, porpoises, and seals; and 300 feet for whales (U.S. Marine Mammal Act, 1972).

## 5.0 DUST CONTROL/AIR POLLUTION

Planned field activities will not generate air emissions in the form of fugitive dust. All vehicle traffic will be limited to existing roadways and hardscapes. IDW that is anticipated to be generated will be limited to a small volume of saturated sediment during sample collection and decontamination fluid. Potential dust emissions will be minimized by utilizing BMPs in accordance with the Final Basewide Dust Control Plan, Revision 1 (Tetra Tech, 2010), as applicable.

## 6.0 NOISE CONTROL

Occupational Safety and Health Administration (OSHA) defines the permissible exposure limit (PEL) for noise exposure at 90 decibels for an 8-hour time-weighted average. Field activities are not anticipated to generate noise levels that approach the OSHA PEL. For guidance on hearing protection see the project-specific Accident Prevention Plan (APP) and the Site Safety and Health Plan (SSHP), provided under separate cover.

## 7.0 DECONTAMINATION AND CLEANUP

Decontamination procedures for equipment and personnel will be performed in accordance with the Work Plan and APP/SSHP prepared for this project (provided under separate cover). Decontamination of all non-disposable sampling equipment will be performed to prevent the introduction of extraneous materials into samples and to prevent potential cross-contamination between samples. Decontamination of non-disposable sampling equipment, including the Van Veen sampler, will consist of cleaning the equipment with a brush, using laboratory grade detergent (e.g., Liquinox) and a potable water solution; rinsing with potable water and rinsed again with deionized water. The equipment will be scrubbed and rinsed in three separate five-gallon buckets. All used decontamination materials will be containerized on-site pending analysis in accordance with the Waste Management Plan (Appendix C of the Work Plan).

All equipment, supplies, materials, and debris will be removed at the conclusion of site activities. Debris will be disposed at a local landfill; sanitary sewer; waste treatment, storage, and disposal facility; and/or approved recycling facility.

## 8.0 SITE SECURITY

A secured mobile storage container (e.g., Conex box) will be utilized for equipment staging and temporary waste storage. Warning and/or "No Trespassing" signs will be posted in both English and Spanish. Signs will be provided around contaminated areas as described in Title 22 California Code of Regulations (Section 66265 14). Access to the site will be limited to authorized personnel only and will be controlled at the site entrances and exits. The Site Superintendent will maintain a field log for signing personnel in and out and checking the log to ensure personnel have left the site at the end of each workday.

## 9.0 REFERENCES

California Code of Regulations Title 22.

California Stormwater Quality Association, 2003. *Stormwater Best Management Practice Handbook: Construction Activity Handbook.*

ERS Joint Venture (ERS). 2012. *Final Removal Action Completion Summary Report, Pier Radiological Surveys and Removal, Hunters Point Naval Shipyard, San Francisco, California.* August 2, 2012.

TetraTech, Inc. 2010. *Final Basewide Dust Control Plan, Revision 1, Hunters Point Naval Shipyard, San Francisco, California.* November 29.

U.S. Marine Mammal Protection Act (1972, amended 1994)

# APPENDIX C

Waste Management Plan

**FINAL**

# APPENDIX C

# WASTE MANAGEMENT PLAN

## Sediment Investigation and Bathymetric Survey for Parcel F

## Hunters Point Naval Shipyard
## San Francisco, California

**Contract Number: N62473-12-D-2004, CTO 0014**
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**FINAL**

**APPENDIX C**

# WASTE MANAGEMENT PLAN

## Sediment Investigation and Bathymetric Survey for Parcel F

**Hunters Point Naval Shipyard**
**San Francisco, California**

**Contract Number: N62473-12-D-2004, CTO 0014**
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**November 2017**

Reviewed and Approved by:

| | |
|---|---|
| Mitra Fattahipour, PG<br>Project Manager | Date |

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Table C-1 Waste Material Accumulation and Waste Characterization Methods

# ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| APP | Accident Prevention Plan |
| CCR | California Code of Regulations |
| CFR | Code of Federal Regulations |
| COC | contaminant of concern |
| CSO | Caretaker Site Office |
| ECC-Insight | ECC-Insight LLC |
| HPNS | Hunters Point Naval Shipyard |
| IDW | investigation-derived waste |
| Navy | United States Department of the Navy |
| PCB | polychlorinated biphenyls |
| PPE | Personnel Protective Equipment |
| RCRA | Resource Conservation and Recovery Act |
| SAP | Sampling and Analysis Plan |
| SSHP | Site Safety and Health Plan |
| STLC | Soluble Threshold Limit Concentration |
| TCLP | Toxicity Characteristic Leaching Procedure |
| T&D | transportation and disposal |
| U.S. DOT | United States Department of Transportation |
| U.S. EPA | United States Environmental Protection Agency |
| WET | Waste Extraction Test |
| WMP | Waste Management Plan |

# 1.0 INTRODUCTION AND REGULATIONS

This Waste Management Plan (WMP) was prepared by ECC-Insight LLC (ECC-Insight) and describes project-specific information for waste management, transportation, and disposal (T&D) associated with sediment sampling and bathymetric survey activities at Parcel F, Hunters Point Naval Shipyard (HPNS) in San Francisco, California (Figures 1-1 and 1-2 of the Work Plan).

ECC-Insight has been contracted to perform this work for the Base Realignment and Closure Program Management Office West, United States Department of the Navy (Navy), Naval Facilities Engineering Command Southwest under Contract No. N62473-12-D-2004, Task Order 0014. The work under this task order will be completed in compliance with the contract plans and specifications; and local, state and federal regulations.

## 1.1 Background

Parcel F comprises the offshore portion of HPNS and is a total of approximately 446 acres. This proposed investigation focuses on Parcel F offshore subareas: Areas III (Point Avisadero Area), IX (Oil Reclamation Area), X (South Basin Area), and area where six former Parcel B and C piers and wharf structures were removed in 2011 (ERS, 2012) (Figures 1-2 and 3-2 of the Work Plan).

During historical operations at HPNS, Berths 61 and 64 (Figure 3-2 of the Work Plan), Submarine Piers B and C, and Wharf No. 2 were used for shipbuilding, maintenance and repair. Operations included metal work and sandblasting. Contaminants of concern (COCs) sources to offshore sediments identified in Area III include stormwater discharge, a drainage tunnel that was used to rapidly drain water from Dry Docks 2 and 3, and surface runoff and groundwater discharge from Site IR-26 in Parcel B (BBL, 2005).

The primary COCs that may be encountered during field activities include polychlorinated biphenyls (PCBs), copper, lead and mercury. The highest reported concentrations of COCs in sediment located in the vicinity based on assessment activities are approximately 6,000 mg/kg for PCBs, 500 mg/kg for copper, 275 mg/kg for lead, and 252 mg/kg for mercury (Barajas, 2008; KCH, 2016).

## 1.2 Scope of Work

The Work Plan provides a detailed scope of work, including the following:

- A bathymetric survey will be conducted within several subareas of Parcel F (Figure 3-1 of the Work Plan),
- The collection of grab sediment samples from the seafloor surface within six separate site areas where Former Parcel B and C pier and wharf structures have been removed (ERS, 2012) (Figure 3-2 of the Work Plan), and

- The data will be used to fill identified data gaps in the Conceptual Site Model, address regulatory comments on the removal action at the former Parcel B and C piers and wharf structures, and refine the remedial alternatives for Parcel F in support of the Proposed Plan, Record of Decision and the Remedial Design.

This WMP presents hazardous and non-hazardous waste storage, handling, and characterization; and waste T&D from field activities at Parcel F. Implementation of this plan will be coordinated with the HPNS Caretaker Site Office (CSO) and Resident Officer In Charge of Construction.

# 2.0 WASTE STORAGE, HANDLING, AND CHARACTERIZATION

Investigation-derived waste (IDW) and other wastes (e.g., trash) will be generated during onsite field activities at Parcel F. Attempts will be made to minimize the wastes generated during field activities. Anticipated waste streams include solid and liquid wastes consisting of excess sediment from grab samples, disposable sampling equipment, used personal protective equipment (PPE), general refuse, and decontamination fluid.

The substantive requirements of regulations of the state and federal hazardous waste generation, characterization, treatment, and management of Title 22 California Code of Regulations (CCR) Sections 66261, 66262, 66264, and 66265 and 40 Code of Federal Regulations (CFR), Parts 260, 261, 262, 264, 265, and 268 are applicable to the management of waste generated during project activities. Field staff will follow the procedures described in this WMP to maintain compliance with the applicable state and federal regulations.

## 2.1 Types of Waste

The types of IDW that will be generated during field activities at Parcel F are:

- Excess sediment from sampling activities,
- Fluid from equipment decontamination,
- General refuse (such as sample packaging materials) generated during field activities,
- Miscellaneous material and other disposable sampling materials generated during field activities, and
- Used PPE.

IDW management will be performed as IDW is generated.

## 2.2 Waste Minimization

Waste generation will be minimized by planning activities to ensure efficient use of supplies, generating the minimal amount of sediment needed during sampling, and preventing the mixing of contaminated and clean materials. Uncontaminated materials (such as general refuse) will be recycled when possible.

The following waste minimization techniques will be observed during field activities to reduce the volume of waste generated:

- Do not place media of different hazard classifications together,
- Use drop cloths or other absorbent material to contain small spills or leaks,
- Use volume reduction techniques when practicable, and
- Verify that waste containers are solidly packed to minimize the number of containers.

## 2.3 Waste Handling and Storage

Wastes will be stored based upon the activity that generated the waste, composition of the waste, quantity of waste, and anticipated classification. General refuse will be placed in a general refuse dumpster. Cardboard and other easily recyclable material will be recycled offsite. Other wastes will be handled as described in this WMP.

Hazardous wastes are not anticipated to be encountered during project activities; however, if encountered, hazardous wastes will be stored in a hazardous waste accumulation area. The waste accumulation area will be coordinated with the CSO prior to the start of fieldwork. The hazardous waste accumulation area will be secured to prevent unauthorized access and will be labeled "Hazardous Waste Accumulation Area – Unauthorized Personnel Keep Out". The accumulation area will be kept clean and debris or obstructions to prevent access to containers or equipment. Adequate aisle space will be maintained so that personnel can access containers and read the labels on containers.

The Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP), provided under separate cover, contain emergency information for the Parcel F activities. This information includes the names and telephone numbers of emergency coordinators, telephone number of the fire department, and fire equipment and requirements for a spill kit to be maintained at Parcel F for use in case of a fire or spill. The following subsections describe the waste streams and the containers that will be used to store the waste streams.

### 2.3.1 Solid Waste

Excess sediment generated during sampling activities will be segregated based on the pier structure area from which it was collected. The sediment will be collected in sealable 5-gallon buckets on the survey vessels and be transported to a properly labeled 55-gallon drum(s) located in the designated waste accumulation area. Containerized sediments will be stored within a secure mobile storage container (e.g., Conex box) equipped with locking door to prevent unauthorized entry. Container lids will be closed and secured at the end of each workday. Drums and other storage containers will be labeled according to the procedures described in Section 2.6. Waste materials will be stored at the staging area, pending waste characterization.

### 2.3.2 Aqueous Wastes

Equipment decontamination fluids utilized on the survey vessels will be transported in sealable 5-gallon buckets to a 55-gallon drum (or polyethylene container) and stored with secondary containment. Waste will be stored adjacent to the project site or at a temporary storage area located at the site. Aqueous waste generated at the site is anticipated to be non-hazardous waste. Secondary containment will be monitored for the accumulation of precipitation or will be covered to prevent the accumulation of precipitation, if necessary. This will allow for monitoring of potential leaks from containers used to store wastes. Containerized waste will be stored within a

secure mobile storage container (e.g., Conex box) equipped with locking door to prevent unauthorized entry.

### 2.3.3 Personal Protective Equipment

Work at the Site is anticipated to be conducted in Level D PPE in accordance with the approved APP/SSHP (provided under separate cover). Used disposable PPE will be placed in properly labeled 55-gallon drums, or other suitable containers.

### 2.3.4 Empty Containers

Empty containers that are not used to transport waste offsite, but did contain wastes (excluding general refuse and recyclable materials) will be decontaminated before disposal. After decontamination, the containers will be inspected to ensure material has been removed from the container.

## 2.4 Training

Personnel who work with or handle hazardous waste or potentially hazardous wastes will have completed Occupational Safety and Health Administration 40-hour training. Training certificates and applicable refresher training documentation will be maintained onsite.

## 2.5 Inspection of Waste Storage Areas

IDW storage areas will inspected at a minimum weekly during field activities by the field team leader. Field logbooks will be used to record inspections and describe the following:

- Condition of containers; containers that are in poor condition or leaking will have the contents transferred to another container,
- Containers will be checked to make sure construction is intended for the contents that are stored in the container. Manufacturer descriptions of the containers will be reviewed before storing wastes in the containers. Manufacturers will be contacted if it cannot be determined if the container is appropriate for the waste,
- Containers will be checked to make sure the lids are closed except when adding or removing waste,
- Containers will be inspected to make sure they are labeled correctly per Section 2.6,
- Fire extinguishers will be inspected to verify they are operable, and
- The waste accumulation area has the correct signage posted.

## 2.6 Waste Labeling Procedures

Waste will be contained in 55-gallon steel drums or polyethylene storage containers. Containers will be labeled according to the determination of the class of waste at the time of generation.

Indelible ink will be used to label containers of potentially hazardous waste with a detailed description, including accumulation start date, and the words "INVESTIGATION-DERIVED WASTE/REMEDIAL WASTE SAMPLED – PENDING ANALYSIS." Container labels will be revised based upon waste characterization. IDW generated from sampling activities, decontamination of equipment, PPE, will be labeled as "Investigation-Derived Waste" and will list the Navy as the generator, location where the wastes were generated, description of contents, and date of generation. Each IDW container will be assigned a number. Logbook entries describing sampling and decontamination activities will include the container number that the wastes are stored. In addition to the waste descriptions in the logbook, a container inventory log will be maintained in the waste storage area. Analytical results from sampling activities will be used to characterize the wastes. If containers are determined to contain hazardous waste, the containers will immediately be labeled with a completed "Hazardous Waste" label that will include:

- United States Environmental Protection Agency (U.S. EPA) identification number of the generator,
- Name and address of the generator,
- U.S. EPA waste code,
- United States Department of Transportation (U.S. DOT) shipping name (prior to off-site shipment),
- Description of contents, and
- Date of generation.

In addition, weekly inspections of container storage areas will be conducted and logged while wastes remain in these areas to ensure the integrity of the containers and secondary containment, to check for leaks or spills, and to ensure that labels and markings are in good condition.

## 2.7 Waste Characterization

Characterization is necessary to determine if wastes must be managed as hazardous or nonhazardous wastes and if the wastes are subject to other laws and regulations. Waste characterization is required to determine the proper storage, treatment, and disposal options. Waste characterization sampling and analyses will be performed in accordance with the approved Sampling and Analysis Plan (SAP), provided as Appendix A in the Work Plan. Analytical criteria will depend on the requirements of the waste treatment, storage, and disposal facility.

Table C-1 presents the analyses anticipated for the waste materials generated during site activities. Actual waste characterization analyses will be performed based on waste treatment, storage, and disposal facility requirements. Additional sampling and analysis may be conducted using the Toxicity Characteristic Leaching Procedure (TCLP) or the Waste Extraction Test (WET) to meet waste treatment, storage, and disposal facility requirements. TCLP analysis is not required when

total concentrations are less than 20 times the TCLP regulatory level. WET analysis is not required when total concentrations are less than 10 times the Soluble Threshold Limit Concentration (STLC) regulatory level. Results from the analyses discussed above will be used to classify waste as Resource Conservation and Recovery Act (RCRA) hazardous waste, California hazardous waste, or non-hazardous waste. Solid waste excluded from the definition of hazardous waste under federal RCRA regulations (Title 40 of the CFR, Part 261.4[b]) may be regulated as hazardous waste under California regulations (CCR, Title 22, Section 66261.20). Soil will be classified as RCRA hazardous waste if concentrations exceed TCLP regulatory limits. Soil will be classified as California hazardous waste if concentrations exceed Total Threshold Limit Concentration or STLC regulatory limits based on WET results.

One soil sample will be collected from each drum of solid waste (such as excess sediment). This sample will be collected as a composite sample from two discrete locations within the drum or at another frequency acceptable to the waste treatment, storage, and disposal facility. One grab sample will be collected from each drum or container of liquid waste (such as decontamination fluid). The SAP (Appendix A of the Work Plan) provides additional details and procedures for collecting waste characterization samples.

# 3.0 HAZARDOUS AND NON-HAZARDOUS WASTE TRANSPORATION AND DISPOSAL

Transportation of hazardous and non-hazardous waste is regulated by 22 CCR Section 66262 and U.S. DOT requirements. Waste generated during this work at Parcel F is anticipated to be non-hazardous.

## 3.1 Documentation and Reporting

Hazardous and non-hazardous waste being shipped offsite will be accompanied by the appropriate bill of lading or hazardous waste manifest. The Navy's CSO will sign the bill of lading or hazardous waste manifest. The bill of lading or hazardous waste manifest will accompany the waste. When the solid waste material is transferred from the custody of the transporter to the designated disposal waste treatment, storage and disposal facility, the new party must sign the manifest and take custody of the waste material in accordance with RCRA, California, and U.S. DOT requirements. The waste treatment, storage, and disposal facility will mail copies of the bill of lading or hazardous waste manifest back to the Navy, who will provide copies to the project team. Copies of the bill of lading or hazardous waste manifest will be maintained in the project file.

Hazardous waste manifests will be completed in accordance with 22 CCR Sections 66262.2 through 66262.3 and 22 CCR Section 66268.7. The Manifest will use the Navy's U.S. EPA identification. Manifests and bills of lading will be acquired from disposal facilities for actual wastes hauled offsite.

## 3.2 Subcontractor Requirements

ECC-Insight will subcontract a waste loading, hauling, and disposal contractor to handle waste generated during site activities. The waste disposal contractor will supply labor, equipment, material, and trucks necessary to load, transport, and dispose of solid and aqueous waste materials from the Site. The waste disposal contractor will supply the appropriate means for loading the solid and aqueous waste materials into the transport vehicles. The waste disposal contractor will be responsible for ensuring sufficient spill containment and equipment is on site during the transfer process. The field team leader will verify the spill containment and equipment adequacy.

The waste disposal contractor will have appropriate licenses, insurance, medical certifications, permits, and registrations (including but not limited to a California Department of Toxic Substances Control hazardous waste transporter registration and a U.S. EPA identification number for hauling waste). The waste disposal contractor shall have a written U.S. DOT Hazardous Material Security Plan in effect, and personnel will have been trained in its requirements.

The waste disposal contractor workers, operators, and drivers will adhere to site health and safety requirements, as well as to the Site Health and Safety Plan. PPE including (at a minimum) hard

hats, steel-toed boots, safety glasses, and high-visibility vests with reflective stripes, are required during transportation and disposal activities.

## 3.3 Waste Transportation and Disposal

Waste profile forms and waste manifests will be provided to the CSO for approval and signature. Waste manifests may include (1) a Uniform Hazardous Waste Manifest if the waste is either a RCRA or California hazardous waste or (2) a bill of lading or non-hazardous waste manifest if the waste is a non-hazardous waste. Waste will be transported off site to an approved licensed waste treatment, storage, and disposal facility. The quantity of waste materials transported off site, signed certificates of disposal, and weight tickets will be obtained.

If analytical results indicate that wastewater meets discharge requirements, the wastewater will be released into an industrial sewer discharge point, determined by the Navy. A disposal permit may be obtained from the San Francisco Public Utilities Commission before discharging wastewater to the sewer system. If analytical results do not meet wastewater discharge requirements, the wastewater will be transported off site to an approved licensed waste treatment, storage, and disposal facility.

General refuse (such as packaging materials) typically is managed as non-hazardous waste and transferred to an on-site industrial dumpster, and contents are routinely disposed of in a municipal landfill. PPE will also be managed as non-hazardous solid waste and disposed of in a municipal landfill. Waste materials will be recycled whenever possible.

In accordance with requirements at 40 CFR 262.34, waste identified as hazardous will not be stored on site for more than 90 days after its generation.

## 4.0 REFERENCES

Barajas and Associates (Barajas), 2008. *Final Feasibility Study for Parcel F, Hunters Point Shipyard, San Francisco, CA.* April 30, 2008.

Battelle, Blasland, Bouck & Lee, Inc. (BBL), and Neptune & Company. 2005. *Final Hunters Point Shipyard Parcel F, Validation Study Report, San Francisco Bay, California.* May 2, 2005.

CH2M HILL Kleinfelder, JV (KCH) 2016. *Final Addendum to the Feasibility Study Report for Parcel F, Hunters Point Naval Shipyard, San Francisco, California.* January 2016.

ERS Joint Venture (ERS). 2012. *Final Removal Action Completion Summary Report, Pier Radiological Surveys and Removal, Hunters Point Naval Shipyard, San Francisco, California.* August 2, 2012.

Title 40 Code of Federal Regulations, Parts 260, 261, 262, 264, 265, and 268.

Title 22 California Code of Regulations, Sections 66261, 66262, 66264, and 66265.

United States Marine Mammal Protection Act (1972, amended 1994)

# TABLES

## TABLE C-1: WASTE MATERIAL ACCUMULATION AND WASTE CHARACTERIZATION METHODS

| Waste | Accumulation Method | Waste Characterization Method |
|---|---|---|
| **Solid Wastes** | | |
| Excess sediment from sampling activities | 55-gallon drum or smaller U.S. DOT-approved container | HPNS Parcel F COCs: PCBs, copper, lead, and mercury and additional analyses, as required by the waste treatment, storage, and disposal facility |
| Discarded PPE | Plastic bag | Visual, based on appearance (staining) |
| Disposable sampling equipment | Plastic bag | Visual, based on appearance (staining) |
| **Liquid Wastes** | | |
| Decontamination fluids | 55-gallon drum or smaller U.S. DOT-approved container | HPNS Parcel F COCs: PCBs, copper, lead, and mercury and additional analyses, as required by the waste treatment, storage, and disposal facility |
| Miscellaneous fluids from spills of gasoline, hydraulic fluids, or cleaning solutions | 55-gallon drum or smaller U.S. DOT-approved container | HPNS Parcel F COCs: PCBs, copper, lead, and mercury and additional analyses, as required by the waste treatment, storage, and disposal facility |

Notes:
COC contaminant of concern
HPNS Hunters Point Naval Shipyard
PCB polychlorinated biphenyl
PPE personal protective equipment
U.S. DOT United States Department of Transportation

# APPENDIX D

# RESPONSE TO COMMENTS

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

*The following comments were received from Ms. Judy C. Huang, Remedial Project Manager at United States Environmental Protection Agency-Region IX on August 28, 2017:*

| Comment | | Response |
|---|---|---|
| | **General Comments:** | |
| 1. | Since this is a data gap study, it is unclear why the work plan:<br>a. limited the analysis to the three constituents of concerns (COCs) in the Parcel F Record of Decision (copper, mercury, and PCBs) instead of using the entire list of metals commonly found at Hunters Point Naval Shipyard and PCBs.<br>b. only proposed to collect surface sediment instead of investigating the contamination to depth. Please provide a basis why only surface sediment investigation is proposed. | *a) The focus of this investigation is to address COCs identified in the Feasibility Study (FS; Barajas, 2008), in the area of the former Piers, to support the Proposed Plan, ROD and remedial design for Parcel F. As documented in the Parcel F FS (Barajas, 2008) and FS Addendum (KCH, 2017), Parcel F sediments have been adequately characterized through numerous investigations including the 2005 Validation Study (Battelle, BBL, and Neptune & Company, 2005), which established the Parcel F COCs as copper, lead, mercury, and PCBs. These four COCs were identified as the site-specific risk drivers determined by human health and ecological risk assessment. A Technical Memorandum: Optimized Remedial Alternative for Parcel F (ECC-Insight and CDM Smith, 2017) was recently prepared that documents the establishment of the Parcel F COCs and their respective PRGs.*<br><br>*Lead has been added to the list of analyses in Section 3.2 as a bullet, second paragraph, (see below) as well as per the respective edits in the SAP and as documented in the response to U.S. EPA General Comment 2:*<br><br>• *"**Lead by EPA Method 6020**"*<br><br>*b) Surface sediment samples are appropriate to determine if any COCs identified in the FS are present in the area beneath the former Piers and wharf structure. Surface sediment chemistry was used to conduct the human health and ecological risk assessments as well as develop preliminary remediation goals (PRGs). If the results of this surface sediment investigation identify COCs requiring remedial action to meet HPNS Parcel F Remedial Action Objectives (RAOs), additional sediment* |

| Comment | | Response |
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| | | *samples will be collected with depth as part of the pre-design investigation following the remedial design. The following has been added to Section 4.4 of the Work Plan:*<br><br>*"Surface sediment samples will be collected from approximately the upper five centimeters of the sediment surface. Depth (vertical elevation) of samples will be calculated from the survey data. If the results of the surface sediment sampling indicate that subsurface sediment samples are required, additional sampling will be conducted as part of the pre-design investigation in support of the remedial design."* |
| 2. | In each area, the Workplan proposes to collect numbers of subsamples to be composite into one. Please provide the rational for the number of sub samples indicated for each area and why a composite sample is adequate. | *The sediment screening and testing guidelines documented in Table 7 of the Water Board (2000), 'Draft Staff Report, Beneficial Reuse of Dredged Materials Sediment Screening and Testing Guidelines', was used as a basis for selecting the number of composite samples for each test sample. This guidance identifies that four subsamples be collected for each composite test sample. In review of the number of subsamples, additional test samples were included for former Piers B, C, and Wharf #2 per updated Figure 3-2. The following revisions were made to the text in Section 3.2, with changes in bold and italics. Additional changes were made to the SAP Worksheets (WSs) to reflect addition of lead (Pb) and increase in total number of samples per the wording below:*<br><br>*"3.2 Sediment Sampling*<br><br>*Surface sediment sampling will be conducted for analysis of Parcel F COCs in the area beneath the six former Parcel B and C pier and wharf structures that have been removed (ERS, 2012; Figure 3-2). Sediment samples will be collected from each of the following six (6) former structures: Berth 64, Berth 61, Pier B location, Pier C location, wooden Quay Wall, and Wharf #2 area (Figure 3-2). The proposed sampling density and approach* ***follows the sediment screening and testing guidelines documented in Table 7 of the Water Board (2000), 'Draft Staff Report, Beneficial Reuse of Dredged Materials Sediment Screening and Testing Guidelines'. Although the*** |

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| | | ***purpose of the sampling described in this work plan is not specifically for sediment beneficial reuse, the referenced guidance does provide a basis for the determination of total number of samples to be collected and use of composite sampling of sediment in San Francisco Bay.*** *These procedures recommend the collection of one composite sediment sample, consisting of four individual samples, for sediment volumes ranging between 5,000 cubic yards (135,000 cubic feet) and 20,000 cubic yards (540,000 cubic feet) (Water Board, 2000). Because we are collecting surface sediment samples* ***from the upper five centimeters of sediment surface,*** *it is proposed that one composite sample be collected* ***for Former Berth 64 and 61 and Quay Wall Areas****, with a minimum of four individual sub-samples collected per composite sample.* ***To ensure a denser coverage in the larger areas, 8 individual sub-samples will be composited into two composite samples (four sub-samples per composite sample) for Former Pier B, Pier C, and Wharf #2 Areas.*** *Individual sub-samples will be collected in the field. The sub-samples will be composited for each former pier area by the laboratory. Proposed individual surface sediment sample point locations for each pier area are depicted on Figure 3-2. A total of* ***36*** *individual sub-samples will be collected in the field. The individual surface sediment sub-samples will be composited by the laboratory to create* ***9*** *surface composite sediment samples from each of the former pier areas (SAP Worksheet #14) as follows:*<br>• *Former Berth 64 Area (approx. 16,000 square feet [sf]): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for copper, mercury,* ***lead****, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,*<br>• *Former Berth 61 Area (approx. 6,000 sf): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the* |

| Comment | | Response |
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| | | *composite sample for copper, mercury, **lead**, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,*<br>• *Former Pier B Area (approx. 40,000 sf): **Eight (8)**-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare two composite surface sediment sample**s** and analyze the composite samples for copper, mercury, **lead**, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,*<br>• *Former Pier C Area (approx. 40,000 sf): **Eight (8)**-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare **two composite** surface sediment sample**s** and analyze the composite samples for copper, mercury, **lead**, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory,*<br>• *Former Quay Wall Area (approx. 15,000 sf): Four (4)-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare one composite surface sediment sample and analyze the composite sample for copper, mercury, **lead**, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory, and*<br>• *Former Wharf #2 Area (approx. 24,000 sf): **Eight (8)**-individual surface sediment sub-samples will be collected in the field. The laboratory will prepare **two** composite surface sediment sample**s** and analyze the composite samples for copper, mercury, **lead**, and PCBs. The remaining individual sub-samples will be retained on hold by the laboratory."* |

**Response to Comments on**
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**Sediment Investigation and Bathymetric Survey for Parcel F**
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| Comment | | Response |
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| 3. | The Sampling and Analysis Plan (SAP) in Appendix A presents inconsistent information for the collection of field duplicates. Worksheet #12 states that field duplicates will not be collected because sediment sampling is heterogeneous and also references the nature of the composite sampling. However, duplicate samples of both the discrete and the composite samples should be collected to document the observed heterogeneity. Further, Worksheet #37 states that field sampling precision will be evaluated through the analysis of field duplicates, which will be collected at a frequency of 10 percent. It is unclear how field sampling precision will be assessed if field duplicates are not collected. Please revise the SAP to consistently include the analysis of field duplicates. | *Field duplicates have been added to SAP WS#12.* |
| 4. | SAP Worksheet #20 indicates a field blank will be collected, but Worksheet #12 does not include field blanks. Please revise the SAP to clarify if a field blank will be collected and if so, provide the measurement performance criteria in Worksheet #12. | *Field blanks have been added to SAP WS#12.* |
| 5. | SAP Attachment Al-2, Laboratory Standard Operating Procedures (SOPs), does not include the SOP for mercury analyses by the latest version of Method 7471 as identified in Worksheet #23. 1n addition, Worksheet #23 does not list the metals solid sample digestion SOP (MET 2.4) and subsampling and compositing SOP (CS 2.3) provided in Attachment Al-2. Please revise Attachment Al-2 and Worksheet #23 to consistently provide and identify the laboratory SOPs to be used for this project. | *SAP Attachment A1-2 has been updated with the SOPs identified. SAP WS #23 has been updated accordingly.* |

| **Comment** | | **Response** |
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| 6. | The data management information in the SAP does not include sufficient information regarding the procedures that will be used to document and archive the project records and electronic data. For example, it is unclear what information will be included in the laboratory reports and data validation reports (DVRs) identified in Worksheets #29 and #34-36. In addition, it is unclear where hard copy project documents and electronic files will be archived and the length of time that these files will be archived before disposal. Please revise the SAP to specify the information that will be included in the laboratory reports (e.g., quality control [QC] results, raw data, etc.) and DVRs (e.g., the QC parameters evaluated, the acceptance criteria used to evaluate each QC parameter, a list of all QC exceedances as well as the extent of the exceedance, the samples associated with each exceedance, and the qualifiers applied). Please also include the procedures for archiving all project records and files. | *SAP WS#29 has been updated with the contents of Level III and IV data packages as follows:*<br>*"Level III data packages will include sample logins, COCs, analytical results (including LOQ/LOD/DL), results of QC data specified in WS#'s 12, 24, 25, and 28.*<br>*Level IV data packages will include all Level III data as well as raw instrument data, sample weight forms, and chromatograms (where applicable) for specified samples to allow recalculation of final results."*<br><br>*SAP WS#14, "Data Management," has been updated as follows:*<br>*"PDF's of laboratory SDGs, data validation packages, and EDDs will be stored on an ECC-Insight server for the duration of the project +7 years. Physical copies of laboratory SDGs and data validation packages will be stored onsite at ECC-Insight for the duration of the project +7 years. In addition, all reports and data packages are archived by NAVFAC SW Administrative Records, in accordance with Navy policy and guidance. "* |
| 7. | Insufficient information is provided for the data validation to be performed on the results. Worksheet #11 indicates that 80 percent of the results will be validated at Level 3 and 20 percent of the results will be validated at Level 4, but the SAP does not specify what will be included in each level of validation. In addition, Worksheet #11 indicates data validation will be performed using EWI No. 1, the SAP, EPA National Functional Guidelines (NFG) for Superfund Organic Methods Data Review and for Inorganic Superfund Data Review, and the Guidance for Labeling Externally Validated Laboratory Analytical Data for | *The third party data validator, LDC, validation checklist has been added to the laboratory SOPs as an attachment.*<br>*SAP WS#34-36, "Validation Packages" row, has been updated as follows:*<br>*"All validation packages will include a review* ***<u>and discussion</u>*** *of all QC Parameters indicated in SAP WS#12, #24, and #28."*<br>*SAP WS#'s 11, Section 6, paragraph 6, & 34-36, "Laboratory data packages" row, have been updated to be consistent and reference EWI #1, the SAP, DoD QSM, the NFGs, and the EPA labelling guidance.* |

| Comment | | Response |
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| | Superfund Use. However, Worksheets #34-#36 indicate the SAP and the Department of Defense Quality Systems Manual (DoD QSM), Version 5.1 will be used. The SAP should clearly define the specific procedures that will be used to apply qualifiers for QC exceedances (e.g., when results will be qualified estimated/rejected and if individual or all samples in a batch will be qualified). These procedures can be defined in tables or validation checklists. Please revise the SAP to specify what is included in Levels 3 and 4 validation and to provide data validation procedures for each method. | |
| 8. | Note 1 in Worksheet #18 states that every fifth sample will be validated at Level 4, but only six composite samples are proposed to be collected. Therefore, it is unclear if only the fifth sample (i.e., 16 percent of the samples) will be validated at Level 4, or if results for two samples will be validated at this level (i.e., 33 percent of the samples) to meet the 20 percent requirement indicated elsewhere in the SAP. Please clarify the number of samples to be validated at Level 4. | *Note 1 in SAP WS #18 has been updated to the following with changes in bold:*<br><br>*"**A minimum of 20% of** sample**s** will be validated to level IV and marked on the chain-of-custody,"* |
| | **Specific Comments:** | |
| 1. | **Section 2.5, Current Conceptual Site Model, Page 2-2:** This section referred to the conceptual site model figures in the Feasibility Study and the Feasibility Study addendum. Please include these conceptual site model figures in the report. | *The Conceptual Site Model Figures have been replicated from the FSDGs Investigation Technical Memorandum and included as Figures 2-2 and 2-3 to the Work Plan and SAP. Hence, the first sentence of Section 2.5 has been updated as follows, with revisions in bold:*<br><br>*"A current CSM for Parcel F sediments is provided in detail in the FS (Barajas, 2008) and **the FSDGs Investigation Technical Memorandum (Figures 2-2 and 2-3; Barajas and Associates, 2007)**."* |

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| Comment | | Response |
|---|---|---|
| 2. | **Section 2.5.2, Contaminant Extent and Distribution in Area III, Page 2-4:** According to the third paragraph of Section 2.5.2, lead exceeded ambient threshold levels in sediment, but the Work Plan does not include analysis of lead for the proposed sediment samples. Lead was a constituent of paint, so spent sandblast grit that was discharged to San Francisco Bay could have high concentrations of lead. Please revise the Work Plan to include analysis of lead in the proposed sediment samples. Alternatively, please revise the Work Plan to explain why samples will not be analyzed for lead. | *Lead has been added to all sediment sampling. Please see revised text outlined in response to U.S. EPA General Comment 1 and 2. Lead has been added to respective worksheets in the SAP.* |
| 3. | **Section 3.1, Bathymetric Surveys, Page 3-1:** The last sentence on page 3-1 states that water surface elevations "will be checked against a tide board or real-time tide gauge," but the Work Plan provides no criteria for deciding which source to use for this check. Please revise Section 3.1 to include criteria for determining whether a tide board or real- time tide gauge will be used to check against water surface elevations. | *The last sentence in the second paragraph of Section 3.1 of the Work Plan, second paragraph, and Worksheet #17, "Bathymetric Survey," of the SAP has been revised as follows:*<br><br>*"Differential corrections for the vessel position will be conducted against the GPS survey control points identified in Figure 1-2."* |
| 4. | **Section 3.1, Bathymetric Surveys, Page 3-1 and Appendix A, Worksheet #17, Sampling Design and Rationale, Page 36 of 70:** Section 3.1 and Worksheet #17 indicate that survey lines will be spaced approximately 100 feet apart, but the text does not discuss whether this spacing will provide complete coverage. Survey line spacing should be selected to provide complete survey coverage, depending on the equipment used and the water depth. Please revise the Work Plan to discuss how the survey line spacing was selected and to ensure spacing is selected to provide complete coverage. | *The 100-foot spacing is appropriate for the technology and depth of water. The same vendor, Fugro, completed the 2003 bathymetric survey. Fugro is planning on using the same transverse lines for the 100-foot spacing as was used in 2003 to allow for the most complete survey comparison.*<br><br>*The following text has been added after the fifth sentence, first paragraph of Section 3.1 of the Work Plan and Worksheet #17, "Bathymetric Survey," of the SAP to discuss the rationale for selecting the line spacing:*<br><br>*"Since the depth to water in Area IX/X is less than 6 feet (Battelle, BBL, and Neptune & Company, 2005), the 100-foot spacing in this shallow area will provide sufficient resolution with the single beam survey. For the deeper waters beneath the former pier and wharf structures and Area III, the multi-* |

| Comment | | Response |
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| | | *beam survey provides greater resolution (see Section 3.1.1) that will identify change in bathymetry between the 100-foot lines. The 100-foot spacing was utilized in the previous 2003 bathymetric survey which will provide a direct comparison."* |
| 5. | **Section 3.1.2, Single-Beam Survey System, Page 3-2 and Appendix A, Worksheet #17, Sampling Design and Rationale, Page 36 of 70:** Section 3.1.2 and Worksheet #17 indicate that the single-beam echo sounder used for the bathymetric survey in Area I X/JX collects data "below the vessel;" however, the deeper the water the wider the mapped area will be. The South Basin (Area IX/X) is very shallow, so it is unclear how wide a strip of sediment will be mapped using this procedure. For example, if the single-beam echo sounder only maps a strip 20 feet wide in shallow water, then the 100-foot spacing for the survey lines is too large to provide complete coverage. Please revise the Work Plan to clarify the size of the strip of sediment that will be mapped in Area IX/X using the single-beam echo sounder. Please also discuss whether the 100-foot survey spacing is appropriate given the shallow depth of South Basin. | *Please see response to U.S. EPA Specific Comment #4.*<br><br>*The single-beam survey in Area IX/X is necessary due to the shallow water depths in this area, where a multi-beam survey is not feasible due to loss of efficiency. Also please note that the variation in the topography in the shallow area of Area IX/X is very small (estimated 2-6 feet) (Battelle, BBL, and Neptune & Company, 2005).*<br><br>*The multi-beam survey in the deeper water of Area III and below the former piers is of high quality with a swath width of approximately 4 times the water depth below the multi-beam sensor, which provides real-time coverage, and will provide higher resolution. The single beam sensor measures directly below the transducer below the vessel.*<br><br>*The following sentence was added before the last sentence of Section 3.1.1,"Multi-Beam Survey System" and in SAP WS #14:*<br><br>*"The multi-beam swath width is approximately 4 times the water depth below the multi-beam sensor, providing detailed resolution in the deeper areas."* |
| 6. | **Section 3.2, Sediment Sampling, Page 3-3:** This section states that "[t]he proposed sampling density and approach exceeds the procedures developed by the San Francisco Bay Regional Water Quality Control Board (Water Board, 2000) for testing of dredged materials for determining the suitability sediments for beneficial reuse." Please clarify why the Water Board's guidance for sediment beneficial reuse is relevant and applicable to sediment investigation at Hunters Point Shipyard. | *Although the referenced document describes sampling and testing guidelines for beneficial reuse of dredged materials, it does provide specific composite sampling and testing guidelines that are useful for comparison of sediment sampling density in the area. Please see revisions to the text in Section 3.2 identified in response to U.S. EPA General Comment 2.* |

| Comment | | Response |
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| 7. | **Section 3.2, Sediment Sampling, Page 3-4 and Appendix A, Worksheet #19, Field Sampling Requirements, Page 39 of 70:** Section 3.2 specifies Method 6010 for analysis of copper and Method 7471 for analysis of mercury, while Worksheet #19 specifies Method 6020 for analysis of copper and Method 7471A for analysis of mercury. Please revise the Work Plan to consistently identify the methods for each analyte. | *Referenced Section 3.2 of the Work Plan has been updated with EPA Method 6020 for Copper and lead and EPA Method 7471A for Mercury.* |
| 8. | **Section 3.2, Sediment Sampling, Page 3-5; Appendix A, Worksheet #11, Project Quality Objectives/systematic Planning Process Statements, Page 23 of 70; and Appendix A, Worksheet #17, Sampling Design and Rationale, Pages 36 of 70 and 37 of 70:** The second to last paragraph of Section 3.5 states that "If composite sediment · sample COC [contaminant of concern] concentrations from any former pier area exceed the PRGs [preliminary remediation goals], then individual sub-samples placed on hold in the laboratory for that area, will be analyzed for the Area III COCs;" however, the text does not clearly indicate whether some or all of the individual sub-samples for a given area will be analyzed. If some will be analyzed, then the text should explain how the individual sub-samples will be selected for analysis. 1n addition, the text does not specify whether individual sub-samples will be analyzed for the same constituents as the composite samples or only those constituents exceeding PRGs (e.g., just metals or just polychlorinated biphenyls [PCBs], etc.). Similarly, Worksheets #11 and #17 indicate that individual sub-samples will be analyzed if COC concentrations exceed PRGs, but does not clearly indicate whether some or all of the individual sub-samples for a given area will be analyzed and does not specify | *The referenced sentence in Section 3.2 has been modified as follows:*<br><br>*"If composite sediment sample COC concentrations from any former pier area exceed the PRGs, then **all** individual sub-samples placed on hold in the laboratory for that area, will be analyzed for the Area III COCs **(PCBs, copper, mercury, and lead)** to further refine any area of contamination below the former piers."*<br><br>*In addition, SAP WS#11, Item 3, last bullet of Table 11-1 has been updated as follows:*<br><br>*"If laboratory composited sediment samples exceed Project Action Levels (PALs), **all** individual sub-samples that make up the composite will be analyzed for the **same site** COCs **as the composite**."*<br><br>*SAP WS#17, "Sediment Sampling," has been updated as follows:*<br><br>*"In the event that composite sample analytical results exceed the PALs established, **all** the individual sub-samples **that make up that composite** will be analyzed **for all** the same COCs (Cu, Hg, **Pb**, and PCBs)."*<br><br>*The following has been added as the last sentence of SAP WS#17:*<br><br>*"Laboratory analysis will be performed on a 10 business day turnaround time (TAT) for preliminary data, which will allow for ample time to review the data for PAL exceedances that would require subsample analysis, per the hold times listed in WS#19."* |

| Comment | | Response |
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| | the constituents that will be analyzed (i.e., all of the COCs or only the one with the exceedence). In addition, the Work Plan should specify a timeline to ensure that individual sub-samples are analyzed within holding times. For example, the holding time for analysis of mercury is 28 days, so if individual sub-samples are analyzed for mercury, subsample analysis must occur within 28 days from sample collection. Please revise the Work Plan to clarify whether some or all of the individual sub-samples for a given area will be analyzed if PRGs are exceeded. Please also revise the Work Plan to clarify whether individual sub-samples will be analyzed for the same constituents as the composite·samples or only those constitue1its exceeding PRGs. Please also revise the Work Plan to specify the timeline for analysis of individual sub-samples to ensure analysis occurs within applicable holding times. | |
| 9. | **Appendix A, Worksheet #11, Project Objectives/Systematic Planning Process Statements, Table 11-1, Information Inputs, Page 21 of 70:** Table 11-1 includes bathymetric surveys for Area III and Area IX/X, but the area adjacent to the former piers as depicted on Figure 3-1 is not listed. Please revise Table 11-1 to include the bathymetric survey in the area adjacent to the former piers. | *SAP WS #11, Table 11-1, "Bathymetric Survey" row, has been updated with the following bullet point:*<br>• *"Survey area adjacent to former piers and former wharf, and"* |

| | | |
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| 10. | **Appendix A, Worksheet #11, Project Quality Objectives/Systematic Planning Process Statements, Pages 21of 70 through 24 of 70:** The data quality objectives (DQOs) should include additional detail for both the bathymetric survey and the sediment sampling. For example, Steps 1 and 2 do not include the bathymetric survey, but Worksheet #10 indicates a study goal is to determine whether erosional and bathymetric changes have occurred within Parcel F. In addition, Step 4 indicates the lateral boundaries of the study are the survey and sampling areas, but it is unclear how these areas were determined. Please revise the DQOs to include the bathymetric survey in each step and to clarify how the proposed sampling and survey plans were determined to meet these project goals. | *SAP WS#11 DQOs have been updated to add the bathymetric survey to Steps 1, 2, and 5.*<br><br>*The following changes have been made to Step 1 of the DQOs (new text bolded):*<br><br>***"1. State the Problem***<br><br>*Sediment sampling will be performed at the locations of several former piers* ***and a wharf structure*** *at Parcel F in order to evaluate if COCs are present in the footprint of the former piers* ***or former wharf. A bathymetric survey will be conducted in the area of the former piers and wharf structure in order to determine if bathymetric changes have occurred since removal of the piers and wharf structure. Current bathymetry surveys of Areas III, IX, and X are also required to accurately estimate the areas and volumes of sediments requiring remediation, to identify the placement of fill and capping materials during implementation of the selected alternative, and to estimate sediment erosion/deposition since the bathymetric survey in 2003. The bathymetric survey will also be used to determine whether the installation of cofferdams is a viable option in Areas IX/X.***<br><br>*The following sentences have been added to Step 2 of the DQOs:*<br><br>• *"Have erosion or bathymetric changes occurred in and around the former piers, wharf, Area III, Area IX, or Area X?*<br><br>• *Are cofferdams a viable option in Areas IX/X during remediation?"*<br><br>*The following sentence have been added to Step 4 of the DQOs:*<br><br>*"The proposed lateral boundaries for Areas III, IX, and X were defined in the Final FS Report for Parcel F (Barajas, 2008)."* |

| Comment | | Response |
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| 10. | **Continued** | *The following sentences have been added to Step 5 of the DQOs:*<br>• *"If bathymetric survey results are conducive to cofferdams for Areas IX/X, then cofferdams may be utilized during remediation in these areas per specifications identified during the remedial design.*<br>• *If the bathymetric survey shows no erosion or bathymetric changes, then no further action will be necessary, and*<br>• *If the bathymetric survey shows significant bathymetric changes, then the results will be reviewed and reported to the Navy with input from the BCT to determine if any further action is needed."*<br>*Sediment and sampling rationale is already described in the DQOs.* |
| 11. | **Appendix A, Worksheet #16, Project Schedule/Timeline Table, Page 35 of 70:** The schedule should include entries for analyzing the samples and validating the results, as well as reanalysis of the discrete samples if project action limits (PALs) for the composite samples are exceeded. This will ensure there is sufficient time to complete the reanalysis of the discrete samples within the sample holding times (e.g., 28 days for mercury). Please revise the schedule to include the analysis and validation timeframes, and ensure there is sufficient time for the reanalysis of the discrete samples if the composite samples exceed PALs. | *The following detail has been added to the Project Schedule in SAP WS #16 and Work Plan Table 5-1 (Section 5):*<br>- *"Mobilize field crew and equipment - November 27, 2017*<br>- *Collect composite samples of former piers and wharf structure (10 day TAT) - November 27-December 13, 2017*<br>- *Request additional sampling of individual sub samples pending results of composite sampling above (as needed) –December 7 -23, 2017*<br>- *Complete Bathymetric Survey – December 7, 2017*<br>- *Laboratory Validation Complete - January 31, 2018"*<br><br>*In addition, the following note has been added:*<br>*"Note: Schedule and duration may change pending subcontractor availability, weather conditions, and field logistics as well as regulatory and Navy approvals."* |

| Comment | | Response |
|---|---|---|
| 12. | **Appendix A, Worksheet #17, Sampling Design and Rationale, Page 36 of 70:** Worksheet #17 specifies that the bathymetric survey lines will be approximately 100 feet apart, but does not provide the rationale for why this spacing is sufficient to achieve project goals. Please revise Worksheet #17 to provide the rationale for the spacing of the bathymetric survey lines. | *Please see response to U.S. EPA Specific Comments 4 and 5.* |
| 13. | **Appendix A, Worksheet #17, Sampling Design and Rationale, Pages 36 of 70 and 37 of 70:** Worksheet #17 states that "At each former pier area, individual sub-samples will be collected for the composite at a rate of one sub-sample per 4,000 sf [square feet]," but does not provide the rationale for why this sampling rate is sufficient to meet project goals. Please revise Worksheet #17 to provide the rationale for the sampling frequency. | *Please see response to U.S. EPA Specific Comment 6.* |
| 14. | **Appendix A, Worksheet #18-1, Location-Specific Sampling Methods/SOP Requirements Table for Sediment Sampling, Page 38 of 70:** Worksheet #18-1 provides the sample identification (ID) number for the composite sample, but there are no sample ID numbers provided for the individual sub-samples. The individual sub-samples are to be retained by the laboratory for potential analysis, and therefore should have sample ID numbers. Please revise Worksheet #18-1 to include sample ID numbers for the individual sub-samples. | *The following note has been added to SAP WS#18-1:*<br>4. *"Sub-samples at each location will have the sample ID number appended with a letter from A-I. For example, HPP-FSS-001A, -001B, etc. "* |
| 15. | **Appendix A, Worksheet #18-1, Location-Specific Sampling Methods/SOP Requirements Table for Sediment Sampling, Page 38 of 70:** According to the second note of Worksheet #18-1, "Depth below surface of water will vary, and be noted in the field during sampling;" however, the Work Plan does not specify the | *SAP Worksheet 18.1 Note 2 has been modified as follows:*<br>*"Samples will be collected from* ***approximately the upper five centimeters of*** *the sediment surface* ***using a Van Veen grab sampler or equivalent.*** *Depth below surface of water will vary, and be noted in the field during sampling.* ***Depth (vertical elevation) of samples will be calculated from the survey data,*** |

| Comment | | Response |
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| | potential sample depth range that can be achieved with the Van Veen grab sampler. In addition, the Work Plan does not specify how the sample depth will be determined in the field. Please revise the Work Plan to specify the potential sample depth range that can be achieved with the Van Veen grab sampler and to describe how the sample depth will be determined in the field. | *and"*<br>*Please also see revisions to Section 4.4 of the Work Plan as indicated in response to U.S. EPA General Comment 1. Please note that due to the nature of grab sampling using the Van Veen sampler, the vertical penetration of the sampler into sediment is estimated based on volume of material collected and type of lithology. The volume and type of material will be documented in the field during sample collection. See also "Sediment Sampling" in SAP WS #14.* |
| 16. | **Appendix A, Worksheet #19, Field Sampling Requirements, Page 39 of 70:** The preservation requirements for mercury analyses do not specify that samples will be kept cool. The latest version of Method 7471 indicates that solid samples should be refrigerated to less than 6 degrees Celsius. Please revise this table to indicate that solid samples for analyses of mercury will be cooled. | *SAP WS #19, sample preservation for metals, has been updated to state: "Cool, ≤6°C".* |
| 17. | **Appendix A, Worksheet #22, Field Equipment Calibration, Maintenance, Testing, and Inspection Table, Page 43 of 70:** Worksheet #14 indicates a boat-mounted global positioning system (GPS) unit will be used to mark the sediment locations, but this table does not include the GPS. Please revise Worksheet #22 to include the GPS and the necessary accuracy for this measurement. | *GPS calibration has been added to SAP WS #22.* |
| 18. | **Appendix A, Worksheet #28, Laboratory QC Samples Tables, Pages 54, 56, and 58 of 70:** Control limits for the proposed analytes are provided, but the source of these limits is not identified. It appears that several of the limits are from the DoD QSM, but it is unclear if laboratory limits are also provided. Please note that laboratory QC limits should be provided for all analytes to ensure the measurement performance criteria can | *Limits provided in SAP WS#28 are from the laboratory. Some of the laboratory limits do match DoD QSM limits, and the QSM is the standard that is applied.*<br>*The following footnote has been added to SAP WS#28:*<br>*"Limits in WS#28 are provided by the laboratory."* |

| Comment | | Response |
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| | be met. Please clarify the source of the control limits for each analyte, and ensure the: laboratory limits for all analytes are provided. | |
| 19. | **Appendix A, Worksheet #28, Laboratory QC Samples Tables, Pages 53 to 54 of 70:** The corrective action for the Method 6020 matrix spike/matrix spike duplicate (MS/MSD) should indicate that the post-digestion spike (PDS) will be analyzed when the MS/MSD fails to meet acceptance criteria. Please revise the corrective action for the MS/MSD to indicate that a PDS will be analyzed when the MS/MSD are outside the acceptance limits. | *Post digestion spike has been added to SAP WS #28.* |
| 20. | **Appendix A, Worksheet #28, Laboratory QC Samples Tables, Pages 57 to 58 of 70:** Second column confirmation is missing from this table. As noted in the laboratory SOP SVOC 3.2, Section 9.5, the laboratory uses second-column confirmations for all samples and reports the higher of the two detected results from the columns. Please revise this table to include second-column confirmation for detections of PCBs. | *Second column confirmation has been added to SAP WS#28.3.* |
| 21. | **Appendix A, Worksheet #37, Usability Assessment, Pages 67 to 69 of 70:** This worksheet indicates a data usability assessment report will be generated to discuss the findings of the data evaluations, but does not specify what will be included in this report. For example, the data usability assessment report should include a detailed description of how precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) were evaluated for | *SAP WS #37, first paragraph, has been updated with a more detailed description of the data usability assessment report.*<br>*"The report will include a description of how PARCC parameters were evaluated, a discussion of any significant trends and biases in the QC results, a summary of the data validation findings, and sufficient information to support data usability conclusions."* |

| Comment | | Response |
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| | the project data and QC results and provide sufficient information to support the data usability conclusions. In addition, significant trends and biases in the QC results should be evaluated and discussed in this report. Please revise the SAP to indicate that the data usability assessment report will include detailed discussions of the data usability evaluations (e.g., PARCCS, trends and biases), along with sufficient information to support the data usability conclusions. | |
| 22. | **Appendix B, Section 4.0, Wildlife Resources, Page 4-1:** Section 4.0 states that "If marine wildlife is spotted during offshore activities, work in the vicinity of the wildlife will be postponed, to the extent practicable, until wildlife is a safe distance from the vessel;'.' however, the text does not specify the distance that qualifies as "safe." In addition, Section 4.0 does not specify how a safe distance will be managed (e.g., a spotter will be assigned to watch for marine wildlife, periodic observations will be made, etc.). Please revise Section 4.0 of Appendix B to specify the "safe distance" for working in the vicinity of marine wildlife. Please also revise Section 4.0 to specify how the safe distance will be managed. | *The last paragraph of Appendix B, Section 4 has been modified as follows:*<br>*"Periodic observations of the seascape will be made by the field crew during offshore activities. If marine* ***mammals*** *~~wildlife~~ are spotted during offshore activities, work in the vicinity of the wildlife will be postponed, to the extent practicable, until wildlife is a safe distance from the vessel.* ***Safe distances from marine mammals will be maintained in accordance with the Marine Mammal Protection Act as follows: 150 feet for wild dolphins, porpoises, and seals; and 300 feet for whales (U.S. Marine Mammal Act, 1972).****"* |

**END OF EPA COMMENTS**

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

*The following comments were received from Ms. Nina Bacey, Project Manager at Department of Toxic Substances Control on August 29, 2017:*

| **Comment** | | **Response** |
|---|---|---|
| | **General Comments:** | |
| 1. | Section 3.2 and Worksheet 11, Table 11-1, Worksheet 17 - It's unclear at what range of depth sediment samples will be collected. It appears to be surface sediment collection only. Previous sediment investigations in parcel F included sample collection from multiple depths (0 to 90 cm at 5 to 30 cm increments, FS Data Gaps Investigation, 2003; To a depth of 10 ft at 2 ft increments, Validation Study, 2001). In addition, samples collected at similar depths were composited. No justification has been provided to conduct only surface sediment samples rather than samples from multiple depths to fully characterize the six areas in question. DTSC recommends samples be collected at multiple depths, and similar depths be composited as was conducted in the past. | *Please see response to U.S. EPA General Comment 1.* |
| 2. | DTSC understands the heterogeneity concern when collecting duplicate sediment samples. That said, DTSC asks that you still consider collecting duplicate samples. Both the sample size collected by Van Veen sampler, and the composite process to be conducted by the laboratory, will allow for a duplicate samples to be collected. | *Field duplicates have been added to SAP WS#12.* |

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

| Comment | | Response |
|---|---|---|
| | **Specific Comments:** | |
| 1. | **SAP, Worksheet #3,** Nina Bacey phone number should be (510) 540-2480. Please correct. | *Nina Bacey's phone number has been corrected.* |
| 2. | **SAP, Worksheet 14,** Equipment Decontamination - Indicates equipment will be cleaned after each use. This conflicts with the text of the Work Plan (Sect. 4.5) which indicates equipment will be cleaned between each sample location. Please correct. | *The SAP WS #14, "Equipment Decontamination," has been updated to match the Work Plan Section 4.5.* |

**END OF DTSC COMMENTS**

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

*The following comments were received from Ms. Tina Ures, Engineering Geologist at San Francisco Bay Regional Water Quality Control Board (Water Board) on August 29, 2017:*

| Comment | | Response |
|---|---|---|
| | **Specific Comments:** | |
| 1. | **Section 1.3 Work Plan Objectives:** Explain why only three chemicals of concern (COCs) (copper, mercury, and polychlorinated biphenyls (PCBs)) are being addressed. The objective of this document is to provide additional data to address previously identified data gaps based on BCT comments. U.S. EPA recommended samples be analyzed for metals, polynuclear aromatic hydrocarbons and constituents of creosote, pentachlorophenol, PCBs, dioxins, and radionuclides to address data gaps. | *Please see response to U.S. EPA General Comment 1a.* |
| 2. | **Section 2.3 Remedial Action Objectives and Remediation Goals:** Include lead in the sediment sample analysis. Characterization of the extent of lead has been identified in the Feasibility Study Report for Parcel F (Feasibility Study)[1] as a remedial action objective (RAO). | *Lead has been added as a COC and the text in Section 2.3, second paragraph, has been modified as follows (text additions bolded):*<br>*"**The COCs (copper, lead, mercury, and total PCBs) in sediment were identified based on potential risks to ecological receptors.** In addition, numerical remediation goals were developed for the COCs found in Parcel F sediments **with the exception of lead. A numerical remediation goal was not calculated for lead because of the uncertainty associated with both the bioavailability and toxicity of lead. Instead, lead will be addressed qualitatively (Barajas, 2008).**"* |

| Comment | | Response |
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| 3. | **Section 3.2 Sediment Sampling:** Include proposed sampling depths and the rationale for the depths. This information is not presented in the Draft Work Plan. | *Please see response to U.S. EPA General Comment 1b.* |
| 4. | **SAP Worksheet #10: Conceptual Site Model, Nature and Extent of Contamination:**<br>**a.** Revise the second sentence of the first paragraph to state "potential contamination at Parcel F is associated with metals (copper, mercury, and lead)." Lead should be included as a potential contaminant at Parcel F, since it is listed as a COC for Parcel F in the Feasibility Study.<br>**b.** Explain how the proposed sample depths will be representative of previously detected concentrations. The text in Section 3.2 Sediment Sampling states "surface sediment samples (above 1 feet bml)" will be collected. However, some of the highest concentrations of copper were reported at depths of approximately 2.0 to 3.0 feet below mudline. Regional Water Board staff does not concur with collecting samples from only the surface sediment. | *a. The sentence in SAP WS #10, "Nature and Extent of Contamination," has been revised as indicated.*<br>*b. Please see response to U.S. EPA General Comment 1b. In addition, the sentence referenced in Section 3.2, first paragraph, has been modified to reflect sediment samples will be collected from the upper five centimeters of the sediment surface* |
| 5. | **SAP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements, 2. Identify the Goals of the Study:** List lead as a COC. While a cleanup goal has not yet been established, the RAOs include characterization of lead, and, therefore, lead should be characterized qualitatively to be consistent with the Feasibility Study. | *Lead has been added to the SAP and WP as a COC. Please see response to U.S. EPA General Comments 1a and 2.* |

| Comment | | Response |
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| 6. | **SAP Worksheet #14: Summary of Project Tasks, Data Gap Investigation, Sediment Sampling:** Confirm that Van Veen grab sampler can collect samples at multiple depths below mudline. | *Please see response to Water Board Comment 4b.* |
| 7. | **SAP Worksheet #15.2: Reference Limits and Evaluation Tables for metals by EPA 6020/7471A:** Correct the copper project action level to 271,000 micrograms per kilogram to be consistent with the Feasibility Study. | *The PAL for copper has been corrected in SAP WS#15.2.* |

**END OF WATER BOARD COMMENTS**

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

*The following comments were received from Ms. Amy D. Brownell, Environmental Engineer at City and County of San Francisco Department of Public Health on August 30, 2017:*

| Comment | | Response |
|---|---|---|
| | **Specific Comments:** | |
| 1. | **Subsection 2.5, Current Conceptual Site Model, page 2-2:** The second sentence stating "The flow charts provide a summary of the relationships between potential chemical sources, release mechanisms, potentially complete Sediment Investigation and Bathymetric Survey transport pathways, exposure media, potential current and future exposure pathways and receptors." For ease of access and clarity, please include these flow charts in this workplan, or with SAP#10 in the attached Draft Sampling and Analysis Plan. | *The Conceptual Site Model figures have been added as Figures 2-2 and 2-3, and referenced in Section 2.5 and response to U.S. EPA Specific Comment 1.* |
| 2. | **Subsection 2.5.3, Data Gaps in Conceptual Site Model, page 2-4:** BCT comments included in Attachment 1 also requested that the Navy analyze samples for PAHs and creosote constituents that may have migrated from and beneath former piers. The Navy responded that that they were considering sampling for these constituents as part of the upcoming Data Gap Investigation in the South Basin but did not mention analyzing sediment form Former Parcels B or C pier structure locations. We suggest adding these chemicals to the SAP or providing rationale as to why these analyses are not needed. | *Please see response to U.S. EPA General Comment 1a.* |

| Comment | | Response |
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| 3. | **Subsection 3.2, Sediment Sampling, page 3-4:** The fourth sentence from the top of the page, stating "To ensure a denser coverage in the larger areas, 6 individual sub-samples will be composited for Former Wharf #2, and 10 individual subsamples will be composited for former Piers B and C." We question this approach due to collecting and compositing more than 4 individual samples at Former Wharf#2 and at former Piers B and C. This is not in conformance with the San Francisco Bay Regional Water Quality Control Board guidance (Water Board, 2000[1]) for testing of dredged materials for determining the suitability sediments for beneficial reuse. The Water Board suggests four samples be composited for the indicated areas and volumes at each location. Compositing more than four samples may dilute the COCs such that concentrations appear to be below the final remediation goals. We suggest collecting and compositing no more than four samples for each analysis.<br><br>[1] Water Board, 2000. Draft Staff Report, Beneficial Reuse of Dredged Materials: Sediment Screening and Testing Guidelines. May. | *Concur. The collection of sediment samples in the Work Plan and the SAP has been modified to follow the Water Board (2000) recommendations that a maximum of 4 subsamples be used to generate a composite sample. Please see response to U.S. EPA General Comment 2.* |
| 4. | **SAP Worksheet #18-1 in the attached Draft Sampling and Analysis Plan:** Please adjust the number of composited samples according to the comment #4 in this letter. | *SAP WS# 18.1 has been updated accordingly.* |

**Response to Comments on**
**Draft Work Plan and Sampling and Analysis Plan (SAP)**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – June 2017**

| Comment | | Response |
|---|---|---|
| | **Minor Comments:** | |
| 1. | **Subsection 2.5.2, Contaminant Extent and Distribution in Area III, Page 2-4,** first paragraph**,** second sentence, stating **"**The focus of this investigation beneath the former piers is designed to address the COCs to support the Proposed Plan, ROD and Remedial Design for Parcel F." Please consider re-phrasing to read "This investigation is focused on the areas beneath the former piers and designed to address the COCs, in support of the Proposed Plan, ROD, and Remedial Design for Parcel F." | *The referenced sentence has been modified accordingly.* |
| 2. | **Section 3.0 Technical Approach**, in second sentence, stating "Sediment sampling will be conducted at six locations for Parcel F COCs where the Parcel B and C pier and wharf structures have been removed as summarized in the Removal Action Completion Report (ERS, 2012; Figure 3-2)." Please consider revising and adding commas as follows "Sediment sampling will be conducted at six locations for Parcel F COCs, where the Parcel B and C pier and wharf structures have been removed, as summarized in the Removal Action Completion Report (ERS, 2012; Figure 3-2)." | *The referenced sentence has been modified accordingly.* |

**END OF SFDPH COMMENTS**

**Enclosures:** Draft Final Figures including updates to Figures 2-2, 2-3, and 3-2; Work Plan track changes; SAP track changes; Appendix B track changes.

**Response to Comments on**
**Draft Final Work Plan**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – September 2017**

*The following comments were received via email from Ms. Judy C. Huang, Remedial Project Manager at United States Environmental Protection Agency (EPA)-Region IX October 12, 2017:*

| Comment | | Response |
|---|---|---|
| 1. | **Response to General Comment 1a and Specific Comment 2:** The response addresses the comment; however, there are applicable parts of the Redline Draft Final Work Plan Sediment Investigation and Bathymetric Survey for Parcel F, Hunters Point Naval Shipyard, San Francisco, California, September 2017 (the Redline Work Plan) where lead has not been incorporated. For example, lead has not been added to the reference limits table presented in Worksheet #15.2 of Appendix A. In addition, no numerical remediation goal was developed for lead due to uncertainty about its bioavailability. The Redline Work Plan should explain how a qualitative evaluation will be conducted if lead is detected. Please ensure lead has been incorporated into all applicable parts of the Redline Work Plan. Please also revise the Redline Work Plan to how a qualitative evaluation will be conducted if lead is detected since no numerical remediation goal was developed for lead. | *The National Oceanic and Atmospheric Administration (NOAA) Effects Range Median (ER-M) value for the protection of the benthic community (218 mg/kg) will be used as the reference comparison for lead. Based on further discussions between the Navy and Ms. Judy Huang on October 31, 2017, it was agreed that one-half (½) of the PRG or ER-M (lead) value would be used as a screening value for the composite sample to determine if additional analyses are needed for the respective subsamples. All references to conducting a qualitative evaluation for lead have been removed. In addition, the following edits are proposed:*<br>1. *The ER-M reference for lead has been added as a Project Action Level (PAL) to SAP Worksheet #15.2.*<br>2. *SAP Worksheet #11, 4 – Define the Boundaries of the Study. The second paragraph has been revised as follows, with changes in bold:*<br>*"Composite samples will be analyzed for Site COCs. Site COCs with RGs can be found in Worksheet #15 and are presented as PALs. PALs are equivalent to the PRGs established in the FS (Barajas, 2008),* ***with the exception of lead. The established PAL for lead is the National Oceanic and Atmospheric Administration (NOAA) Effects Range Median (ER-M) value for the protection of the benthic community (NOAA, 1999).*** *Sample analysis by location can be found in Worksheet #18."*<br>3. *The second and third sentence in the second to last paragraph of Section 3.2 of the Work Plan has been revised as follows, with changes in bold:*<br>*"Composite sediment sample results will be compared to the RAO2 "area-weighted average" PRG for PCBs and the RAO1 "do-not-exceed" PRGs for metals,* ***with the exception of lead****. T****he National Oceanic and Atmospheric administration (NOAA) Effects Range Median (ER-M) value for the protection of the benthic community will be used as a numeric screening value for lead*** |

| Comment | | Response |
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| | | ***(218 mg/kg).*** *If composite sediment sample COC concentrations from any former pier area exceed* ***half (½)*** *the Project Action Levels (PALs), then all individual sub-samples placed on hold in the laboratory for that area, will be analyzed for the Area III COCs (PCBs, copper, mercury, and lead) to further refine any area of contamination below the former piers."* |
| 2. | **Response to General Comment 2, Specific Comment 6, and Specific Comment 13:** The response addresses the comment; however, if four-sample composites are collected, then the screening criteria should be divided by four. If one of the samples has elevated concentrations, then the other three samples would dilute the results and the screening criteria may not be exceeded. Please revise the responses to discuss the reduction of the screening criteria to account for potential dilution during composite sampling. | *Due to some uncertainty in the heterogeneity of individual samples comprising the composites, EPA requested in an October 31, 2017 telephone conference with the Navy that composite sample results be screened against ½ the PALs as a conservative measure to take into account dilution of potential hot spot areas. Therefore, the following text will be revised as specified:*<br><br>*SAP Worksheet #11, Table 11-1 last bullet, with changes in bold :*<br><br>• *If laboratory composited sediment samples exceed ½ Project Action Levels (PALs)* ***[RAO 2 – Area Weighted Average PRG for PCBs, RAO1 – Do Not Exceed PRGs for Cu and Hg, and ER-M value for Pb]****, all individual sub-samples that make up the composite will be analyzed for all the same site COCs as the composite as well.*<br><br>*SAP Worksheet #11, 5 – Develop the Analytical Approach, with changes in bold:*<br><br>• ***If composite sediment sample COCs are below ½ the PALs (RAO2 – Area Weighted Average PRG for PCBs, RAO1 – Do-Not-Exceed PRGs for Cu and Hg, and ER-M value for Pb), then no further action will be necessary for the associated pier area, and***<br><br>• *If composite sediment sample COCs are above ½ the PALs* ***(RAO 2 – Area Weighted Average PRG for PCBs, RAO1 – Do-Not-Exceed PRGs for Cu and Hg, and ER-M value for Pb)****, then individual sub-samples retained by the laboratory remaining after preparation of the original composite sample,* ***will be*** *analyzed for COCs to further refine the location of the COC exceedance. The individual sub-samples will be screened against the RAO 1 –* ***Do-Not-Exceed*** *PRGs or ER-M value for* |

| Comment | | Response |
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| | | *Pb. Please note that the laboratory will be requested to retain the remaining individual sub-samples after they have prepared composite samples.*<br>• *If individual sediment sub-sample COC results are below the Do-Not-Exceed PALs, then no further action will be necessary for the associated pier area.*<br>• *If individual sediment sub-sample COC results are above the Do-Not-Exceed PALs, then there will be a discussion with the Navy and the BCT to determine the next course of action.*<br>*In addition, Worksheet #17 is revised as follows, with the changes in bold:*<br>*"In the event that composite sample analytical results exceed **½** the PALs (e.g. RAO2 Area weighted Average for PCBs) established, all the individual sub-samples that make up that composite will be analyzed for all the same COCs (Cu, Hg, Pb, and PCBs). "*<br>*Also, for clarification regarding PRGs/PALs, composite screening levels, and individual sub-sample screening levels, two new tables; Table 2-1 (Remedial Action Objectives and Preliminary Remediation Goals for Chemicals of Concern) and Table 3-1 (Sediment Sample Screening Levels), have been added to Work Plan Sections 2.2 and 3.2, respectively, and are attached for your reference.*<br>*The following footnotes have also been added to SAP Worksheets 15.1 and 15.2:*<br>*Worksheet 15.1: Reference Limits and Evaluation Tables for PCBs by EPA 8082:*<br>*Sub-sample Analysis:* ***If the composite sample exceeds ½ the RAO2 Area weighted average PRG for PCBs, all sub-samples associated with that sample will be analyzed for all tests.***<br>*Worksheet 15.2: Reference Limits and Evaluation Tables of metals by EPA 6020/7471A:* |

| Comment | | Response |
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| | | *General footnote:* ***PALs are based on the PRGs established in the FS (Barajas, 2008), with the exception of lead. The PAL for lead is based on the NOAA ER-M value for the protection of the benthic community (NOAA, 1999).***<br><br>***Sub-Sample Analysis: If the composite sample exceeds ½ the PAL for metals, all sub-samples associated with the parent sample will be analyzed for all tests.*** |
| 3. | **Response to Specific Comment 3:** The response does not address the comment. The comment asks for criteria for determining whether a tide board or real time tide gauge will be used to check against water surface elevations; however, the response discusses corrections for vessel position against global positioning system survey control points. Please revise the response to provide criteria for determining whether a tide board or real time tide gauge will be used to check against water surface elevations. | *The bathymetric survey vendor, Fugro, will establish a Real-Time Kinematic (RTK) base station on a recovered or established control point on land. This base station will transmit Compact Measurement Record (CMR+) corrections to the RTK Rover on the survey vessel to obtain real time tide levels using Hypack navigation software. After further evaluation, due to logistical considerations, neither a tide board nor real time tide gauge will be utilized for quality control of the RTK tide level measurements. To confirm Hypack tide levels, a QC point will be established using RTK techniques on a nearby dock, if possible, and real time water line measurements will be read from this point to establish tide levels. If a dock QC point cannot be established, direct water line measurements will be made from the RTK Rover to confirm Hypack RTK tide readings as collected.*<br><br>*The following has been added to the second paragraph of Section 3.1 of the Work Plan, and Worksheet #17, "Bathymetric Survey," of the SAP as follows:*<br><br>*"A quality control point will be established using RTK techniques on a nearby dock, if possible, to confirm real time tide levels collected with the Hypack navigation software aboard the vessel. If a viable dock QC control point cannot be established, direct water line measurements will be made from the RTK Rover to confirm Hypack RTK tide readings as collected. "*<br><br>*All references to utilizing a tide board and/or tide gauge have been removed.* |
| 4. | **Response to Specific Comment 4:** The response addresses the comment, but does not state the width that will be mapped in Area IX/X. It is understood that the single-beam survey is necessary, but it is unclear what percentage of the | *The bathymetric survey vendor, Fugro, will utilize a 200 kHz narrow beam (3°) transducer for single-beam data acquisition within Area IX/X due to the shallow water in this area (less than 6 feet). The whole area will be mapped but the bathymetric data evaluation will be interpolated through mathematical modelling between each track. Due to the fan shape of the emitted sound pulses,* |

| Comment | | Response |
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| | area will actually be mapped given the 100-foot spacing of the survey lines. Please revise the response to provide the percentage of the area that will be mapped in Area IX/X. | *the width of the transducer footprint is variable based on the depth to seafloor along each survey line. The width of coverage for a 3° narrow single-beam transducer is equal to approximately 5% of the distance between the water surface and the seafloor. So, at 10' above the seafloor the transducer will achieve approximately 0.5'-wide coverage along the survey track, and at 5' above the seafloor it will achieve approximately 0.25'-wide coverage along the survey track.*<br><br>*In multiple discussions with Fugro and further analysis of the historic bathymetric data, it appears that only 2010 bathymetric data (Lidar) is available for the shallow portion of the South Basin (Areas IX/X) for historical comparison. Hence, since a direct comparison to the 100-foot single-beam survey spacing is not available for this area, the Navy agrees that a tighter spacing may be beneficial to support the Remedial Design phase. Hence, a **50**-foot spacing is proposed for Area IX/X. This response changes our response to the referenced U.S. EPA Specific Comment 4 and the text in Section 3.1 of the Work Plan and SAP Worksheet #17, "Bathymetric Survey." In addition, Figure 3.1 has been updated to reflect this change.*<br><br>*1. The following text has been added to the first paragraph of Section 3.1 of the Work Plan and Worksheets #11 and #17, with changes in bold:*<br><br>*"Bathymetric surveys will be conducted within three areas: 1) area in footprint adjacent to sections of the former Parcel B and C shoreline piers and wharf structures removed in 2011 (ERS, 2012); 2) Area III; and 3) Area IX/X (Figures 2-1 and 3-1). Due to the differences in water depths between these areas, different survey technologies are proposed. For the deeper areas (former Parcel B and C piers, wharfs and Area III), a 27-foot survey vessel equipped with a multi-beam bathymetric echo sounder is proposed. For the shallow areas (Area IX/X), a survey skiff equipped with a single-beam echo sounder is proposed. Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet apart **for the deeper areas and 50-feet apart in the shallower areas (Area IX/X).** Since the depth to water in Area IX/X is less than 6 feet (Battelle, BBL, and* |

| Comment | | Response |
|---|---|---|
| | | *Neptune & Company, 2005), the **50**-foot spacing in this shallow area will provide sufficient resolution with the single beam survey. **A narrow single-beam (3°) transducer will be utilized for data acquisition within Area IX/X. Due to the fan shape of the emitted sound pulses, the width of the transducer footprint is variable based on the depth to seafloor along each survey line. The width of coverage is equal to approximately 5% of the distance between the water surface and the seafloor (e.g., at 10' above the seafloor the transducer will achieve approximately 0.5'-wide coverage along the survey track, while at 5' above the seafloor it will achieve approximately 0.25'-wide coverage along the survey track).** For the deeper waters beneath the former pier and wharf structures and Area III, the multi-beam survey provides greater resolution (see Section 3.1.1) that will identify change in bathymetry between the 100-foot lines. The resulting bathymetric survey data will be processed to provide 1-foot contours. Proposed survey areas and approximate line paths are provided on Figure 3-1."*<br><br>*2. In addition, the data will be compared to historic data (e.g. 2010 and 2003 data, as available). Hence, the last sentence in Section 3.1.6 was changed as follows with changes in bold:*<br><br>*"Resulting data sets from this bathymetric survey will be compared against **historical** bathymetric survey data and a comparison map will be provided to assist in showing areas of sediment deposition and erosion."*<br><br>*3. The following sentence was changed to the second sentence in Section 4.3 of the Work Plan, with changes indicated in bold:*<br><br>*"Surveys will be conducted perpendicular to the shoreline along survey lines approximately 100 feet **(multi-beam bathymetric survey) and 50 feet apart (Area IX/X).** "*<br><br>*4. The information was also updated in the SAP in the following sections as follows, with changes in bold:*<br><br>*"Current bathymetry surveys of Areas III, IX, and X are also required to accurately estimate the areas and volumes of sediments requiring remediation, to identify* |

| Comment | | Response |
|---|---|---|
| | | *the placement of fill and capping materials during implementation of the selected alternative, and to estimate sediment erosion/deposition* ***compared to historic data****."* |

**END OF EPA COMMENTS**

**Response to Comments on**
**Draft Final Work Plan**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – September 2017**

The following comments were received via email from Ms. Nina Bacey, Project Manager, Cal EPA, CA Dept. of Toxic Substances Control (DTSC), on October 23, 2017:

| Comment | Response |
|---|---|
| **General Comment 1:**<br>1b - Navy Response - Surface sediment samples are appropriate to determine if any COCs identified in the FS are present in the area beneath the former Piers and wharf structure.<br><br>How so? Per the 2008 FS Report, results of a literature review indicated that the depth of the biologically active zone in marine sediments averages about 10 centimeters. DTSC does not agree that surface sediment samples to a depth of 5 cm is appropriate and requests samples be collected at a minimum at 5 and 10 cm.<br><br>Navy Response - If the results of the surface sediment sampling indicate that subsurface sediment samples are required, additional sampling will be conducted as part of the pre-design investigation in support of the remedial design.<br><br>Has the Navy considered the sediment accumulation rate for this area and the affect it would have on past contamination deposits? It is likely COCs would not be identified in the top 5 cm due to sediment accumulation but may be identified at a deeper depth. DTSC request samples be collected at least to a depth of 10 cm.<br><br>The rest of the RTCs are acceptable. If you have any questions, please feel free to contact me. | *During several telephone conversations between the DTSC, Water Board, and Navy RPMs and their contractors on October 24-26, 2017, agreement was reached to maintain the surface sample depth interval of 0-5 cm, which is consistent with the surface sampling depths of previous Parcel F studies.* |

**END OF DTSC COMMENTS**

**Response to Comments on**
**Draft Final Work Plan**
**Sediment Investigation and Bathymetric Survey for Parcel F**
**Hunters Point Naval Shipyard, San Francisco, CA – September 2017**

The following comments were received via email from Ms. Tina Ures, Project Manager, Regional Water Quality Control Board San Francisco Bay Region (Water Board), on October 25, 2017:

| Comment | Response |
|---|---|
| **General Comment 1:**<br>Navy's Response to 4b: "Please see response to U.S. EPA General Comment 1b. In addition, the sentence referenced in Section 3.2, first paragraph, has been modified to reflect sediment samples will be collected from the upper five centimeters of the sediment surface"<br><br>How will the proposed sample depth (five centimeters) be representative of previously detected concentrations? Regional Water Board staff does not concur with collecting samples from only the upper five centimeters of the sediment surface. | *During several telephone conversations between the DTSC, Water Board, and Navy RPMs and their contractors on October 24-26, 2017, agreement was reached to maintain the surface sample depth interval of 0-5 cm, which is consistent with the surface sampling depths of previous Parcel F studies.* |

**END OF WATER BOARD COMMENTS**

**Table 2-1**
**Remedial Action Objectives and Preliminary Remediation Goals for Chemicals of Concern**

| RAO | COC | PRG/PAL (µg/kg) | Basis |
|---|---|---|---|
| **RAO 1** | Copper | 271,000 | Not to exceed threshold |
| **RAO 1** | Lead | NE; 218,000 [1] | |
| **RAO 1** | Mercury | 1,870 | |
| **RAO 1** | PCBs (total) | 1,240 | |
| **RAO 2** | PCBs (total) | 1,350 | Area-weighted average |
| **RAO 3** | PCBs (total) | 200[2] | |

[1]A PRG for lead was not established, due to uncertainty associated with bioavailability and toxicity of lead. Lead is collocated with PCBs in sediment, so achieving the cleanup goals for PCBs is expected to address any risks associated with lead. However, the National Oceanic and Atmospheric Administration Effects Range-Median (ER-M) value for the protection of the benthic community will be used as a numeric screening value for lead (218,000 µg/kg).

[2] 200 µg/kg total PCBs is based on background total PCB estimates for nearshore sediments in San Francisco Bay.

µg/kg – micrograms per kilogram
COC – chemical of concern
NE – not established
PAL – project action level
PCB – polychlorinated biphenyl
PRG – preliminary remediation goal
RAO – remedial action objective

**Table 3-1**
**Sediment Sample Screening Levels**

| COC | PAL (µg/kg) | Composite Sample Screening Level (µg/kg) [5] | Individual Sub-Sample Screening Level (µg/kg) [6] |
|---|---|---|---|
| Copper (Cu) | 271,000[1] | 135,500 | 271,000[1] |
| Lead (Pb) | 218,000[4] | 109,000 | 218,000[4] |
| Mercury (Hg) | 1,870[1] | 935 | 1,870[1] |
| PCBs (total) | 1,240[1]/1,350[2]/200[3] | 675 | 1,240[1] |

[1] RAO 1 – Not to exceed threshold.
[2] RAO 2 – Area-weighted average.
[3] RAO 3 – long-term goal based on an area-weighted average background near-shore PCB concentration.
[4] No PRG has been established for lead. The PAL for lead is based on the NOAA ER-M value for the protection of the benthic community (NOAA, 1999).
[5] Composite sample screening levels are ½ of the PALs (RAO 1 – Not-To-Exceed PRGs for Cu and Hg, ER-M value for Pb, and RAO 2 – Area Weighted Average PRG for PCBs).
[6] Individual sub-sample screening levels are the RAO 1 – Not-To-Exceed PRGs for Cu, Hg, and PCBs, and the ER-M value for Pb.

µg/kg – micrograms per kilogram
COC – chemical of concern
ER-M – Effects Range-Median
NOAA – National Oceanic and Atmospheric Administration
PAL – project action level
PCB – polychlorinated biphenyls
PRG – preliminary remediation goal
RAO – remedial action objective