20 May 2016

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Subject: Submittal of Final Work Plan for the Pilot Scale Abatement Study if Hangar 1, dated May 20, 2016.

Dear Ms. Huang and Ms. Wells,

On behalf of Planetary Ventures ("PV"), and Erler & Kalinowski, Inc. ("EKI") ACC Environmental Consultants, Inc. is transmitting the *Final Work Plan for the Pilot Scale Abatement Study of Hangar 1* ("Revised Work Plan") dated 20 May 2016 for your records.

This document satisfies all conditions and comments previously received from the United States Environmental Protection Agency, Region 9 ("U.S. EPA") and the California Regional Water Quality Control Board, San Francisco Bay Region ("Water Board") in response to the following correspondence.

- 20 October 2015 submittal by National Aeronautics and Space Administration ("NASA") of the Planetary Ventures, LLC ("PV") *Draft Work Plan for the Pilot Scale Abatement Study of Hangar 1* ("Draft Work Plan"), dated 21 September 2015, to the United States Environmental Protection Agency, Region 9 ("U.S. EPA") and the California Regional Water Quality Control Board, San Francisco Bay Region ("Water Board").

- 11 December 2015 U.S. EPA and Water Board comments on the Draft Work Plan to NASA.

- 11 March 2016, EKI submittal of the revised *Work Plan for the Pilot Scale Abatement Study of Hangar 1* ("Revised Work Plan") dated 11 March 2016 to the U.S. EPA and Water Board accompanied by a memorandum responding to the Agencies comments of 11 December 2015.

- 22 March 2016, ACC, EKI and PV walkthrough meeting with Ms. Judy Huang, U.S. EPA and Ms. Elizabeth Wells, Regional Water Board.

- 12 April 2016, U.S. EPA and the Regional Water Board advised EKI by e-mail correspondence of the completion of their review of the Work Plan and provided additional comments.

- 22 April 2016, EKI response to the April 12 comments and additional comments provided through phone conversations between the Agencies and EKI.

- 9 May 2016, EKI received additional review comments which were responded to by EKI on 16 May 2016.

- 17 May 2016, EKI received an e-mail from Judy Huang, EPA expressing that EPA and the Water Board reviewed the RTC and the attached revised document and were satisfied with the responses.

Please accept the attached documents as the final submittal of the *Work Plan for the Pilot Scale Abatement Study of Hangar 1* for your records. If you have any questions or wish further information please us know.

Sincerely,

Jim Wilson
President/CEO

Final

# Work Plan for the Pilot Scale Abatement Study of Hangar 1

September 21, 2015

Revised, March 11, 2016

Final, May 20, 2016

Hangar One, Moffett Federal Airfield

Mountain View, California

Prepared For:

Planetary Ventures, LLC

Kevin Antonelli, Project Executive, Development

1600 Amphitheater Parkway, Mountain View CA. 94043

ACC Project #:
1591-001.01

Proposed Final

# Work Plan for the Pilot Scale Abatement Study of Hangar 1

Hangar 1, Moffett Federal Airfield
Mountain View, California

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# Acronyms and Abbreviations

ACC: ACC Environmental Consultants
ACM: Asbestos-Containing Material
AFD: Air Filtration Device
ANSI: American National Standards Institute
BAAQMD: Bay Area Air Quality Management District
BRAC: Base Realignment and Closure
CAAQS: California Ambient Air Quality Standards
Cal/EPA: California Environmental Protection Agency
CAM: California Administrative Manual
CCR: California Code of Regulations
CERCLA: Comprehensive Environmental Response, Compensation, and Liability Act
CFR: Code of Federal Regulations
CM15: Carbomastic 15 epoxy coating
CMU: Concrete Masonite Unit
COC: Contaminant of Concern
DIR: The State of California Department of Industrial Relations
DOP: Dispersed Oil Particulate
DOSH: The State of California Division of Occupational Safety & Health
DOT: Department of Transportation
EE/CA: Engineering Evaluation/Cost Analysis
EIMP: Environmental Issues Management Plan
FFA: Federal Facilities Agreement
FFS: Focused Feasibility Study
GFCI: Ground-fault Circuit Interrupter
gpm: gallons per minute
HAZWOPER: Hazardous Waste Operations and Emergency Response Standard
HEPA: High-Efficiency Particulate Air
ITRC: Interstate Technology & Research Council
ISM: Incremental Sampling Methodology
LTMP: Long-Term Management Plan
$m^3$: cubic meter
mg/L: milligrams per liter
$mg/m^3$: milligrams per cubic meter
MFA: Moffett Federal Airfield
mg/kg: milligrams per kilogram
NAS: Naval Air Station
NASA: National Aeronautics and Space Administration
Navy: U.S. Department of the Navy
NIOSH: National Institute of Occupational Safety and Health
NIST: National Institute of Standards and Technology
NTCRA: Non-Time-Critical Removal Actions
OSHA: Occupational Safety and Health Administration
PAPR: Powered Air Purifying Respirators
PCB: Polychlorinated Biphenyls
PCS: Performance Characterization Sheet
PPE: Personal Protective Equipment
PV: Planetary Ventures, LLC.

PVC: Polyvinyl Chloride
QAPP: Quality Assurance Project Plan
QAM: Quality Assurance Manager
SCBA: Self-Contained Breathing Apparatus
SF: square feet
SSHSP: Site Specific Health and Safety Plan
TAT: Turn Around Time
TCLP: Toxicity Characteristic Leaching Procedure
TCRA: Time-Critical Removal Action
TSCA: Toxic Substances Control Act
TSDF: Treatment, Storage, Disposal Facility
TWA: time-weighted average
ug: micrograms
ug/100 $cm^2$: micrograms per 100 square centimeters
ug/$ft^2$: micrograms per square foot
ug/$m^3$: micrograms per cubic meter
U.S.: United States
USEPA: U.S. Environmental Protection Agency
VOC: Volatile Organic Compounds
Water Board: California Regional Water Quality Control Board, San Francisco Bay Region
WET: Waste Extraction Test
XRF: X-Ray Fluorescence Spectrometer

# 1. Introduction

On behalf of Planetary Ventures, LLC (PV), ACC Environmental Consultants (ACC) has prepared this Work Plan for the Pilot Scale Abatement Study of Hangar 1 to determine the feasibility of reducing PCB and lead concentrations in accessible structural steel elements and concrete masonry unit (CMU) walls within the Hangar 1 structure. The Project Plan for Quality Assurance has been prepared as Attachment E of this document and describes the quality assurance/quality control ("QA/QC") procedures to be used by the project team, and selected analytical laboratories to perform pilot scale study to ensure that the data collected is of sufficient quantity and known quality to be usable.

Hangar 1 is located within the former Naval Air Station (NAS) Moffett Field approximately 35 miles south of San Francisco and 10 miles north of San Jose (Figure 1). The former NAS Moffett field was commissioned in 1933 to serve as a base for the West Coast dirigibles of the lighter-than-air program and Hangar 1, located to the west of the airfield runways, was constructed to house the USS Macon dirigible. Between 1933 and 1994, the station was operated continuously by the United States (U.S.) Military. By 1950 when jet aircraft were introduced, NAS Moffett Field was the largest naval air transport base on the West Coast and became the first all-weather NAS. Between 1973 and 1994, the mission of NAS Moffett Field was to support anti-submarine warfare training and patrol squadrons (PRC, 1996). No heavy manufacturing or major aircraft maintenance was conducted during this last period of operation of NAS Moffett Field, although some maintenance activity occurred (Harding, 2000).

In 1987, the U.S. Environmental Protection Agency (USEPA) placed NAS Moffett Field on the National Priority List and on 10 September 1990 the U.S. Department of the Navy (Navy) signed a Federal Facility Agreement (FFA; USEPA, 1990) with the USEPA and the California Regional Water Quality Control Board, San Francisco Bay Region (Water Board) to conduct remedial actions at NAS Moffett Field pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and attendant regulations. This agreement was amended in December 1993 (USEPA, 1993).

In 1991, NAS Moffett Field was designated for closure as an active military base under the Department of Defense Base Realignment and Closure (BRAC) Program and in 1994, NAS Moffett Field was transferred to the National Aeronautics and Space Administration (NASA) and renamed Moffett Federal Airfield (MFA) (PRC, 1996).

In 2015, PV entered into a Lease with NASA for an approximately 1,000-acre parcel of land (MFA Leasehold) within NAS Moffett Field and includes Hangar 1. As part of the lease agreement, PV is responsible for "re-skinning" Hangar 1.

## *1.1 Hangar 1*

Hangar 1 is a large steel structure measuring approximately 1,133 feet long by 308 feet wide and 198 feet tall. Originally, the steel frame of Hangar 1 was covered with corrugated siding and a built-up asphalt roof, and the interior contained multi-story offices and shops located on both sides of the hangar deck, concrete electrical vaults, and a concrete floor. The area surrounding the hangar is paved, with the exception of several small areas of bare soil located on the eastern side of the hangar. A trench drain that discharges to the storm drain system surrounds the perimeter of Hangar 1.

Hangar 1 is listed as a National Landmark on the National Park Service Historic Registry and is a Civil Engineering Landmark of Northern California.

### 1.1.1 *Prior Investigations and Remedial Actions*

In 1997, NASA observed the presence of Aroclor 1268, a relatively uncommon polychlorinated biphenyl (PCB) mixture, in a storm water settling basin that receives storm water runoff from the western portion of the former NAS Moffett Field. In 1999, Aroclor 1260 and 1268 were detected in a storm water sample collected from a manhole downstream of Hangar 1. Subsequent investigations, implemented between 1999 and 2002 by NASA, determined that the Hangar 1 siding, commercially known as Robertson Protected Metal, contained PCBs and asbestos and that the lead-based paint used to cover both the siding and steel frame of the hangar also contained PCBs. Bulk samples of the lower (gray) siding were found to contain Aroclor 1260 and 1268 at concentrations as high as 5,500 mg/kg and 35,000 mg/kg, respectively (Benchmark, 2003).

In 2003, NASA and the Navy completed Time-Critical Removal Actions (TCRAs) that consisted of removing contaminated sediments from the storm water collection trench and coating the corrugated siding with an asphalt emulsion to mitigate the migration of PCBs from the exterior surfaces of the hangar. Following the TCRAs, the Navy proposed to perform Non-Time-Critical Removal Actions (NTCRA) to address the known PCB contamination present in the Hangar 1 siding materials as a more effective long-term remedy than the Navy's TCRA. Thirteen alternatives to mitigate the known PCB contamination at Hangar 1 and reduce the potential negative impacts to human health and the environment from these materials were evaluated in the Engineering Evaluation/Cost Analysis (EE/CA; Navy, 2008) for the NTCRA. The preferred alternative involved the following:

- the complete removal of the Hangar 1 siding;
- the deconstruction of interior structures;
- the removal of debris to appropriate off-site disposal or recycling facilities; and,
- the application of an epoxy coating (Carbomastic® 15; CM15) to the hangar's structural steel frame (AMEC, 2013).

Implementation of the NTCRA began in June 2010 and was completed in December 2012. During the NTCRA, with the exception of the (1) structural steel frame; (2) CMU walls surrounding six electrical vaults, the former hazardous materials storage room, and former restrooms; and (3) the door operating mechanisms, all other areas of the hangar (e.g., the surface of the concrete floor, the top and bottom sides of the metal mezzanine decks and I-beams supporting the mezzanine decks, etc.) were remediated completely to remove all PCB contamination. In areas where PCBs remain (i.e., the structural steel frame, CMU walls, and door operating mechanisms), the structures were over coated with a CM15 epoxy coating (RORE, 2013; AMEC, 2013). The NTCRA completion efforts included a final wash down and decontamination of the Hangar 1 structural steel frame, concrete floor, and storm water conveyance trenches and drains.

Following implementation of the NTCRA, a Focused Feasibility Study (FFS; RORE, 2013) was prepared for the Navy to evaluate short- and long-term options to ensure the protectiveness of the NTCRA. The Navy's Proposed Plan for Hangar 1 (Navy, 2013) summarized the information detailed in the FFS and announced that the Navy's preferred alternative was the implementation of institutional controls.[1]

As paint containing PCBs and lead remain at Hangar 1, the Navy prepared a Long-Term Management Plan (LTMP) for PCB contamination at Hangar 1 (AMEC, 2013) that outlines the coating inspection and

[1] Select potential institutional controls identified in the Navy's Proposed Plan (Navy, 2013) include: (1) installation and maintenance of signs notifying of the potential exposure hazard, (2) administrative arrangements for access for future monitoring/maintenance, (3) property owner and tenant commitment to inspection and maintenance of the CM15 epoxy coating, (4) sediment sampling, and (5) regulatory agency approval of building modifications that might damage the remedy components.

maintenance methods and procedures and storm water sediment monitoring program that are required to maintain the viability and effectiveness of the NTCRA; the LTMP has not been approved by the regulatory agencies. The LTMP indicated that these long-term management activities will present various job hazards to workers, including the potential for PCB exposure. In addition, the LTMP indicated that the owner/operator of the Hangar 1 structure would need to develop procedures for controlling the use of the hangar and that modifications to the hangar such as cutting, drilling, grinding, abrasion, welding, fastening, or impact that could damage the CM15 epoxy coating and expose the underlying PCB contamination.

In letters to the USEPA dated 23 October 2015 and 22 December 2015, NASA confirmed to the US EPA that NASA assumed the Navy's obligations with respect to IR Site 29 (defined as including the above ground frame and concrete floor of the Hangar, existing exposed surface soils located outside the eastern side of the Hangar, the door opening mechanisms, and the stormwater trench that surrounds the Hangar); NASA has not accepted responsibility for any of the contamination that may exist in the soil or groundwater beneath Hangar 1. NASA, the USEPA, and the Water Board have agreed to amend the November 2014 NASA FFA Site Management Plan to include the IR Site 29 obligations.

### *1.2 Objectives of Pilot Scale Abatement Study*

The primary objectives of the pilot scale abatement study (Pilot Study) are to determine the feasibility of reducing PCB and lead concentrations in accessible[2] structural steel elements and CMU walls within the Hangar 1 structure to:

1) Concentrations that could potentially eliminate the need for institutional or engineering controls at Hangar 1;
2) Minimize the potential exposure of future workers to PCBs and lead during long-term operations and maintenance activities; and,
3) Minimize the need for institutional controls such as the installation and maintenance of signage regarding the potential exposure hazard and the necessity of obtaining regulatory agency approval of building modifications that might damage remedy components.

The data collected and observations made during this Pilot Study will aid in determining the best means and methods to achieve the overall project goal of the safe removal of accessible PCB- and lead-impacted coatings from the Hangar 1 structure. The Pilot Study will also assess whether target Post-Abatement Criteria goals can be achieved using any of the three different abatement technologies and analyze the implementability and production rates of these technologies. The following criteria will be considered in selecting the appropriate technology for full-scale abatement.

- Effectiveness in achieving post abatement acceptance criteria
- Waste minimization: Which method produced the least waste?
- Water use: Which method used the least amount of water?
- Ease of use

[2] Accessible surfaces are all surfaces throughout the Hangar 1 structure that are not tight-tolerance mated surfaces (e.g., steel members riveted together) and will include all mated surfaces with greater than a ½" gap. Caulk or other coatings will be removed prior to determining whether a surface is a tight-tolerance mated surface.

- Safety of use: PPE efficiency, personal air sampling comparisons
- Equipment performance: Which equipment required least maintenance?

Each removal method will be rated on each of these criteria (best, acceptable, and not acceptable). A removal method will be eliminated if it fails to achieve the concentrations specified in the post abatement criteria. A final decision will be made by weighing the performance of each option against the desirable characteristics. Historical data is presented herein for informational purposes only; it will not be referenced or used in this study. The areas abated during the Pilot Study will be recoated with the CM15 coating and inspected by a qualified independent inspector.

NASA's Environmental Management Division will oversee all Pilot Study activities and will be responsible for the final inspection and sign off of the Pilot Study.

## 2. Site Conditions and Background

The results of a number of historical building material surveys conducted at Hangar 1 were provided to PV by NASA and the Navy. These surveys indicated that PCBs, lead, and asbestos-containing materials (ACM) were present in the Hangar 1 building materials. On completion of the NTCRA, the Navy's Final After Action Completion Report (AMEC, 2013) indicated that only PCBs and lead remained in the paint on the (1) structural steel frame, (2) CMU walls surrounding six electrical vaults, the former hazardous materials storage room, and former restrooms, and (3) the door operating mechanisms. Post-NTCRA inspections, sample collection, and analysis activities are described below.

### *2.1 Condition of Hangar 1 and the Implemented NTCRA Remedy*

During pre-lease negotiations between NASA and PV, NASA indicated that the CM15 epoxy coating had deteriorated in several areas. As a result, PV commissioned ACC Environmental Consultants (ACC) to perform a visual screening of the CM15 epoxy coating and collect wipe and bulk samples of remaining building materials within the Hangar 1 structure to:

(1) Document existing conditions at Hangar 1;

(2) Evaluate the integrity of the CM15 epoxy coating and assess the effectiveness of the implemented remedy; and,

(3) Identify potential health and safety precautions that may be necessary during construction and renovation activities as well as potential long-term occupancy-related activities associated with the reuse of Hangar 1 structure due to the residual PCBs and lead.

The results of the visual inspection and sampling are presented in ACC's *PCB, Lead, and Asbestos Sampling Report*, dated February 24, 2015 (ACC, 2015) and summarized briefly below. While ACC's visual inspection encompassed the ground and mezzanine levels and roof-top catwalk area, ACC's sampling was limited to areas that could be accessed from the ground and mezzanine levels of Hangar 1.

#### 2.1.1 *Visual Inspection*

During the visual inspections conducted on April 7, 2014 and June 24, 2014, ACC observed four general types of coating issues related to the CM15 epoxy coating:

(1) Isolated coating failure, where the encapsulant had delaminated from the existing substrate;

(2) Encapsulant deterioration around edges due to separation of the underlying substrate from the structure (e.g., peeling paint);

(3) Evidence of rust-related breakthrough of the coatings; and,

(4) Missing or thinly applied coatings.

Based on visual observations on the ground floor and mezzanine levels of Hangar 1, ACC typically observed between approximately 50 and 100 square feet of delaminated or deteriorated coatings per major truss section; extrapolating these observations over the ground floor and mezzanine levels of the entire Hangar 1 structure, ACC estimates between 2,600 and 5,200 square feet of deteriorated coatings. The majority of the deteriorated conditions appeared to be in areas where the original coatings were loose and peeling. While the largest area of deteriorated coatings was approximately 8 to 10 square feet in size, most of the deteriorated areas were less than 3 square feet in size.

During ACC's inspection of the structural steel coating visible from roof-top catwalk area, ACC observed between approximately 20 and 40 square feet of rust-related breakthrough of the coatings on each side of the catwalk area within each major truss section of the hangar, for an estimated 500 to 1,100 square feet of deteriorated coatings.

### 2.1.2 *Wipe Sampling*

On April 21, 2014 and August 6, 7, 11, and 13, 2014, ACC collected wipe samples from accessible exposed surfaces within Hangar 1 to assess potential exposure levels due to degradation of the CM15 epoxy coating. A total of 31 wipe samples were collected within the Hangar 1 structure during April 2014 and 21 additional wipe samples were collected during August 2014. The wipe samples were analyzed for Aroclor 1254, Aroclor 1260, Aroclor 1268, and lead and other CAM 17 metals.

PCB concentrations in the wipe samples collected during the April 2014 sampling event were all below the analytical reporting limit (0.005 micrograms per 100 square centimeters (ug/100 $cm^2$)) and the NTCRA target criterion of 10 ug/100 $cm^2$. These results were confirmed by the August 2014 wipe sampling results, with the exception of the wipe samples that were collected from the concrete floor (4 wipe samples) and horizontal structural steel members (8 wipe samples) where there was evidence of ponding and/or sediment accumulation.. In the four August 2014 wipe samples collected from the concrete floor in areas where there was evidence of ponding and sediment accumulation, Aroclor 1268 was detected at concentrations ranging between approximately 100 ug/100 $cm^2$ and 740 ug/100 $cm^2$. Of the eight wipe samples collected from horizontal structural steel members with ponded water during the August 2014 sampling event, Aroclor 1268 was detected in one sample at a concentration of 0.78 ug/100 $cm^2$.

Lead was detected in all of the wipe samples collected during the April and August 2014 sampling events at concentrations ranging between 2.5 micrograms per square foot (ug/$ft^2$) and 30,000 ug/$ft^2$. At 7 of the 8 different structural elements sampled, the geometric mean lead concentration[3] for each structural element tested exceeded the NTCRA target criterion of 40 ug/$ft^2$ and at 3 of the 8 structural elements sampled, the geometric mean lead concentration exceeded NASA's Lead Management Plan criterion of 400 ug/$ft^2$. While other CAM 17 metals were detected in the wipe samples collected during the April and August 2014 sampling events, there are no established regulatory guidelines for acceptable levels of these metals.

During the August 2014 sampling event, ACC also collected 12 wipe samples from unpainted surfaces upwind and downwind of the Hangar 1 structure (6 upwind and 6 downwind) to assess whether the Lead and PCBs observed within the Hangar 1 structure were potentially from an off-site source. As PCBs were not detected above the analytical reporting limit in any of upwind or downwind samples and the observed lead concentrations were relatively low, ACC concluded that it was unlikely that the PCBs and lead observed in the wipe samples collected within the Hangar 1 structure were from an off-site source. In addition, as the Hangar 1 structure, concrete floors, and storm water conveyance trenches and drains were cleaned as part of the NTCRA completion efforts (AMEC, 2013), the assumed source of the PCBs and lead detected in the August 2014 wipe samples from inside the Hangar 1 structure is from degradation of the CM15 epoxy coating.

### 2.1.3 *Air Sampling*

For comparison purposes with the Hangar 1 wipe sampling results, air samples were collected from upwind, downwind, and within the Hangar 1 structure on August 6, 7, and 11, 2014 at the request of NASA, the

[3] The geometric mean lead concentration for each type of structural element was calculated using all of the wipe samples collected from that type of structural element (e.g., the geometric mean lead concentration for the concrete floor is the geometric mean of the lead concentrations detected in the 8 wipe samples collected from the concrete floor).

USEPA, and Water Board. None of the samples contained concentrations of lead or other CAM 17 metals above the laboratory reporting limits.

#### 2.1.4 *Building Material Sampling*

On August 11 and 13, 2014, ACC also collected samples of remaining materials (e.g., concrete, paint chips, surficial sediments, etc.) within the Hangar 1 structure to:

(1) Assess the remaining concentrations of PCBs, metals, and asbestos in existing coatings that may be disturbed as part of future renovation, abatement, or operations and maintenance activities;

(2) Assess whether building material chemicals of concern in areas of delaminated or deteriorated coatings may have re-contaminated previously cleaned stem walls and concrete slabs; and,

(3) Identify other potential chemicals of concern.

A total of 34 samples were collected from 16 different building materials within the Hangar 1 structure. In these samples:

- PCBs were detected in 26 of the 34 samples collected and analyzed for PCBs, at concentrations ranging from approximately 0.7 mg/kg to 1,900 mg/kg. Of the 15 building materials analyzed for PCBs, PCBs were detected in all but one material;[4]
- lead was detected in all 34 samples of 14 of the building materials analyzed for lead at concentrations ranging between approximately 39 mg/kg and 250,000 mg/kg;
- antimony, arsenic, chromium, and mercury were detected in one or more of the 34 bulk material samples at concentrations above applicable screening levels for soil under a Commercial/Industrial Land Use scenario; and,
- asbestos was detected in one of the 16 samples obtained of materials identified as suspect to contain asbestos. This material was described as a residual dark brown adhesive.

On completion of the building material sampling, all building material sampling locations were recoated with CM15 epoxy coating by a qualified contractor and inspected by NASA and a third party qualified coating inspector.

In addition to the samples collected from building materials, ACC also collected two samples of surficial sediments from trenches and/or trench drains within the Hangar 1 structure. In these samples, PCBs were detected at concentrations of 12 mg/kg and 36 mg/kg and lead was detected at concentrations of 1,300 mg/kg and 12,000 mg/kg.

Based on the visual observations and the wipe, building material, and surficial sediment sampling results, ACC concluded that the presence of PCBs and lead in the existing building materials and deterioration of the CM15 epoxy coating are the likely source of the lead observed in the wipe sampling and the PCBs and lead in the surficial sediments within the Hangar 1 structure. The report also concluded that repair of the encapsulating CM15 epoxy coating is necessary and that due to its exposure to the weather, the CM15 epoxy coating should be inspected more frequently until the Hangar 1 structure is re-skinned and the coating is protected from the elements (ACC, 2015).

[4] PCBs were not detected in the one sample of oil-soaked poly tape that was collected from one of the northeast Hangar door trucks (ACC, 2015).

## 3. Pilot Scale Abatement Study Planning

As described above in Section 1.2, the primary objective of the pilot scale abatement study is to determine the feasibility of reducing PCB and lead concentrations in accessible[5] structural steel elements and CMU walls within the Hangar 1 structure to:

1) Concentrations that could potentially eliminate the need for institutional or engineering controls at Hangar 1;
2) Minimize the potential exposure of future workers to PCBs and lead during long-term operations and maintenance activities; and,
3) Minimize the need for institutional controls such as the installation and maintenance of signage regarding the potential exposure hazard and the necessity of obtaining regulatory agency approval of building modifications that might damage remedy components.

The data and observations gained from this Pilot Abatement Study will aid in determining appropriate means and methods to achieve the overall project goal of the safe removal of accessible PCB- and lead-impacted coatings from the Hangar 1 structure. As discussed in Section 4.2.3, three different blasting technologies will be assessed as part of this Pilot Study.

All work will be conducted in accordance with applicable federal, state, and local government laws and regulations including, but not limited to, those of the USEPA, the Occupational Safety and Health Administration (OSHA), the State of California Division of Occupational Safety and Health (DOSH), the State of California Department of Industrial Relations (DIR), the State of California Department of Public Health (CDPH), Water Board, the recommendations of National Institute of Occupational Safety and Health (NIOSH). Whenever there is a conflict or overlap in the aforementioned references, the most stringent requirement will be used.

This section describes:

- Communication pathways (Section 3.1);
- Qualifications and training (Section 3.2);
- Pilot Study tasks and the anticipated schedule (Section 3.3);
- Selection of the Pilot Scale Abatement Study Area (Section 3.4);
- Target Post-Abatement Acceptance Criteria (Section 3.5);
- Airborne Action Levels (Section 3.6);
- Planned sample collection activities, procedures, and field quality control activities (Section 3.7);
- Proposed analytical laboratories and analytical methods (Section 3.8); and,
- Quality assurance and quality control protocols that will be implemented during the Pilot Study (Section 3.9).

[5] Accessible surfaces are all surfaces throughout the Hangar 1 structure that are not tight-tolerance mated surfaces (e.g., steel members riveted together) and will include all mated surfaces with greater than a ½" gap. Caulk or other coatings will be removed prior to determining whether a surface is a tight-tolerance mated surface.

### *3.1 Communication Pathways*

Project roles and communication pathways are listed below; one individual may take on more than one role.

- Owner: The Owner, the National Aeronautics and Space Administration (NASA), has ultimate responsibility for the Hangar 1 project.
- Master Lessee: Planetary Ventures, LLC (PV), the Master Lessee, is providing financing for the Hangar 1 project. PV is responsible for communication with Owner and Regulatory Agencies. Currently Kevin Antonelli represents PV.
- General Contractor: DPR is the General Contractor and will communicate project status and potential concerns during project implementation. DPR will communicate directly with the Master Lessee's Project Coordinator.
- Project Coordinator: This is the Master Lessee's representative overseeing the planning and implementation of the project. Currently, Andy Meade is PV's designated Project Coordinator. The Project Coordinator has been given authority from PV for communicating with Owner, Master Lessee, General Contractor, Project Managers/Field Team Leaders, and Regulatory Agencies.
- Project Managers/Field Team Leaders: ACC has been engaged by the Master Lessee to develop and implement the Pilot Scale Abatement Work Plan and is responsible for managing field staff and assuring that field activities meet quality objectives. ACC communicates with Master Lessee, General Contractor, and Project Coordinator.
- Field Staff: Implements field tasks and communicates with their supervisors. The field staff includes ACC personnel and Eco Bay Environmental Services, Inc. (Eco Bay; Abatement Contractor) personnel and sub-contractors; ACC personnel will be conducting all analytical sampling prior to, during, and after abatement activities (with the exception of the Abatement Contractor's personal air sampling), performing inspections of containments, reviewing contractor submittals, observing contractor activities from mobilization, during the abatement activities through contractor de-mobilization and managing data in the field. Eco Bay personnel will construct and manage the containment, and will conduct all abatement and decontamination activities within the Pilot Study area including restoration of the area post abatement.
- Quality Assurance Manager (QAM): Monitors the implementation of this Work Plan, coordinates audits and data validation, and reviews deliverables. The QAM interfaces with the analytical laboratory and guides field sampling, data verification, and data validation. Jill Henes will be the QAM for the project.
- Data Manager: Manages incoming data and the Site database, and determines whether data deliverables meet project specifications. Heather Sobky (ACC) will be the Data Manager for the project.
- Analytical Laboratory Director: Implements the control system allowing sample possession to be traced from the laboratory's receipt to final disposition.
- Project Health and Safety Coordinator: Verifies that each contractor and subcontractor has site specific Health and Safety Plans and addenda and monitors health and safety activities. The General Contractor DPR will verify the SSHSP for each party performing work at the site.
- Regulatory Agencies: The USEPA's and the Water Board's Remedial Project Managers, Judy Huang

and Elizabeth Wells, respectively, will review the results of the work.

- Data Validation/Quality Assurance Consultant: Verifies and Validates Stage 2B (EPA, 2009) data packages and assigns appropriate qualification codes to analytical results. Provides Quality Assurance assistance when needed. The Data Validation/Quality Assurance Consultant for the Pilot Scale Abatement Study is Jill Henes.

### *3.2 Qualifications and Related Training for Contractors and Subcontractors*

Strict Safety and Security requirements apply at this job site. All Abatement Contractor, Sub-contractors and Consultant personnel shall attend site-specific safety training provided by the General Contractor (DPR). DPR manages access and orientation to the site to all site users and has responsibility for monitoring all visitors to the site. ACC does not anticipate visitors to the site who are not presently oriented and qualified to be present. The Abatement Contractor (Eco Bay) bears the responsibility for ensuring that all Contractor and sub-contractor staff are appropriately trained and equipped to perform the work described in this work plan.[6]

Table 1, Table 2, and Table 3 provide details regarding the qualifications for the Consultant and all Contractor and Sub-contractors who will be working on this project.

[6] Fall Protection Plan - Contractor must adhere to the DPR Site Specific Fall Protection Plan if working above the height of 6 feet.

Table 1: List of Contractors with Qualifications by Division of Work

| Division of Work | Contractor | Qualifications | License/Certification Number |
|---|---|---|---|
| Consultant | ACC Environmental Consultants, Inc. | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor/Monitor<br>40 Hour – HAZWOPER | N/A |
| Abatement Work | Eco Bay Environmental Services, Inc. | Classifications:<br>A—General Engineering<br>B—General Contracting<br>C-2—Insulation<br>C-21—Demolition<br>ASB—Asbestos Abatement<br>HAZ—Hazardous Waste | CA State Lic. Board No. 912328 |
| Scaffold | SAFWAY Services, LLC | D-39 Scaffold | CA State Lic. Board No. 972968 |
| Re-Coating | C & O Painting | C-33 Painting | CA State Lic. Board No. 501260 |
| Re-coating Inspector | CTS-Construction Testing Services | AWS<br>- Prestressed Concrete Special Inspector<br>- Reinforced Concrete Special Inspector<br>- Structural Masonry Special Inspector<br>- Structural Steel and Welding Special Inspector<br>- Structural Steel and Bolting Special Inspector<br>- Structural Welding Special Inspector<br>NACE<br>- Coating Inspector (Levels 1 and 2)<br>ACI<br>- Concrete Field Testing Technician – Grade I | 10120931<br><br><br><br><br><br><br><br><br>32579<br><br>01029398 |
| Waste Hauler | World Environmental & Energy | N/A | EPA ID Number: CAR000037283 |
| Waste Disposal Site | Clean Harbors Buttonwillow Landfill | N/A | EPA ID # CAD980675276 |
| Data Validation/Quality Assurance Consultant | JBHenes | BA/BS degree in Chemistry or equivalent<br>10 years of Environmental Quality Assurance | N/A |

Abbreviations:
N/A: not applicable

Consultant:  ACC Environmental Consultants, Inc. is responsible for monitoring and oversight of the Pilot Study and will be on site to perform inspections, all analytical sampling (air, wipe, bulk and waste), with the exception of the Abatement Contractor's personal air sampling and oversight of contractor performance prior to, during and post abatement work activities. ACC will also perform data usability assessments and write final report of findings. ACC employees on site will have the following qualifications specific to the contaminants of concern:

Table 2: Consultant Personnel Qualifications and Requirements

| Employee Designation | Qualification | Compliance Requirements |
|---|---|---|
| Project Manager | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor/Monitor<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq.<br>Cal-OSHA – 8 CCR 5192 |
| Project Geologist | 40 Hour – HAZWOPER | Cal-OSHA – 8 CCR 5192 |
| Sr. Site Technician(s): | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq.<br>Cal-OSHA – 8 CCR 5192 |
| Site Technician | DOSH: Certified Site Surveillance Technician<br>CDPH: Lead Supervisor or Sampling Technician<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq.<br>Cal-OSHA – 8 CCR 5192 |

Eco Bay Services Inc. (Abatement Contractor) employees, who will engage in blasting, cleaning, personal air sample collection and project supervision activities will have the following qualifications specific to the contaminants of concern:

Table 3: Abatement Contractor Personnel Qualifications and Requirements

| Employee Description | Qualification | Compliance Requirements |
|---|---|---|
| Project Supervisor: | AHERA Asbestos Contractor/Supervisor<br>CDPH – Lead Supervisor<br>40 Hour – HAZWOPER | 8 CCR 1529<br>CDPH – 17 CCR 35001 et seq. and Cal-OSHA 8 CCR 1532.1<br>Cal-OSHA – 8 CCR 5192 |
| Laborer | AHERA Asbestos Worker<br>CDPH – Lead Worker<br>40 Hour – HAZWOPER | 8 CCR 1529<br>CDPH – 17 CCR 35001 et seq. and Cal-OSHA 8 CCR 1532.1<br>Cal-OSHA – 8 CCR 5192 |

### *3.3 Project Tasks and Schedule*

Identified project tasks and the anticipated schedule are presented below in Table 4. It is anticipated that the entire Pilot Study will take approximately 25 working days.

Table 4: Project Tasks and Anticipated Schedule

| Task | Description | Start Date (a) | End Date (a) | Approximate Duration |
|---|---|---|---|---|
| | Pre-Abatement Activities | | | |
| Pre-Construction Meeting | At least one meeting will be held between the Contractors (Eco Bay and DPR), and Consultants (ACC and EKI) in advance of Eco Bay's Mobilization to the site. The meeting will review project scope, coordinate lines of communication, define roles and responsibilities of each party and align contractor and consultant activities to achieve the goals of the project. | To be scheduled Upon receipt of Agency Approval | N/A | 1 day |
| Pre-abatement Equipment Sampling | ACC will collect samples from equipment that will be used onsite and expedite their analysis. | N/A | N/A | 3 – 5 days |
| Contractor Mobilization | Contractor will mobilize personnel and all necessary equipment to the site. | 1 | 2 | 2 days |
| Pre-abatement cleaning | Remove debris and surficial sediments from the Pilot Study Area. The debris and surficial sediments will be collected using vacuums equipped with HEPA filters, stored in drums, and characterized for disposal. | 2 | 3 | 1 – 2 days |
| Pre-Abatement Enclosure Setup | Setup of a fully encapsulated enclosure around the Pilot Study Area. Shrink wrap polyethylene will be used for the enclosure roof and siding and 50 mil polyvinyl chloride will be used for the enclosure floor. | 2 | 6 | 5 days |
| | Baseline Sampling | | | |
| XRF Screening | Survey lead concentrations in existing painted structural steel members and CMU walls that will be abated during the Pilot Study. | 7 | 7 | 1 day |
| Baseline Sampling | Collection of wipe samples from the concrete floor (b) and bulk material samples (i.e., paint/coating chips, CMU wall, soil, and sediment) within, and immediately outside, the Pilot Study Area. | 7 | 8 | 2 days |
| Perimeter Air Monitoring and Sampling | Collection of upwind and downwind perimeter air samples for lead, copper, and PCBs and monitoring upwind and downwind particulate aerosol concentrations using direct-read aerosol monitors. | 7 | 9 | 3 days |
| Cover Exposed Soil | Cover exposed soil outside the Pilot Study area with visqueen. | 9 | 9 | 1 day |
| | Abatement Activities | | | |
| Enclosure | Install Air Filtration Devices (AFDs) equipped with HEPA filtration to establish negative-pressure enclosure. | 7 | 8 | 1 day |
| Enclosure Inspections | Inspect critical areas (e.g., seams and areas where blasting media and/or water accumulate) of the enclosure and monitor the differential pressure between the interior of the enclosure and ambient air. Inspections will be conducted, at a minimum, at the start and end of each shift, and twice during each shift. | 8 | 20 | 13 days |
| Perimeter Air Monitoring and Sampling (c) | Collection of upwind and downwind perimeter air samples for lead, copper (d), and PCBs and monitoring upwind and downwind particulate aerosol concentrations using direct-read aerosol monitors. | 10 | 20 | 11 days |

| Task | Description | Start Date (a) | End Date (a) | Approximate Duration |
|---|---|---|---|---|
| Abatement Activities and Methods | In general, it is anticipated each of the blasting technologies listed below will be used to remove coatings from the designated test areas during a single day. The spent media and removed coatings will be collected vacuums equipped with HEPA filters and the abated areas will be wet wiped.<br><br><u>Abatement Methods</u><br>1) Media Blasting – Abate select areas using copper slag abrasive media.<br>2) Ultra-High-Pressure Water Blasting – Abate select areas using ultra-high-pressure water.<br>3) Vapor-Media Blasting – Abate select areas using aerosolized water and ultrafine garnet abrasive media | 10 | 15 | 6 days |
| Inspections and Sampling | Following each abatement method and decontamination, verification wipe sampling, bulk CMU wall sampling, and XRF screening will be conducted in the abated area. | 11 | 15 | Concurrent with Abatement Activities and Methods Task |
| Waste Handling | Spent media, removed coatings, and other decontamination wastes will be placed in 55-gallon metal drums and characterized for disposal. | 10 | 20 | 11 days |
| Additional Abatement (if required) | In the event that the abated structural elements do not meet the Post-Abatement Acceptance Criteria, the area will be re-abated. After decontamination of the re-abated area, verification wipe sampling will be conducted in the re-abated areas. After the re-abatement activities, the area will be recoated with CM15 epoxy coating. | 16 | 20 | 0 – 5 days |
| <u>Post-Abatement Activities</u> | | | | |
| Decontamination | Final decontamination of enclosure and equipment such as scaffolding within the enclosure. Decontamination materials and wipe testing of equipment (e.g., scaffolding) used within enclosure. | 21 | 22 | 2 days |
| Bulk Material Sampling | Collection of post-abatement soil samples, if needed (e). | 22 | 23 | 0 – 1 day |
| Removal of Enclosure | Removal of enclosure from Pilot Study Area. | 22 | 23 | 2 days |
| Recoating and Inspection | Recoating of all abated structural elements with CM15 epoxy coating. On completion of recoating activities, an independent third party qualified coating inspector will inspect the coatings. | 23 | 24 | 2 days |
| Waste Characterization | Waste characterization samples will be collected from all remaining uncharacterized wastes generated during the Pilot Study. | 24 | 25 | 2 days |
| Evaluate Data | Evaluate analytical data, amounts of materials used, waste generated, ease of use, water use, safety considerations, and required maintenance activities in relation to the Pilot Study Objectives (Section 1.2). | N/A | N/A | See Section 5 |

<u>Notes:</u>
(a) Anticipated dates represent working days beyond the start of the project.
(b) Wipe sampling of the concrete floor will occur after pre-abatement cleaning and before setup of the abatement enclosure. It is anticipated this sampling will occur on Day 3.

(c) Perimeter air monitoring and sampling will only be conducted on the days that blasting activities are conducted (i.e., days 10, 12, 14 and as needed for additional abatement between days 16 and 20).
(d) Perimeter air samples will only be analyzed for copper on the day that media blasting with copper slag is conducted (i.e., day 10 and as needed for additional abatement between days 16 and 20).
(e) Post-abatement soil samples will only be collected if a breach in containment is observed during abatement activities.

Abbreviations:

CMU: concrete masonry unit
HEPA: high-efficiency particulate air
N/A: not applicable
PCB: polychlorinated biphenyl
XRF: x-ray fluorescence spectrometer

### *3.4 Pilot Scale Abatement Study Area*

The proposed pilot scale abatement study area (Pilot Study Area) is located in the southeastern corner of Hangar 1 (Figure 2A, Figure 2B, and Figure 2C) and is approximately 120 feet long by 30 feet wide by 20 feet high. Prior to the selection of this area for the proposed Pilot Study, ACC visually inspected the area for potential asbestos-containing materials and did not observe any.[7] A trench drain is located along the eastern edge of the study area and small area of bare soil is present east/southeast of the Pilot Study Area (Figure 2A).

The Pilot Study Area was selected as it contains a number of different structural steel elements and CMU walls and is considered representative of the entire Hangar 1 structure. Figure 3A, Figure 3B, and Figure 3D illustrate the different types of interior and exterior structural steel elements and Figure 3C illustrates the subject CMU wall.

### *3.5 Target Post-Abatement Acceptance Criteria*

Post-abatement acceptance criteria for PCBs and lead in wipe samples are listed in Table 5.

[7] The only known asbestos containing material remaining in Hangar 1 is described as approximately 40 lineal feet of dark brown adhesive materials located on a concrete wall at column line 2.5 which is to the east of the Pilot Study Area and will not be impacted by the work. No suspect asbestos materials are present in the Pilot Study Area. During the Pilot Study, if materials are encountered that are suspected of containing asbestos, work will be stopped and the suspect materials sampled and analyzed for asbestos. If asbestos is determined to be present in these materials, the materials will be removed in accordance with applicable laws and regulations before the continuation of abatement activities within the Pilot Study Area.

Table 5: Target Post-Abatement Acceptance Criteria

| Compound of Concern | Applicable Surface | Sample Type | Acceptance Criteria | Source |
|---|---|---|---|---|
| PCBs | Non-porous surfaces (a) | Wipe | ≤10 ug/100 $cm^2$ | NTCRA Project Action Limit / 40 CFR 761.61 |
| | CMU Wall | Wipe | ≤10 ug/100 $cm^2$ | NTCRA Project Action Limit / 40 CFR 761.61 |
| | CMU Wall | Bulk | (b) | -- |
| | Soil (c) | Bulk | 1 mg/kg or no greater than pre-abatement background levels. | NTCRA Project Action Limit / 40 CFR 761.61 |
| Lead | Interior floor surfaces (d) | Wipe | <40 ug/$ft^2$ | NTCRA Project Action Limit / |
| | Building surfaces with high potential for hand-to-mouth contact (d) | Wipe | <40 ug/$ft^2$ | NASA Lead Management Plan (APR 8715.1) |
| | Interior horizontal surfaces | Wipe | <250 ug/$ft^2$ | NTCRA Project Action Limit Title 17, CCR, Division 1, Chapter 8, §35035 (b) |
| | CMU Wall | Wipe | <40 ug/$ft^2$ | NASA Lead Management Plan (APR 8715.1) |
| | CMU Wall | Bulk | (b) | -- |
| | Soil (c) | Bulk | 320 mg/kg or no greater than pre-abatement background levels. | MFA Leasehold Target Concentration Level |
| | Equipment | Wipe | <40 ug/$ft^2$ | NASA Lead Management Plan (APR 8715.1) |

Notes:
(a) Surface cleanup standard for PCBs on non-porous surfaces located in high occupancy areas (unrestricted use); this standard will also be used as the Acceptance Criteria for all equipment used during abatement activities.
(b) An Acceptance Criteria for bulk CMU wall (i.e., a porous material) materials will be determined upon completion of the Pilot Study.
(c) Post-abatement soil samples will only be collected if a breach in containment is observed during abatement activities.
(d) See footnote 7.

For the purpose of this Pilot Study, the proposed Target Post-Abatement Acceptance Criteria are ≤10 ug/100 $cm^2$ for PCBs and <250 ug/$ft^2$ for lead.[8] These Target Post-Abatement Acceptance Criteria are not proposed as final Post-Abatement Acceptance Criteria for the entire Hangar 1 structure abatement project, but instead

[8] The interior floor surfaces acceptance criteria for lead (<40 ug/$ft^2$) was not considered applicable to the Pilot Study as the concrete floor will be covered by an enclosure (see Section 3.6.2.4) and no abatement activities will be performed on the concrete floor. The building surfaces with high potential for hand-to-mouth contact acceptance criteria for lead (<40 ug/$ft^2$) was also not considered applicable to the Pilot Study as none of the abated surfaces will have a high potential for hand-to-mouth contact. The interior horizontal surfaces acceptance criteria for lead (<250 ug/$ft^2$) is proposed for this Pilot Study as this is the acceptance criteria used in the NTCRA and is more conservative than the NASA Lead Management Plan clearance level (<400 ug/$ft^2$).

as criteria that will be used to evaluate the effectiveness of the abatement techniques proposed in this Pilot Study by determining which method will be able to achieve the best results most efficiently. Final Post-Abatement Acceptance Criteria will be developed prior to the initiation of full-scale abatement activities.

### *3.6 Airborne Action Levels*

The Airborne Action Levels for PM10, Total Dust, PCBs, lead and copper are summarized in Table 6. Additional detail regarding the selection of these Airborne Action Levels is provided below.

Table 6: Airborne Action Levels

| Chemical of Concern | Airborne Action Level |
|---|---|
| PM10 | 0.11 mg/m$^3$ (8-hour time weighted average) |
| PCBs | 0.013 mg/m$^3$ (8-hour time weighted average) |
| Lead | 0.001 mg/m$^3$ (8-hour time weighted average) |
| Copper | None |

Abbreviations:
PM10: particulate matter with an effective diameter of 10 microns or less
mg/m$^3$: milligrams per cubic meter

*PM10 Dust:* The California Ambient Air Quality Standard (CAAQS) for PM10 dust is 0.050 milligrams per meter cubed (mg/m$^3$), measured as a 24-hour time-weighted average (TWA). An 8-hour time-weighted average action level can be calculated for PM10 by adjusting the 24-hour CAAQS using the background PM10 levels for the 16 non-working hours of the day. More specifically, the average annual PM10 concentration measured at the nearest Bay Area Air Quality District (BAAQMD) PM10 monitoring station (San Jose, California) was 0.0199 mg/m$^3$ in 2014 (BAAQMD, 2015). Assuming that this concentration is the background concentration present at the Site for 16 hours while abatement activities are not being conducted, a PM10 Action Level during the 8-hour workday can be calculated from the equation below.

$$PM10_{8\text{-hour-TWA}} = \frac{24 \times PM10_{24\text{-hour-TWA}} - 16 \times PM10_{background}}{8}$$

where $PM10_{8\text{-hour-TWA}}$ = 8-hour TWA PM10 for an 8-hour shift (mg/m$^3$),

$PM10_{24\text{-hour-TWA}}$ = 24-hour TWA PM10 (0.050 mg/m$^3$), and

$PM10_{background}$ = Background PM10 (0.0199 mg/m$^3$).

The resulting Action Level for PM10 during abatement activities (based on an 8-hour workday) is approximately 0.11 mg/m$^3$.

*Lead:* The CAAQS for airborne lead of 0.0015 mg/m$^3$ is based on a 30-day TWA and is not directly applicable for short-term airborne concentrations. Instead, BAAQMD regulates airborne lead concentrations at ground level at a limit of 0.001 mg/m$^3$ based on a 24-hour TWA (BAAQMD Rule 11-1-302); BAAQMD Rule 11-1-302 does not require any foreknowledge of background lead concentrations at the site. To be conservative, an 8-

hour TWA concentration of 0.001 mg/m$^3$ will be used as the Action Level for airborne lead during the Pilot Study.

*PCBs:* As there are no CAAQS or BAAQMD regulatory limits for airborne PCBs, a risk-based Action Level of 0.013 mg/m$^3$ was calculated for PCBs to protect nearby workers from potential exposure to PCBs during this pilot study (Attachment C). This airborne Action Level is health protective because these concentrations would have to be sustained at the location of the exposed receptors for the entire duration of the Pilot Study to yield a Hazard Index of 1 or an incremental lifetime cancer risk of $10^{-6}$, which is highly unlikely.[9]

*Copper:* There are no CAAQS or BAAQMD regulatory limits for airborne copper. Due to the low toxicity of copper and the limited number of days copper slag will be used during the Pilot Study, no airborne Action Level for copper will be developed. Instead, during each day of perimeter air monitoring, downwind sampling results will be compared to upwind and background sampling results to determine whether additional mitigation measures are necessary.[10]

### *3.7 Planned Sample Collection Activities*

The sample collection activities described below have been designed to assess: (1) effective means and methods to remove the coatings of the building superstructure, (2) assess coating removal technologies and engineering controls that will minimize the exposure of abatement personnel to Site chemicals of concern (COCs) and the potential for fugitive emissions at the site perimeter, (3) evaluate the exposure of potential receptors at the site perimeter during the removal of impacted coatings, and (4) evaluate the characteristics of waste streams generated during the Pilot Study to determine appropriate disposal options. Specific sample collection activities and their respective goals include:

1. Measurement of pre-abatement baseline conditions for PCB and lead concentrations on the structural components (structural steel members, CMU walls, and the concrete slab) of Hanger 1 as well as in soil near Hangar 1 and the collection of post-abatement data to determine the concentrations of COCs remaining on the structural components. These data will be used to evaluate effectiveness of the three different abatement techniques. An X-Ray Fluorescence Spectrometer (XRF) will be used to conduct a survey of lead to better define initial sampling locations.
2. Collection and analysis of airborne particulate samples during abatement activities to evaluate potential worker exposures and to aid in identifying and establishing effective engineering controls and an air-monitoring program for workers during full-scale abatement and coating removal activities.
3. Monitoring of fugitive dust emissions and/or particulates in ambient air at the site perimeter for fugitive dust emissions to evaluate the effectiveness of implemented engineering controls and the potential need to implement additional engineering controls or modify abatement blasting procedures to meet the project Airborne Action Levels (Section 3.6).
4. Collection of waste characterization data to enable the appropriate disposal of wastes created during the Pilot Study and aid in determining the waste classification and quantities that are likely to be generated by the full-scale abatement project.

[9] If the abatement project is implemented at full-scale, the PCB Action Level would have to be adjusted accordingly to be protective of nearby workers during the full-scale project.

[10] If media blasting with copper slag is selected for the full-scale abatement of Hangar 1, a risk-based Action Level for copper will be calculated before implementation of the full-scale project.

### 3.7.1 *Sampling Rationale*

Samples collected during this project will include baseline soil/sediment, surface and coating samples collected prior to abatement, air samples during abatement activities, and confirmation wipe samples collected once the abatement action is completed. Details on the sampling design and rationale are presented below and are described in detail in Sections 3.7 and 4.2 and Tables 5, 6, 9 and 10.

The outcomes of sampling will provide data that will assist in determining if the primary objectives of the Pilot Study are met, as described earlier in Section 1.2 of this work plan. The data provided by sampling conducted prior to, during and post abatement activities will provide quantitative information that will assist ACC and others to answer critical questions prior to the design of full abatement of the structure.

#### 3.7.1.1 Pre-Abatement Sampling:

Baseline sample collection, as discussed in Section 4.1.2, and Table 9 below, will be conducted to determine pre-abatement PCB and lead concentrations on the concrete foundation, the structural steel and CMU walls within the Pilot Study Area, and in soil directly adjacent and to the south of the Pilot Study Area.

*Wipe Samples*

Because the preparation steps for the analysis of PCBs and lead in wipe samples are different, 2 separate wipe samples must be collected at each location. Thus the total number of wipe samples is twice the number of sample locations.

Wipe samples will be collected from the concrete floor at 4 locations within the proposed work area corresponding to locations where the abatement techniques will be tested. Two (2) samples, one for PCBs and lead will be collected from each location using the method prescribed in Section 3.7.3.1, for a total of eight (8) samples. In addition, (2) duplicate samples will be collected from the area and analyzed separately for PCBs and lead. Additional wipe samples will be collected from the concrete floor in four (4) locations outside the proposed work area. Two (2) samples, one for PCBs and one for lead will be collected from each location using the method prescribed in Section 3.7.3.1., for a total of eight (8) samples. The approximate locations of these samples are shown on Figure 3E.

The purpose of this sampling is to determine pre-Pilot Study PCB and lead concentrations on this media for comparison with PCB and lead concentrations on this media after abatement activities have concluded and the enclosure has been removed.

Six (6) wipe samples (plus 2 duplicate wipes) will be collected from the Concrete Drainage Trench at three (3) locations directly adjacent on the south side of the containment, as described in Section 4.1.2 and Table 9; these samples will be analyzed separately for PCBs and metals and the approximate locations of these samples are shown on Figure 3E. Two (2) wipe samples will be collected and analyzed for PCBs and metals from one (1) additional location within the Concrete Drainage Trench from an area that is generally downwind of the area that will be abated; this sample is also located adjacent to an area of exposed soil. Post-abatement wipe sampling of the Concrete Drainage Trench will only occur if there is a breach in containment on the side that this feature is located.

In addition, before being mobilized to the site, a total of six (6) wipe samples (plus 2 duplicates) will be collected from three (3) locations on the scaffolding that will be used during abatement activities and a total of twelve (12) wipe samples (plus six (6) duplicates) will be collected from the blasting equipment (two locations on each of the three types of blasting equipment will be sampled) prior to the use of this equipment at the

Site. These samples will be analyzed for PCBs and lead, separately, to determine pre-Pilot Study concentrations of contaminants of concern on this equipment.

*Bulk Samples*

The data provided by bulk sample results allows concentrations of COC's in the Pilot Study Area to be compared to historical data of the same or similar building elements in order to establish that the elements abated in the Pilot Study area are representative of the rest of the structure, and increase confidence that the overall data achieved by this study will be applicable to future abatement considerations of the whole structure.

Coatings: Three (3) coating samples (plus 1 duplicate) will be collected from each of the structural steel elements (i.e., the Exterior Steel Members, the Structural Support Steel Members, and the Steel Members under the Mezzanine) and from the CMU Wall that will be abated during the Pilot Study; in total, 12 coatings samples (and 4 duplicate samples) will be collected (see Section 4.1.2 and Table 9). These samples will be analyzed for PCBs and lead.

Bulk CMU Wall Samples: ACC proposes to collect representative shallow bulk samples of the existing CMU wall within the Pilot Study Area for the analysis of lead and PCBs to determine whether PCB and/or lead may have leached from historical coatings into the CMU wall; the CMU wall is considered representative of other shallow porous surfaces within the Hangar 1 structure. The bulk samples taken from the CMU walls will also assist in determining whether these elements will need to be coated, post-abatement, or potentially removed and replaced. A total of three (3) (plus 1 duplicate) will be collected from the CMU wall and analyzed for PCBs and lead, as described in Section 4.1.2 and Table 9.

*Soil Samples*

While the surface soils outside the Hangar 1 structure were remediated as part of the NTCRA, field observations and analytical data collected within the Hangar 1 structure (ACC, 2015) suggest that exposure to the elements and other factors may have degraded the protectiveness of the implemented removal action and that PCBs and lead are present in surficial sediments collected within the Hangar 1 structure; at present it is not known whether the soil adjacent to Hangar 1 has been impacted.

As described in Section 4.1.2 and Table 9, prior to the planned abatement activities six (6) multi-increment near surface soil samples and 2 duplicate samples will be collected from the exposed soil located adjacent and to the south of the area where the abatement techniques will be tested. These samples will be analyzed for PCBs and lead. After the multi-increment soil sampling is complete, and prior to commencement of abatement activities, the exposed soil outside the enclosure will be covered with visqueen to protect them from possible contamination caused by a breach in the containment. No additional sampling of the soil is planned unless there is a breach in containment; in the unlikely event that a breach in containment occurs the soil sampling procedures described above will be replicated to determine if the soil was affected by the breach.

*Air Sampling*

As described in 4.1.2 and Table 9, prior to Abatement activities ambient air samples will be collected and analyzed for PCBs, lead, and copper. The samples will be collected from an upwind and downwind location and will be collected over the course of 3 consecutive days; a duplicate sample will be collected at the downwind sampling location on the first day of baseline sampling. These data will be used to establish ambient air concentrations of PCBs, lead, and copper before abatement activities.

*Particulate Monitoring*

During the baseline ambient air sampling activities described above, particulate concentrations in ambient air will be monitored at the upwind and downwind air sampling locations using direct-read aerosol monitors equipped with PM10 impactor inlets (TSI AM510 or equivalent). These data will be used to establish baseline ambient air PM10 concentrations.

3.7.1.2 Sampling During Abatement Activities:

As described in Section 4.2.4, sampling during abatement consists primarily of air sampling around the exterior of the containment.[11] The sampling will be performed daily in concert with daily monitoring of the containment to determine if any COCs generated by the abatement activities are detectable in the air outside of the containment during work activities.

*Air Sampling*

During active blasting activities (estimated to occur on a total of three days), air samples will be collected from upwind and downwind of the Pilot Study enclosure; these samples will be collected in accordance with the procedures described in Section 3.7.3.5 and analyzed for PCBs and metals (lead and copper[12]). One (1) duplicate sample will be collected from the downwind sampling location on the first day of active blasting activities.

Sampling at the upwind location will provide data of existing conditions at the time of abatement work and sampling from the downwind position will measure if any COCs are detected. If a COC is detected in downwind sampling it will be compared to that of the upwind result at the same time to determine if the source is coming from the containment and the project-specific Airborne Action Levels.

*Particulate Monitoring*

During active blasting activities (estimated to occur on a total of three days), perimeter air particulate concentrations will be monitored at two locations (one upwind and one downwind of the Pilot Study Area) using direct-read aerosol monitors equipped with PM10 impactor inlets (TSI AM510 or equivalent).

3.7.1.3 Post-Abatement Sampling:

As described in Section 4.3.1 and Table 10, post-abatement wipe sampling and bulk CMU wall sampling will be performed to assess the effectiveness of each of the abatement techniques in achieving the target post-abatement acceptance levels. Wipe samples will be collected where the pre-abatement baseline coatings samples were collected and bulk CMU samples will be collected near where the bulk pre-abatement baseline CMU wall material samples were collected. A total of 12 wipes (three (3) from each area the Exterior Steel Members, the Structural Support Steel Members, and the Steel Members under the Mezzanine) and from the CMU Wall) and 4 duplicates (1 from each area) will be collected for PCB analysis and a similar number will be collected for lead analysis. Three (3) bulk wall material samples with one (1) duplicate will also be collected and analyzed for PCBs and lead.

If results from the post-abatement sampling indicate that the target levels have not been reached for a particular area, the area will be re-abated and additional samples will be collected. Post abatement sampling

[11] Personal air sampling of the Abatement Contractor's personnel is the responsibility of the Abatement Contractor and is included in the Abatement Contractor's SSHSP (Attachment B).

[12] Perimeter air samples for copper will only be collected on the days that copper slag is used for abatement.

of soils or the concrete drainage trench will occur only if there is a breach in containment on the side that this feature is located.

#### 3.7.1.4 X-Ray Fluorescence (XRF) Sampling

An X-Ray Fluorescence Spectrometer (XRF) will be used to sample lead concentrations in the painted structural steel members and CMU walls that will be abated as part of the pilot scale abatement activities. The purpose of this sampling is to screen the proposed abatement locations to confirm concentrations of lead are representative of historical data and that those areas will be appropriate for bulk and wipe sampling. A minimum of 3 samples will be taken in each of the proposed abatement areas. Details regarding the field quality control procedures for the XRF survey are presented in Attachment E.

#### 3.7.1.5 Waste Characterization

The collection of waste characterization data will enable appropriate disposal of wastes created during the Pilot Study. As described in Section 4.3.3, ACC will collect samples of each waste stream generated by the abatement activities to determine the appropriate disposal requirements. As described in Table 11, ACC will obtain samples from each of the waste streams generated by the work.

### 3.7.2 *Sampling Identification Nomenclature, Sample Handling, Custody Procedures, and Documentation*

Each sample will be designated with a unique sample number based on type of sample analysis (e.g., SOIL, AIR, WIPE, BULK, XRF), contaminate of concern (e.g., lead (L), PCBs (P), copper (C)); location of sample (e.g., non-porous steel (NPS)) and other appropriate unique identifiers (e.g., date 031516) and sample number. For example, WIPE-L-NPS-031516-01, would represent the first wipe sample that was collected from non-porous steel on 15 March 2016 for the analysis of lead.

If required by the USEPA or NIOSH method, samples will be immediately placed in a pre-chilled insulated container maintained at four degrees Celsius pending transport to the analytical laboratory. Each sample cooler will be chilled with ice and no blue ice containers will be used. Standard chain-of-custody documentation will be maintained at all times. It is anticipated that all samples will be transported to the respective laboratory at the end of each shift for delivery within a 2-6 hour time frame.

Additional details regarding these procedures are presented in Attachment E.

### 3.7.3 *General Sample Collection Procedures and Proposed Analytical Methods*

During the Pilot Study, the following types of samples will be collected and submitted to an analytical laboratory (Section 3.8) for the analysis of PCBs, lead, and sometimes copper:

- Wipe;
- Paint chips and coatings;
- Bulk CMU wall;
- Soil; and,
- Perimeter air.[13]

Table 8 contains information regarding laboratory qualifications and Attachment E describes laboratory-specific detection and quantification limits and quality control procedures. Sample collection procedures and proposed analytical methods for the characterization of wastes generated during the Pilot Study are

[13] Select perimeter air samples may also be analyzed for copper.

discussed in Section 4.3.3.

The sections below present additional information on the general sample collection procedures and proposed analytical methods for these samples.

#### 3.7.3.1 Wipe Sampling

Post-abatement samples will be collected from the abated areas to verify the effectiveness of the abatement technique. Separate wipe samples will be collected to analyze for PCBs and lead.

Wipe samples will be collected in accordance with Association for Testing and Materials (ASTM) D6661-10 (PCBs) or ASTM E1728-16 (lead). The sampling team will be supplied with purchased copies of the referenced ASTM standards and instructed in the proper execution of the defined protocols prior to commencement of work. Additional details are presented below.

*Sampling Procedures for Wipe Sampling:* Identify the sample area (the area to be wiped). The sample area will be a minimum of 1 square foot for lead and 100 square centimeters for PCBs. In general, ACC will use single use sampling templates to define the extent of the sampling area. Prior to, and during installation of the sampling template, ACC will not touch the sampling area. Where the use of a sampling template is not feasible, 2-inch wide low-tack painters tape[14] will be used to define the sampling area; tape used to define the sampling area will not be reused.

PCB wipe samples will be collected in accordance with USEPA 40 CFR Part 761 and ASTM D6661-10 "Standard Practice for Field Sampling of Organic Compounds from Surfaces Using Wipe Sampling" using clean sample wipes saturated with hexane. Sampling technicians will be trained in the sampling methods and will be provided copies of the method for guidance. Each sample will be collected over an area of 100 square centimeters and immediately placed in a glass vial with a tight fitting cap. All samples will be maintained in chilled condition, delivered under standard chain-of-custody protocols to one of the analytical laboratories identified in Section 3.8 and analyzed for PCBs using USEPA Method 8082.

Lead wipe samples will be collected in accordance with CDPH Title 17 25001, and ASTM E1728-16 "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination" requirements using clean sample wipes saturated with deionized water or pre-moistened wipes. Each sample will be collected over an area of 1 square foot and immediately containerized in a plastic, non-porous centrifuge tube. All samples will be maintained in chilled condition, delivered under standard chain-of-custody protocols to one of the analytical laboratories identified in Section 3.8, and analyzed for lead using NIOSH Method 7105 and/or USEPA Method 6020.

Wipe sampling will generally be conducted as follows:

a. Don new, powder-free disposable gloves.
b. Install the sampling template or tape the sampling area without disturbing coatings or settled dust.
c. Select an unused wipe, ensure that it is moist & free from dust, fungus or other materials. For PCBs pre-moisten the wipe with appropriate solvent.
d. Begin sampling at one corner, make first pass side-to-side in an "s" motion within the template opening.
e. Fold wipe over (dirty side in), make second pass, from top-to-bottom in an "s" motion within the template opening.

[14] Low-tack painters tape is designed to not lift or otherwise disturb intact and well-adhered painted surfaces. In the event the tape used during sampling damages the existing CM15 epoxy coating, ACC will mark those areas on a separate drawing for subsequent repair by the designated CM15 recoating contractor and inspection by the designated recoating inspector.

f. If the wipe is for PCBs, go to Step h, if the wipe is for lead, go to Step g.
g. For lead, start with a clean wipe and perform Steps a through e, then fold the wipe over again (dirty side in) and make a third pass around the edges of the template.
h. Fold wipe over and place in a glass vial (PCBs) or centrifuge tube (lead).
i. Label container, note sample location on drawing & log on chain of custody form.
j. Place in cooler / transport container.
k. Record the dimensions and/or size of the sampling area.
l. Discard sampling template and gloves in appropriate debris bags for disposal by Contractor.

#### 3.7.3.2 Bulk Paint and Coating Sampling

Bulk paint and coating sampling requires the removal of small areas of paints and coatings from the original substrate. In general, a sampling area of less than 100 $cm^2$ should result in sufficient material for characterization. ACC will collect samples of major representative paints and coatings at Hangar 1 within the Pilot Study Area. Samples will be collected in sealable plastic baggies and/or immediately containerized in a plastic, non-porous centrifuge tube.

All bulk paint and coating samples will be maintained in chilled condition, delivered under standard chain-of-custody protocols to one of the analytical laboratories identified in Section 3.8, and analyzed for PCBs and lead by USEPA Method 8082 and USEPA Method 6020, respectively.

#### 3.7.3.3 Bulk Sampling of CMU Wall

ACC will use a roto-hammer with masonry drill bit to collect samples from the surface of the CMU wall to approximately 1-inch deep. A small rectangular steel spatula with sides designed for the collection of representative small increments of particulate solids will be used to collect approximately equal sized increments of the drilled material for analysis.

All bulk CMU wall samples will be maintained in chilled condition, delivered under standard chain-of-custody protocols to one of the analytical laboratories identified in Section 3.8, and analyzed for PCBs and lead by USEPA Method 8082 and USEPA Method 6020, respectively.

#### 3.7.3.4 Soil Sampling around the Pilot Study Area

Multi-increment surface soil samples will be collected from the exposed soil immediately adjacent to the Pilot Study Area to determine pre-abatement PCB and lead concentrations in shallow soil; the collection and analysis of the multi-increment soil samples will be performed in accordance with the Interstate Technology & Research Council's (ITRC) *Incremental Sampling Methodology* (ISM) guidance document (ITRC, 2012). The multi-increment soil samples will be collected in 32-ounce glass jars, placed on ice, and submitted to a certified analytical laboratory under standard chain-of-custody procedures. The analytical laboratory will be directed to subsample[15] the soil samples in accordance with the techniques and procedures presented in the ITRC's ISM guidance document and the resulting sample will be analyzed for PCBs by USEPA Method 8082 and lead by USEPA Method 6020. Upon completion of the baseline soil sampling, the Abatement Contractor

[15] A minimum of 30 subsamples shall be collected in a systematic manner from the original sample. Each of the subsamples shall be of approximately equal size and collected with a stainless steel rectangular spatula with sides designed for the collection of representative small increments of particulate solids. For the analysis of PCBs, the total mass of the 30 subsamples shall be approximately 30 grams and the entire 30 gram sample shall be used to prepare an extract for analysis by USEPA Method 8082. For the analysis of lead, the total mass of the 30 subsamples shall be approximately 10 grams and the entire 10 gram sample shall be digested to prepare an extract for analysis by USEPA Method 6020.

will securely cover the soil areas with 12-mil poly sheeting to mitigate potential soil impacts by the abatement activities in the event of a breach in containment.

All soil samples will be maintained in chilled condition, delivered under standard chain-of-custody protocols to one of the analytical laboratories identified in Section 3.8, and analyzed for PCBs and lead by USEPA Method 8082 and USEPA Method 6020, respectively.

#### 3.7.3.5 Perimeter Air Monitoring and Sampling

Perimeter air particulate concentrations will be monitored using direct-read aerosol monitors equipped with PM10 impactor inlets (TSI AM510 or equivalent) calibrated in accordance with manufactures' instructions; particulate matter with an effective diameter of 10 micrometers or less is representative of respirable dust. The inlet to the aerosol monitor will be positioned between 5 and 6 feet above the ground surface. At the end of each day, data from the particulate aerosol monitors will downloaded and reviewed and the time-weighted average concentration will be compared against the PM10 Action Level (Section 3.6).

Air samples will be collected using personal air sampling pumps and filters and the air inlets will be positioned between 5 and 6 feet above ground surface (i.e., within the average person's breathing zone). Air samples for lead and copper (when copper slag is used) will be collected at a flow rate of 1 to 4 liters per minute using 37-millimeter diameter cassettes equipped with 0.8-micron pore size mixed cellulose ester filters and analyzed by NIOSH Method 7105 / EPA Method 6020 or NIOSH Method 7303 / EPA Method 6020 (when copper slag media is used). Air samples for PCBs will be collected at a flow rate of 0.2 liters per minute using glass sorbent tubes (media provided by analytical lab) and analyzed for PCBs by NIOSH 5503. All air samples will be collected over an 8-hour period.

The perimeter air samples will be submitted to one of the analytical laboratories identified in Section 3.8 under standard chain-of-custody procedures.

In general, a minimum of one unused filter or tube per analyte from each batch of filters or tubes used during the Pilot Study will be sent to the analytical laboratory as a media blank sample to determine if the filter media contains detectable concentrations of PCBs, lead, and copper.

#### 3.7.3.6 Meteorological Data Collection

To help determine upwind and downwind directions and document these on a daily basis during baseline sampling (Section 4.1.2) and active abatement activities (Section 4.2.3), ACC will download and print weather data that includes wind speed and wind direction data for Moffett Field from the National Oceanic and Atmospheric Administration (NOAA)'s website (http://www.nws.noaa.gov/view/prodsByState.php?state=CA&prodtype=climate#CF6NUQ).

### 3.7.4 *Field Quality Control*

Field quality control activities will include collection of blank samples and duplicate samples. A summary of frequency of field quality control samples is included in Table 7. The calibration of field instruments is described in Sections 3.7.4.3 and 3.7.4.4 below.

Table 7: Field Quality Control Sample Summary

| Sample Media | Sample Location | Analytes | No. of Samples | No. of Field Duplicates | No. of Field-Equipment Blanks | No. of Media Blanks | No. of Equip. Blanks | Anticipated No. of Samples to Lab |
|---|---|---|---|---|---|---|---|---|
| Wipe | Reusable Equipment (Pre- and Post-Pilot Study) | PCBs<br>lead | 12<br>12 | 6<br>6 | 2<br>2 | 0<br>0 | 0<br>0 | 20<br>20 |
| Wipe | Scaffolding (Pre- and Post-Pilot Study) | PCBs<br>lead | 6<br>6 | 2<br>2 | 2<br>2 | 0<br>0 | 0<br>0 | 10<br>10 |
| Wipe | Concrete Floor (Pre- and Post-Pilot Study) | PCBs<br>lead | 16<br>16 | 4<br>4 | 1<br>1 | 0<br>0 | 0<br>0 | 21<br>21 |
| Wipe | Drainage Trench (Pre-Pilot Study) | PCBs<br>lead | 4<br>4 | 1<br>1 | 1<br>1 | 0<br>0 | 0<br>0 | 6<br>6 |
| Wipe | All Steel elements (Post-Abatement) | PCBs<br>lead | 27<br>27 | 9<br>9 | 3<br>3 | 0<br>0 | 0<br>0 | 39<br>39 |
| Wipe | CMU Walls (Post-Abatement) | PCBs<br>lead | 9<br>9 | 3<br>3 | 3<br>3 | 0<br>0 | 0<br>0 | 15<br>15 |
| Bulk | Soil (Pre-Pilot Study) | PCBs and lead | 6 | 2 | 0 | 0 | 2 | 10 |
| Bulk | Coatings (Pre-Abatement; all Steel elements and CMU Walls) | PCBs and lead | 12 | 4 | 0 | 0 | 2 | 18 |
| Bulk | CMU Wall (Pre- and Post-Abatement) | PCBs and lead | 6 | 2 | 0 | 0 | 4 | 12 |
| Air | Perimeter Air | PCBs<br>lead<br>copper | 12<br>12<br>4 | 2<br>2<br>2 | 0<br>0<br>0 | 2<br>2<br>1 | 0<br>0<br>0 | 16<br>16<br>7 |

#### 3.7.4.1 Field Blank Samples

Field-equipment blanks, media blanks, and equipment blanks will be collected as described below.

Field-equipment blanks for wipe samples: A field-equipment blank wipe sample is a wipe in an unopened sample container (if provided to by the analytical laboratory in this manner) or a clean wipe (with or without solvent) placed in a sample container after handling and exposure to a cleaned template surface. This type of blank is useful in determining whether the wipes or the solvent may be contaminated. ACC will collect field-equipment blanks with a minimum quantity of two or 10% of the total wipe samples collected each day for each type of constituents (e.g., lead and PCBs). ACC will collect field-equipment blank samples at the start (prior to the start of wipe sampling) and end (after the last wipe sample is collected) of each sampling shift and at appropriate intervals (e.g. mid-morning, at lunch break, mid-afternoon), as necessary.

Media blank for air samples: ACC will collect media blanks for air samples with a minimum quantity of one or 10% of the total air samples collected for each type of constituents (i.e., metals and PCBs).

Equipment blanks for bulk material samples: In order to assess the potential for cross contamination due to insufficient decontamination methods, equipment blanks will be collected by pouring distilled water over the decontaminated trowel, drill bit, or chisel and collecting the rinsate into laboratory-supplied, unpreserved one-liter amber jars and 250 milliliter (mL) polypropylene containers pre-preserved with nitric acid. Sample labels

will be affixed to sample containers and will identify the sample ID, sample location, sample time, sample date, sampling technician and requested laboratory analyses.

ACC will collect field equipment blanks with a minimum quantity of two or 10% of each of the total soil, CMU wall, and paint/coating samples collected each day for each Contaminant of Concern COC (e.g., lead and PCBs).

#### 3.7.4.2 Field Duplicate Samples

The duplicate samples will be collected for the following media: wipe samples, bulk building materials and soil. Field duplicates will be collected at a minimum frequency of one per 10 samples for the same analysis as the original sample or one per sampling event if there are fewer than 10 total samples being collected. Field duplicates will be given unique sample identification numbers to provide blind duplicate samples to the analytical laboratory. The locations of duplicate samples and their sample identification numbers will be documented in the field logbook / form.

#### 3.7.4.3 Field Quality Control for XRF Sampling

Only a Niton XRF will be used for this project. The sampling Technician will calibrate the XRF to the Performance Characterization Sheet (PCS; Attachment F) to ensure that the instrument meets calibration check tolerances. A minimum of three calibration check readings using the NIST Standards will be conducted before the beginning if the inspection and after every four hours of testing. A set of calibration check readings will also be collected at the end of each workday. Historically, this instrument is very stable; however, in the unlikely event that an ending calibration should fail, all analyses/readings will be repeated with a compliant Niton XRF.

#### 3.7.4.4 Field Quality Control for Air Monitoring

Routine visits to each sampling station over the sampling period to monitor sampling pump flow rates and confirm that the direct-read aerosol monitors are operating correctly. Sampling pump flowrates will be calibrated on a daily basis and the direct-read aerosol monitors will be zero calibrated on a daily basis. The calibration procedure is included in Section 3.2 of the Project Plan.

### *3.8 Laboratory Selection and Capabilities*

All samples collected during the Pilot Study will be submitted at the end of the sampling day to the independent laboratories with the necessary laboratory accreditations for the designated analytical methods. ACC will deliver the samples to the analytical laboratory in person or through the use of a courier service arranged by the analytical laboratory. In general, all samples will be submitted to the analytical laboratory within 6 hours of the completion of sampling. ACC proposes using one or all of the analytical laboratories presented in Table 8 for the analysis of the soil, air, bulk media, wipe, and waste characterization samples collected during the Pilot Study; however, the final choice of laboratories will be made near the start of the Pilot Study and will depend on the available capacity at that time.

Table 8: Proposed Analytical Laboratories

| Analytical Group | Matrix | Analytical Method | Analytical Laboratories | Available Sample Turn Around Times |
|---|---|---|---|---|
| PCB | Soil | EPA Method 8082 | • T<br>• C | 1-5 Day |
| | Air | NIOSH 5503 | • C<br>• T<br>• M | 1-5 Day |
| | Bulk | EPA Method 8082 | • C<br>• T<br>• M | 1-5 Day |
| | Wipe | EPA Method 8082 | • C<br>• T<br>• M | 1-5 Day |
| Lead | Soil | EPA Method 6020 | • C<br>• T<br>• M | 1-5 Day |
| | Air | NIOSH 7303 / EPA 6020 | • C<br>• T | 1-5 Day |
| | Bulk | EPA Method 3050/6020 | • C<br>• T<br>• M | 1-5 Day |
| | Wipe | EPA Method 3050/6020 | • C<br>• T<br>• M | 1-5 Day |
| Copper | Air | NIOSH 7303 / EPA 6020 | • C<br>• T<br>• M | 1-5 Day |

Abbreviations:
C: Curtis and Tompkins, Berkeley, CA
M: McCampbell Analytical, Inc., Pittsburg, CA
T: Test America, Sacramento, CA

Additional details regarding laboratory qualifications, analytical techniques, hold times, reporting limits, and other quality control measures are presented in Sections 3.1.2 and 3.1.3 and the Project Plan (Attachment E).

### *3.9 Project Plan for Quality Assurance*

The Project Plan for Quality Assurance (Project Plan), Attachment E, describes the quality assurance and quality control protocols that will be implemented during the Pilot Study. The Project Plan summarizes:

- Data quality objectives;
- Proposed sample collection activities (also summarized above);
- Sample handling procedures;
- Analytical methods;
- Quality control requirements,
- Data verification;
- Data validation; and,

- Data usability.

While the Project Plan includes many of the elements of a quality assurance project plan (QAPP) as set forth in *Guidance on Quality Assurance Project Plans,* CIO 2106-G-05 QAPP (EPA, 2012) and Optimized UFP-QAPP Worksheets (IDQTF, 2012) it is not intended to be a fully compliant QAPP.

# 4. Pilot Scale Abatement Activities

Pre-abatement activities including the setup of the Pilot Study enclosure and baseline sampling are presented in Section 4.1, abatement activities including enclosure inspections, blasting techniques, and perimeter air monitoring are presented in Section 4.2, and post-abatement activities including waste characterization, post-abatement sampling, and CM15 recoating and inspection are presented in Section 4.3.

## *4.1 Pre-Abatement Activities*

Prior to conducting pilot scale abatement activities within the Pilot Study Area, debris and surficial sediments on the concrete slab, around the structural steel members, and within the trench drains adjacent to the Pilot Study Area will be removed using vacuums equipped with high-efficiency particulate air (HEPA) filters. HEPA vacuuming of the concrete floor below the work area will be performed to remove debris that could puncture the plastic/membrane and/or containment floor and cause water leaks.

Upon completion of HEPA vacuuming the concrete floors within the Pilot Study Area and the performance of pre-abatement sampling of the concrete floor (Section 4.1.2), a fully-encapsulating enclosure will be constructed around the Pilot Study Area as described in Section 4.1.1. The enclosure will be sealed at floor connections in such a manner that no residual waste water will be allowed to migrate from within. After setup of the enclosure, the CMU walls and structural steel members will be rinsed with potable water using garden-type spray hoses to remove any potential debris and/or windblown dust. Surficial sediments and waste water will be contained within the containment area and will be collected using HEPA vacuums. The waste water from the rinsing operations will be collected in drums and characterized for off-site disposal.

After rinsing the CMU walls and structural steel members, baseline sampling and XRF screening will be conducted at select locations within and around the Pilot Study Area and perimeter air monitoring and sampling will be conducted upwind and downwind of the Pilot Study Area. Additional details on the proposed sampling and monitoring activities are provided below in Section 4.1.2. In total, it is estimated that the pre-abatement activities will take between one to two weeks to complete.

### 4.1.1 *Pre-abatement Enclosure Setup*

A fully encapsulated negative-pressure enclosure will be installed around the Pilot Study Area by the Abatement Contractor prior to the commencement of Baseline Sampling by the Consultant.

The enclosure will be constructed of shrink wrap polyethylene and polyvinyl chloride (PVC). The polyethylene will be a fire-retardant reinforced material capable of handling expected wind loads, interior media blasting activities, exposure to the elements (e.g., 12 mil polyethylene) and will be used to enclose all areas above the concrete floor. Seams will be staggered, overlapped, and taped and heat sealed, and heat guns will also be used to tighten and shrink the materials to form a strong exterior surface.

As blasting media and waste water will accumulate on the floor of the Pilot Study Area during pre-cleaning and abatement activities, 50 mil PVC will be used to cover the concrete floor. As with the polyethylene portion of the enclosure, seams will be staggered, overlapped, and taped and heat sealed. The PVC floor of the enclosure will be installed such that there is adequate vertical rise to capture and contain all anticipated water and/or media wastes that may accumulate during abatement activities.

A three-stage decontamination chamber with a shower and a water tight pan will be connected to the enclosure for the decontamination of personnel and a two-stage decontamination chamber will be connected to the enclosure for the decontamination of equipment. Additional details regarding decontamination are

presented in Section 4.2.5 below. It is anticipated that setup of the enclosure will take approximately 5 business days.

#### 4.1.2 *Baseline Sampling*

After removing debris and surficial sediments from the Pilot Study Area, baseline sampling will be conducted to determine pre-abatement PCB and lead concentrations on concrete foundation,[16] the structural steel, and CMU walls within the Pilot Study Area. Wipe samples will be collected from the concrete foundation and concrete drainage trench adjacent to the Pilot Study Area and bulk material samples will be collected from the paint and coatings on the structural steel elements and from the CMU wall within the Pilot Study Area. Additional information on sample collection procedures is presented in Section 3.7.3.

Scaffolding, equipment and other items used inside containment deemed reusable shall be sampled for site-specific COCs before commencement of work to avoid the use of potentially contaminated materials and/or equipment on site. The primary method of screening these items is to collect representative wipe sampling of the cleaned equipment surfaces in accordance with the wipe sampling protocol outlined in Section 3.7.3.1.

Special attention shall be given to wipe sampling of tight corners, inside hoses, formerly mated surfaces and other hard-to-clean areas to confirm the equipment is safe for reuse. If baseline samples show that materials are contaminated above the established acceptance criteria for COC as described in Table 5 above then the equipment will be rejected or cleaned and re-tested.

[16] As described in Section 4.1, the pre-abatement wipe sampling of the concrete foundation will be conducted after HEPA vacuuming foundation, but prior to the setup of the enclosure (Section 4.1.1).

Table 9: Baseline Sampling

| Sample Type | Sample Media | Number of Samples (Number of Duplicates) | Sampling Location Description [Number of Locations] | Rationale | Proposed Locations |
|---|---|---|---|---|---|
| Wipe | Surface | 8 (2) PCBs<br>8 (2) lead | Concrete Floor [8] | Wipe samples will be collected from the concrete floor in 4 locations within the proposed work area corresponding to locations where the abatement techniques will be tested. Two (2) samples, one for PCBs and lead will be collected from each location using the method prescribed in Section 3.7.3.1, for a total of eight (8) samples. In addition, (2) duplicate samples will be collected from the area and analyzed separately for PCBs and lead. Additional wipe samples will be collected from the concrete floor in four (4) locations outside the proposed work area. Two (2) samples, one for PCBs and one for lead will be collected from each location using the method prescribed in Section 3.7.3.1., for a total of (8) samples. In addition, (2) duplicate samples will be collected from outside the abatement area and analyzed separately for PCBs and lead. The purpose of this sampling is to determine pre-Pilot Study concentrations of contaminants of concern. | Figure 3E |
| Wipe | Surface | 4 (1) PCBs<br>4 (1) lead | Concrete Drainage Trench [4] | 4 wipe samples each for PCBs and lead (plus 1 duplicate sample each) will be collected from the Concrete Drainage Trench outside the enclosure. One additional sample will be collected from an area that is generally downwind of the areas that will be tested; during abatement activities, this area will be covered by the enclosure. These samples are located adjacent to an area of exposed soil. The purpose of this sampling is to determine pre-Pilot Study concentrations of contaminants of concern. All samples will be analyzed for PCBs and lead. | Figure 3E |
| Wipe | Surface | 3 (1) PCBs<br>3 (1) lead | Scaffolding [3] | Three (3) wipe samples each for PCBs and lead (plus 1 duplicate sample each) will be collected from three (3) locations on the scaffolding that will be used during abatement activities; The purpose of this sampling is to determine pre-Pilot Study concentrations of contaminants of concern. | N/A |
| Wipe | Surface | 6 (3) PCBs<br>6 (3) lead | Equipment [6] | Six (6) wipe samples each for PCBs and lead (plus 3 duplicate samples each) will be collected from the three types of blasting equipment. The purpose of this sampling is to determine pre-Pilot Study concentrations of contaminants of concern. | N/A |

| Sample Type | Sample Media | Number of Samples (Number of Duplicates) | Sampling Location Description [Number of Locations] | Rationale | Proposed Locations |
|---|---|---|---|---|---|
| Bulk Material | Coatings | 3 (1) PCBs and lead | Exterior Steel Members [3] | Three (3) bulk samples (plus 1 duplicate sample) of coatings will collected from the exterior steel members that will be abated and analyzed for PCBs and lead. The purpose of these samples is to determine representative pre-abatement concentrations of COCs in this media. | Figure 3A |
| Bulk Material | Coatings | 3 (1) PCBs and lead | Structural Support Steel Members [3] | Three (3) bulk samples (plus 1 duplicate sample) of coatings will collected from the structural support steel members that will be abated and analyzed for PCBs and lead. The purpose of these samples is to determine representative pre-abatement concentrations of COCs in this media. | Figure 3B |
| Bulk Material | Coatings | 3 (1) PCBs and lead | Steel Members under Mezzanine [3] | Three (3) bulk samples (plus 1 duplicate sample) of coatings will collected from the steel members under the mezzanine that will be abated and analyzed for PCBs and lead. The purpose of these samples is to determine representative pre-abatement concentrations of COCs in this media. | Figure 3D |
| Bulk Material | Coatings | 3 (1) PCBs and lead | CMU Wall [3] | Three (3) bulk samples (plus 1 duplicate sample) of the coatings on the CMU wall will be collected from the area that will be abated and analyzed for PCBs and lead. The purpose of this sampling is to determine pre-abatement concentrations of contaminants of concern. | Figure 3C |
| Bulk Material | CMU Wall | 3 (1) PCBs and lead | CMU Wall [3] | Three (3) bulk samples (plus 1 duplicate sample) of the material from the CMU wall will be collected from the area that will be abated. The purpose of this sampling is to determine pre-abatement concentrations of contaminants of concern. | Figure 3C |
| Bulk Material | Soil | 6 (2) PCBs and lead | Exposed soil [6] | Six (6) multi-increment surface soil samples (plus 2 duplicates) will be collected from the exposed soil located generally downwind of the area where the abatement techniques will be tested. The samples will be analyzed for PCBs and lead. The purpose of this sampling is to determine pre-abatement concentrations of contaminants of concern. | Figure 3E |
| Air | Air | 6 (1) PCBs<br>6 (1) lead<br>6 (1) copper | Upwind and Downwind [2 locations per day for 3 days] | Obtain upwind and downwind data for three consecutive days prior to beginning of the Pilot Study and analyze separate samples for PCBs and lead; a duplicate sample will be collected from the downwind location on the first day of sampling. The purpose of this sampling is to determine ambient air concentrations of PCBs, lead, and copper. | Figure 3E |

### 4.1.3 *X-Ray Fluorescence Survey*

An X-Ray Fluorescence Spectrometer (XRF) will be used to survey lead concentrations in the painted structural steel members and CMU walls that will be abated as part of the pilot scale abatement activities. The purpose of the survey is to obtain pre-abatement data that can be used in the field for comparison purposes to determine areas of uniform lead concentrations in the coatings in order to better define sampling locations to ensure that the concentrations of lead are representative of the whole structure. Details regarding the field quality control procedures for the XRF survey are presented in Attachment E.

## *4.2 Abatement Activities*

As multiple layers of paint and/or other coatings are present throughout the Pilot Study Area, the Abatement Contractor will abate the identified Pilot Study Area locations until clean steel or concrete surfaces, free of paint and other residues, are visible; an XRF spectrometer[17] will be used as a screening tool to identify areas which may need additional abatement based on measured lead concentrations. In general, the removal of coatings will be conducted from higher elevations to lower elevations to reduce the potential of re-contaminating a previously abated area. In total, it is estimated that abatement activities will take between 6 and 11 days to complete.[18]

Details on the health and safety requirements and abatement activities are provided below.

### 4.2.1 *Health and Safety Requirements*

Eco Bay Environmental Services (Eco Bay) personnel will be conducting abatement activities within the Pilot Study Area; their EIMP-compliant Site Specific Health and Safety Plan (SSHSP) is included as Attachment B.

All field personnel, including subcontractors, will be required to review Eco Bay's SSHSP plan and provide written acknowledgment of their review. In addition, daily Tailgate Safety Meetings will be held at the beginning of each workday to discuss relevant and/or task-specific safety issues. Records of attendance will be maintained.

During blasting activities within the enclosure, the minimum personal protective equipment (PPE) required for personnel working within the enclosure shall consist of:

- a Tyvek™ inner garment and a saran coated Tyvek™ or "Saranac" outer garment;
- Viton® gloves or equivalent worn over inner latex gloves and neoprene boots worn over top of steel toe boots;[19] and,
- a full-face self-contained breathing apparatus (SCBA) or supplied-air respirator as required by work process (see Attachment B for additional details).

If the Abatement Contractor's personal air monitoring results (see Attachment B for additional details on the personal air monitoring that will be conducted by the Abatement Contractor) indicate that potential PCB and lead exposure levels during blasting are limited, full-face air-purifying respirators equipped with HEPA/organic vapor filter cartridges may be used in place of full-face SCBA or supplied-air respirators. If air-purifying

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[17] The calibration of the XRF will be field-checked as outlined in Attachment E.

[18] Following the setup of the enclosure, active abatement activities will be conducted on a total of three days (days 10, 12, and 14), post-abatement wipe sampling, bulk CMU wall sampling, and XRF screening will be conducted on days 11, 13, and 15), and any additional abatement and or sampling will be conducted thereafter (between days 16 and 20). See Table 4 for additional information.

[19] Gloves and boots shall be taped to the Saranac outer garment to prevent any potential exposure to PCBs, lead, and copper during blasting activities.

respirators are used, the cartridges will be changed at least once daily or more frequently if breathing becomes difficult.

During non-blasting activities within the enclosure, the minimum PPE required for personnel working within the enclosure shall consist of:

- a Tyvek™ garment;
- latex gloves and neoprene boots worn over top of steel toe boots;
- safety glasses;
- hard hat; and,
- half-face or full-face air purifying respirators equipped with HEPA/organic vapor filter cartridges.

Air-purifying respirator filter cartridges will be changed at least once daily.

#### 4.2.2 *Enclosure Inspections*

During active abatement activities within the enclosure, blowers equipped with HEPA filters will be used to maintain a differential pressure between the interior of the enclosure and the ambient air of at least -0.02 inches of water; a data-logging differential manometer shall be used to log differential pressures during abatement activities. All HEPA-filtered devices shall be tested in accordance with ASME N510-1989 and ANSI/ UI 586-1990. Dispersed Oil Particulate testing of all HEPA filtration devices (P100 Level), including vacuum and air filtration devices, will be performed by an independent contractor prior to using any equipment. The testing procedure shall be conducted after construction of the enclosure and must challenge the areas around the HEPA filter/air filtration device seal, the HEPA filter, all seams associated with the construction of the HEPA filtered system, and all other possible penetrations points, including but not limited to, the electrical panel and all components, rivets, screws, wheels, etc.

Throughout the Pilot Study, regular inspections of all critical areas (e.g. seams and in areas of blasting media and/or water accumulation) of the enclosure will be conducted to identify and respond to any materials and/or debris leaving the enclosure. These inspections will be conducted, at a minimum, at the start and end of each shift and twice during each shift.

#### 4.2.3 *Blasting Technologies*

During Abatement Activities in the Pilot Study Area, the Abatement Contractor will test three different blasting technologies on a total of approximately 2,000 square feet (SF) of structural steel members and CMU walls within the enclosure. The three blasting technologies that will be used are: media blasting, ultra-high-pressure water blasting, and vapor-media blasting. Brief descriptions of each of these blasting technologies are provided below. While both media blasting and high-pressure water blasting were used to abate limited areas of the Hangar 1 structure during the NTCRA, there was no evaluation of the implementability or effectiveness of these abatement technologies, no discussion about which technology was more effective at remediating around tight-tolerance mated surfaces or evaluation of the pros and cons of each technology. The three technologies listed below are considered to be most likely to achieve the removal goals by the Abatement Contractor and ACC; other technologies such as soda blasting were considered but rejected as not effective for removing the types of coatings encountered at the Site.

> *Media blasting:* Kleen Blast Copper Slag abrasive media will be used with standard sand blasting equipment to remove existing coatings within the Pilot Study Area. Preliminary estimates by the

Abatement Contractor indicate that between three and four pounds of abrasive media will be required per square foot area being abated, which suggests that a significant amount of solid wastes will need to be disposed of at an off-site facility on completion of abatement activities.

*Ultra-high-pressure water blasting:* Potable water, at pressures between 20,000 to 40,000 pounds per square inch, will be used to remove existing coatings within the Pilot Study Area. Unlike media blasting, this technique does not generate dust, will not result in the impregnation of abrasive media into the structural surfaces being abated, and will not result in a significant amount of solid wastes. Preliminary estimates by the Abatement Contractor suggest that a flow rate between 6 and 8 gallons per minute (gpm) will be required for abatement; while much of this water can be filtered and reused during abatement activities, a significant amount of waste water will need to be characterized and disposed at an off-site facility on completion of abatement activities.

*Vapor-media blasting:* Aerosolized water and ultrafine garnet abrasive media will be used to remove existing coatings within the Pilot Study Area. Similar to the ultra-high-pressure water blasting, vapor-media blasting is a gentler blasting technology than media blasting and will not result in the generation of dust or the impregnation of abrasive media into the structural surfaces being abated. In comparison with media blasting and ultra-high-pressure water blasting, however, relatively small quantities of both recycled water and abrasive media are required which will help minimize the amount of waste water and solid wastes to be disposed of on completion of abatement activities. Preliminary estimates by the Abatement Contractor suggest that approximately 0.5 gpm of recycled water will be used during abatement and approximately 0.5 pounds of ultrafine garnet abrasive media will be required per square foot of area being abated.

Each blasting technology will be used on representative samples of 1) lighter steel beams, 2) thicker structural support steel beams, 3) the steel beams beneath the mezzanine deck, and 4) the CMU walls (Figure 3A through Figure 3D). The primary purpose of assessing the multiple blasting technologies within the Pilot Study Area is to:

1) Determine the implementability of each blasting technology and their effectiveness at removing existing coatings from the different types structural elements within the Pilot Study Area;
2) Identify which technologies are most effective at remediating around tight-tolerance mated surfaces (e.g., steel members riveted together); and,
3) Assess production rates of these technologies.

Post-abatement, verification wipe sampling (Section 4.3.1) will be conducted to determine whether post-abatement PCB and lead concentrations on the abated structural elements meet Post-Abatement Acceptance Criteria (Table 5). Additional details on the proposed post-abatement sampling are presented in Section 4.3.1 and Table 10 (Post-Abatement Sampling). If the abated structural elements do not meet the Post-Abatement Acceptance Criteria, the Abatement Contractor will be directed to re-abate the area one more time.[20] If post-abatement wipe sampling results following re-abatement still do not meet the Post-Abatement Acceptance Criteria, the blasting technology will be considered unsuitable for abatement. All structural elements included in the Pilot Study will be recoated with CM15 epoxy coating. On completion of recoating activities, an independent third party qualified coating inspector will inspect the coatings.

[20] It is understood that original coatings may remain between and around tight-tolerance mated surfaces (e.g., steel members riveted together).

On completion of abatement activities with each blasting technology, the work area will be cleaned using HEPA-equipped vacuums and a visual inspection of the entire work area including the decontamination unit, all plastic sheeting, seals over ventilation openings, doorways, windows, and other openings will be conducted to identify any remaining dust, debris, or residues from the abatement activities. If any such materials are found, the area will be re-cleaned. It is estimated that two working days[21] will be required to complete the blasting, cleanup, and post-abatement sampling activities for each blasting technology. As a result, all blasting-related abatement activities should be complete within 6 working days.

#### 4.2.4 *Perimeter Air Monitoring and Sampling*

During active blasting activities (estimated to occur on a total of three to six days), perimeter air particulate concentrations will be monitored at two locations (one upwind and one downwind of the Pilot Study Area) using direct-read aerosol monitors equipped with PM10 impactor inlets (TSI AM510 or equivalent). On these days, ACC will routinely visit the perimeter air monitoring stations to monitor for the presence of visible dust and evaluate if abatement activities are resulting in elevated particulate concentrations around the site. If visible dust is observed or if PM10 Dust concentrations exceed 0.11 $mg/m^3$ for 5-minutes or more at either of the perimeter air monitoring stations, abatement activities will be temporarily terminated until the source of the particulates is identified and has been controlled. At the end of each day, data from the particulate aerosol monitors will be downloaded and reviewed; average time-weighted concentrations will be compared against the PM10 Action Level (Section 3.6).

In addition, during active blasting activities, air samples for PCBs, lead, and copper will be collected at these locations during the same period using personal air sampling pumps and filters as described in Section 3.7.3.5. Air samples for copper will only be collected on days where copper slag is used as the blast media. One duplicate sample will be collected from the downwind sampling location on the first day of active blasting activities.

The perimeter air samples will be submitted to a certified analytical laboratory under standard chain-of-custody procedures. At the analytical laboratory, the samples will be analyzed on an accelerated turnaround time (TAT) and results will be provided to ACC within 24 hours of sample receipt by the analytical laboratory. Abatement activities will be suspended until the laboratory data has been received and the measured concentrations in the perimeter air samples have been compared to their respective Action Levels (see Section 3.7.6.1) to determine if additional mitigation measures are necessary.

#### 4.2.5 *Decontamination*

All personnel leaving the enclosure will exit through the three-stage decontamination chamber that is a part of the negative pressure enclosure. In the first chamber all visible debris shall be HEPA vacuumed from each person's PPE, in the second chamber, potable water will be used to remove any residual contamination,[22] and in the third chamber, personnel will remove and dispose of their PPE in waste debris bags provided by the contractor.

All reusable equipment (e.g., scaffolding, blasting equipment, etc.) leaving the enclosure will be decontaminated using a two-stage decontamination chamber. The setup of the two-stage decontamination chamber is identical to that of the three-stage decontamination chamber except that the third chamber is not

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[21] It is estimated that blasting activities will be completed within one day and that cleanup and verification sampling activities will be conducted the following day.

[22] All decontamination water will be collected, filtered, characterized, and disposed of at an off-site facility in accordance with applicable laws and regulations.

necessary. Following decontamination, post-abatement wipe sampling[23] (Section 4.3.1) will be conducted to determine if the equipment meets the Post-Abatement Acceptance Criteria (Table 5). During wipe sampling, special attention will be placed on hard-to-clean areas (e.g., tight corners, inside hoses, etc.) to confirm that the equipment is safe for reuse. A total of 9 wipe samples (plus 3 duplicates) will be collected from the decontaminated scaffolding that was used during abatement activities and three (3) wipe samples (plus 1 duplicate) will be collected from the decontaminated blasting, or other portable, equipment to determine whether the decontaminated equipment meets the Target Post-Abatement Acceptance Criteria (Table 5).

## *4.3 Post-Abatement Activities*

Following abatement activities, post-abatement sampling will be conducted to determine whether Post-Abatement Acceptance Criteria have been met, CM15 epoxy coatings will be reapplied, and Pilot Abatement Study wastes will be characterized for disposal and disposed of at an off-site facility. Additional details on these post-abatement activities are provided below. It is anticipated that decontamination and removal of the abatement enclosure, the recoating and inspection of abated areas, and the final post-abatement sampling will be completed within 5 to 10 working days.

### 4.3.1 *Post-Abatement Sampling*

Following the completion of abatement activities with each blasting technology, wipe sampling of all abated materials will be conducted to assess whether the abatement procedures proposed by the Abatement Contractor have been effective and to determine if the Post-Abatement Acceptance Criteria (Table 5) have been achieved. In addition, bulk material samples of the abated CMU wall and an XRF will be used to determine residual remaining lead concentrations on the abated materials and the approximate removal efficiency of each blasting technology. The post-abatement bulk CMU wall and XRF sampling locations will be collected as close as possible to the baseline bulk CMU wall and XRF sampling locations. Post-abatement soil samples and wipe samples of concrete trench drain[24] will only be collected if a breach in containment is observed during abatement activities. Table 10 summarizes the proposed Post-Abatement sampling plan. All samples will be collected in accordance with the procedures outlined in Section 3.7 and in Attachment E.

As described in Section 4.2.3, if the post-abatement sampling results indicate that the target Post-Abatement Acceptance Criteria (Table 5) have not been achieved, the Abatement Contractor will be directed to re-abate the area once and the re-abated area will be resampled. If post-abatement sampling results following re-abatement still do not meet the Post-Abatement Acceptance Criteria, the blasting technology will be considered unsuitable for abatement. On completion of all abatement and sampling activities, the abated materials will be recoated with CM15 epoxy coating (Section 4.3.2).

[23] All wipe samples will be collected in accordance with ASTM D6661-10 (PCBs) or ASTM E1728-16 (lead). Wipe samples will be placed in plastic centrifuge tubes and submitted to a certified analytical laboratory under standard chain-of-custody procedures for the analysis of PCBs by USEPA Method 8082 and lead by USEPA Method 6020.

[24] If necessary, post-abatement soil samples and wipe samples of the trench drain will be collected and analyzed as outlined in Section 3.7.3.4.

Table 10: Post-Abatement Sampling

| Sample Type | Sample Media | Sampling Location Description [Number of Sampling Locations] | Post-Abatement Number of Samples | Rationale For Post-Abatement Verification Sampling | Proposed Locations |
|---|---|---|---|---|---|
| Wipe | Surface | Concrete Floor [8] | 8 (2) PCBs<br>8 (2) lead | The number and location of samples will be the same as Baseline samples for each COC in each of the abatement areas. | Figure 3E |
| | | Concrete Trench Drain [0] | (a) | | Figure 3E |
| | | Scaffolding [3] | 3 (1) PCBs<br>3 (1) lead | | N/A |
| | | Reusable Equipment [6] | 6 (3) PCBs<br>6 (3) lead | | N/A |
| | | Exterior Steel Members [9] | 9 (3) PCBs<br>9 (3) lead | 3 wipe samples will be collected from each of the areas abated using the 3 abatement techniques from each structural element to evaluate whether the abatement method can achieve the Target Post-Abatement Acceptance Criteria. | Figure 3A |
| | | Structural Support Steel Members [9] | 9 (3) PCBs<br>9 (3) lead | | Figure 3B |
| | | Steel Members under Mezzanine [9] | 9 (3) PCBs<br>9 (3) lead | | Figure 3D |
| | | CMU Wall [9] | 9 (3) PCBs<br>9 (3) lead | | Figure 3C |
| Bulk Material | CMU Wall | CMU Wall[25] [3] | 3 (1) PCBs and lead | The number and location of samples will be the same as Baseline samples for each COC in each of the abatement areas. | Figure 3C |
| | Soil | Soil [0] | (a) | | Figure 3E |

Notes:
(a) Post-abatement multi-increment soil samples and concrete trench drain wipe samples will only be collected if a breach in containment is observed during abatement activities.
Abbreviations:
N/A: not applicable

### 4.3.2 *Recoating and Inspection*

As the original PCB and lead containing coatings may remain between and in close proximity to tight-tolerance mated surfaces (e.g., riveted steel members) or some abatement methods may not achieve the Target Post-Abatement Acceptance Criteria, all abated structural elements will be recoated with CM15 epoxy coating by a qualified contractor after all abatement, post-abatement sampling, and decontamination activities have been completed[26]; Attachment D contains additional information on the CM15 epoxy coating and application instructions. Construction Testing Services (Table 1), a third party independent qualified coating inspector, will be contracted to inspect the coating on completion of the recoating.

[25] A roto-hammer with a masonry drill bit will be used to collect samples from the surface of the CMU wall to approximately 1-inch deep.
[26] It is estimated that post-abatement cleanup, decontamination, and removal of the abatement enclosure will take approximately one week.

On completion of recoating and inspection activities, NASA's Environmental Management Division will inspect and sign off on the Pilot Study.

#### 4.3.3 *Waste Disposal*

During abatement activities, as feasible, all lead and PCB-containing materials shall be managed in a manner to maximize construction waste recycling and landfill diversion. All wastes generated during the pilot scale abatement activities, including but not limited to spent media, liquid, and solid and liquid decontamination wastes, will be segregated, classified, labeled, packaged, and transported and disposed of at permitted off-site disposal facilities[27] in compliance with applicable laws and regulations and the MFA Leasehold EIMP. While onsite, all wastes will be stored in accordance with applicable waste storage requirements.

Eco Bay will be responsible for managing waste on-site and ACC will be responsible for the characterization of each waste stream[28] for disposal. On completion of abatement and decontamination activities for each blasting technology, Eco Bay personnel will containerize all solid spent blasting media and decontamination wastes in metal drums; bulk loose and peeling paint coatings will be containerized separately from the spent blasting media and decontamination wastes. Waste water from the abatement activities will be collected using vacuums equipped with HEPA air filters and stored in 100-gallon totes within the enclosure; after 24-hours (or longer if necessary), the water will be siphoned into another 100-gallon tote leaving any settled solids in the original tote undisturbed.

ACC will subsequently collect representative composite samples from each drum of waste and each tote (i.e., both the liquid and settled solids). The waste characterization samples will be representative of all construction wastes, including water effluents, dusts, and other solids, generated by the abatement technology. The samples will be shipped to one of the analytical laboratories identified in Section 3.8 under standard chain-of-custody procedures and characterized for PCBs by USEPA Method 8082 and Title 22 Metals by USEPA Method 6020 on a standard TAT; additional samples may be collected and/or additional analyses may be performed at the request the off-site disposal facility. After sampling by ACC, the drums and totes will be sealed, wet wiped, decontaminated, and removed from the enclosure and placed in a secure, bermed, waste staging area with appropriate signage; the waste staging area is shown on Figure 2C. All wastes will be characterized and properly disposed of within 90 days of completion of Pilot Study abatement activities.

Table 11 provides a list of anticipated waste streams, analytical techniques, storage containers and requirements, and transportation and disposal requirements.

[27] The selected off-site disposal facilities shall be in compliance with the CERCLA Off-Site Rule.

[28] It is anticipated that PPE associated materials including disposable suites and polyethylene sheeting shall be separated and tested separately from other debris in accordance with Table 11 below.

Table 11: Anticipated Waste Streams, Analytical Methods, Storage Containers, Storage, Transportation, and Disposal Requirements

| Anticipated Waste Stream | Anticipated Characterization | Storage Containers | Storage Requirements | Transportation Requirements | Disposal Requirements |
|---|---|---|---|---|---|
| Wastewater from pressure-blasting and decontamination procedures | Samples of wastewater will be collected and analyzed for PCBs by USEPA Method 8082 and Title 22 Metals by USEPA Methods 6010/6020/7470A to determine appropriate management and disposal procedures. All waste storage containers will be sampled for waste characterization.<br><br>Primary COCs: PCBs and lead | 100-gallon totes. It is anticipated that 200-300 gallons of non-hazardous water will be generated from ultra-high pressure water blasting. | The 90-day storage limit applies to containers containing hazardous waste. Containers will be marked as "pending analysis" and include the accumulation date, composition and physical state of the waste, and name and address of generator. Containers will be labeled as either hazardous waste or nonhazardous waste upon receipt of analysis. Containers will be stored in pre-designated hazardous waste storage area. | Off-site shipment and disposal wastewater is anticipated.<br><br>If wastewater is determined to be hazardous, a hazardous waste manifest and DOT vehicle placarding are required. A Cal/EPA-permitted transporter must be used. NASA will sign all waste manifests. | Liquids with PCB concentrations ≥ 5 mg/L but <50 mg/L are regulated by the State of California.<br><br>All wastes will be sent to an appropriate TSDF that has been approved under the CERCLA Off-site Rule. Any TSCA- or state-regulated wastes must be disposed of at an approved facility. See Table 1 in Section 3.2 for the selected Waste Disposal Facility. |
| Blasting materials, building materials, debris, paint chips, dust, etc. that is suspected of containing PCBs or hazardous waste. | The waste material will be sampled to determine appropriate management and disposal procedures. All waste storage containers will be sampled for waste characterization.<br><br>Samples will be analyzed for PCBs by USEPA Method 8082 and Title 22 Metals by EPA Method SW-846. TCLP and/or WET extractions and analyses will be conducted as necessary (a).<br><br>Primary COCs: PCBs, lead, and copper | Hazardous materials will be stored in 55-gallon drums; non-hazardous materials may be transferred to other containers as appropriate.<br><br>It is anticipated that:<br><br>• between 1 and 3 drums of non-hazardous waste will be generated from media blasting with copper slag;<br><br>• approximately one 5-gallon bucket of settled solids will be generated from ultra-high pressure water blasting; and,<br><br>• less than 1 drum of waste will be generated from vapor media blasting. | Building debris and paint chips that contain both RCRA-and TSCA-regulated wastes will be managed in accordance with both. Containers will be marked as "pending analysis" and include the accumulation date, composition and physical state of the waste, and name and address of generator. Containers will be labeled as either hazardous waste or nonhazardous waste upon receipt of analysis. Containers will be stored in pre-designated hazardous waste storage area. | If the waste is determined to be hazardous, a hazardous waste manifest and DOT vehicle placarding are required. A Cal/EPA-permitted transporter must be used. NASA will sign all waste manifests. | Associated building debris and solids with concentrations <50 ppm PCBs are not regulated by TSCA. All such wastes will be sent to an appropriate TSDF that has been approved under the CERCLA Off-site Rule and their permits. Any TSCA- or state-regulated wastes must be disposed of at an approved facility. In accordance with 40 CFR 761.62 subpart R, all TSCA-regulated PCB-containing wastes will be disposed of at a hazardous waste landfill. |

| | | | | | |
|---|---|---|---|---|---|
| Personal Protective Equipment | PPE (i.e. disposable suits, gloves, booties) waste material will be sampled to determine appropriate management and disposal procedures.<br><br>Representative samples will be analyzed for PCBs by USEPA Method 8082 and Title 22 Metals by EPA Method SW-846. TCLP and/or WET extractions and analyses will be conducted as necessary (a).<br><br>Primary COCs: PCBs and lead | PPE wastes will be placed in double plastic bags at the point of generation and used to transport to the wastes to DOT-approved 55-gallon drums. | Labeling should consist of a completed commercial hazardous waste label. Containers will be sealed/covered when not being loaded/unloaded. Containers will be stored in pre-designated hazardous waste storage areas with secondary containment. | If material is a hazardous waste, a hazardous waste manifest and DOT vehicle placarding are required. A Cal-EPA permitted transporter must be used. NASA will sign all waste manifests.<br><br>Decontaminated PPE may be placed into non-hazardous solid waste 55-gallon drums to be picked up by contracted solid waste disposal contractor. | Associated PPE waste materials with concentrations <50 ppm PCBs are not regulated by TSCA. All such wastes will be sent to an appropriate TSDF that has been approved under the CERCLA Off-site Rule and their permits. Any TSCA- or state-regulated wastes must be disposed of at an approved facility. In accordance with 40 CFR 761.62 subpart R, all TSCA-regulated PCB-containing wastes will be disposed of at a hazardous waste landfill. |
| Materials with low potential for contamination (e.g., trash, inert demolition debris, job control waste, clean polyethylene liners, etc.) | Decontaminated other waste material will be sampled to determine appropriate management and disposal procedures.<br><br>Representative samples will be analyzed for PCBs by USEPA Method 8082 and Title 22 Metals by EPA Method SW-846. TCLP and/or WET extractions and analyses will be conducted as necessary (a).<br><br>Primary COCs: PCBs and lead | Waste to be stored in non-hazardous roll-offs or 55-gallon drums. It is anticipated that 6-12 cubic yards will be generated. | Non-hazardous waste will be stored separate from hazardous waste and labeled accordingly to prevent commingling of hazardous and non-hazardous wastes. | No special transporter requirements for wastes determined to be non-hazardous. NASA will sign all waste manifests. | Associated waste materials with concentrations <50 ppm PCBs are not regulated by TSCA. All such wastes will be sent to an appropriate TSDF that has been approved under the CERCLA Off-site Rule and their permits. Any TSCA- or state-regulated wastes must be disposed of at an approved facility. In accordance with 40 CFR 761.62 subpart R, all TSCA-regulated PCB-containing wastes will be disposed of at a hazardous waste landfill. |

Notes:

(a) If the total concentration of a metal exceeds 10 times its Total Threshold Limit Concentration, a Waste Extraction Test (WET) is required. If the total concentration of a metal exceeds 20 times its Toxicity Characteristic Leaching Procedure (TCLP) concentration, a TCLP extraction will be performed on the sample.

## 5. Reporting

On completion of the pilot scale abatement study activities, PV will prepare an Implementation Report that:

1) summarizes the field activities conducted, abatement techniques tested, and any significant deviations from the Work Plan;
2) presents all pre-abatement field data, XRF screening, and all reusable equipment, building material, soil, and air sampling results;
3) presents all abatement air sampling results and summarizes worker monitoring results;
4) presents all post-abatement field data, XRF screening, and all wipe and CMU wall sampling results;
5) presents maps showing the abatement areas and sampling locations;
6) presents an evaluation of the effectiveness of each abatement technology and a discussion regarding the pros and cons of each abatement technology;
7) summarizes information regarding the disposal of Pilot Study wastes, including, but not limited to, tables summarizing laboratory analytical results for the waste characterization samples and the volumes and/or masses of impacted materials disposed of at off-site facilities, a compilation of laboratory analytical data report sheets, and waste manifests; results of quality control and quality assurance assessments and the usability assessment, and,
8) documents the post-abatement reapplication of the CM15 epoxy coating and inspections within the Pilot Study Area.

The Implementation Report will be incorporated into an Engineering Evaluation/Cost Analysis Report (EE/CA) for the full-scale abatement of Hangar 1 and submitted to the USEPA, Water Board, and NASA as documentation of the actions taken; the Implementation Report will be revised by PV as necessary to adequately document the Pilot Scale Abatement Study.

## 6. References


ACC, 2015. *PCB, Lead, and Asbestos Sampling Report, Hangar One, Moffett Federal Air Field, Mountain View, California,* ACC Environmental Consultants, February 24, 2015.

AMEC, 2013. *Final After Action Completion Report for Non-Time-Critical Removal Action for Polychlorinated Biphenyl (PCB) Contamination, Installation Restoration (IR) Site 29, Hangar 1, Former Naval Air Station (NAS) Moffett Field, Moffett Field California,* AMEC Environment & Infrastructure, Inc., November 2013.

BAAQMD, 2015. *Bay Area Air Pollution Summary – 2014,* BAAQMD, 2015. Downloaded from: http://www.baaqmd.gov/~/media/Files/Communications%20and%20Outreach/Annual%20Bay%20Area%20Air%20Quality%20Summaries/pollsum2014.ashx?la=en

Benchmark, 2003. *Polychlorinated Biphenyl, Lead, and Asbestos Sampling Report. Hangar 1/Moffett Field, Mountain View, California,* Benchmark Environmental Engineering, January 9, 2003.

Harding, 2000. *Environmental Baseline Survey, NASA Research Park Parcel 1, Moffett Federal Airfield, Moffett Field, California,* Harding Lawson Associates, October 18, 2000.

IDQTF, 2012. *Optimized UFP-QAPP Worksheets,* Intergovernmental Data Quality Task Force Uniform Federal Policy (IDQTF) for Quality Assurance Project Plans

ITRC, 2012. *Technical and Regulatory Guidance, Incremental Sampling Methodology,* Interstate Technology & Regulatory Council, February 2012.

Navy, 2008. *Engineering Evaluation/Cost Analysis Revision 1, Installation Restoration Site 29, Hangar 1, Former Naval Air Station Moffett Field, Moffett Field, California*, Base Realignment and Closure Program Management Office West. July 30, 2008.

Navy, 2013. *Proposed Plan for Hangar 1, Former Naval Air Station Moffett Field Site 29,* U.S. Department of the Navy, July 2013.

PRC, 1996. *Final Station-Wide Remedial Investigation Report, Moffett Federal Airfield, California,* PRC Environmental Management, May 21, 1996.

RORE, 2013. *Focused Feasibility Study Installation Restoration Site 29 (Hangar 1) Former Naval Air Station Moffett Field, California,* RORE, Inc., May 2013.

USEPA, 1990. *Federal Facility Agreement Under CERCLA Section 120 Between the U.S. Environmental Protection Agency and U.S. Department of the Navy and the State of California Represented by the California Department of Health Services and the California Regional Water Quality Control Board, San Francisco Bay Region,* August 1990.

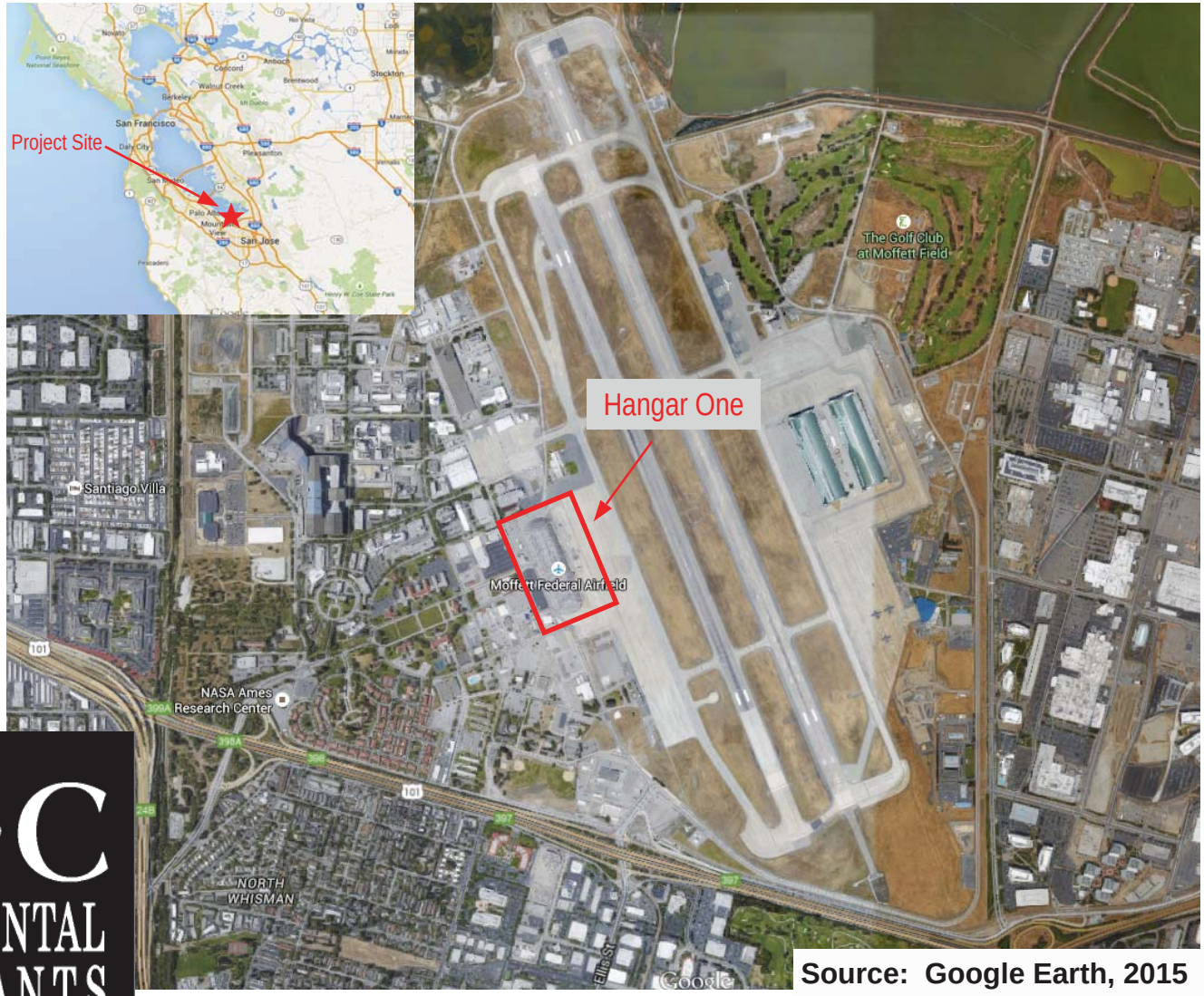
USEPA, 1993. *Amendment of the Federal Facility Agreement, NAS Moffett Field, U.S. Environmental Protection Agency and U.S. Department of the Navy and the State of California Represented by the California Department of Toxic Substances Control and the California Regional Water Quality Control Board, San Francisco Bay Region,* December 17, 1993.

USEPA, 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use,* OWSER No. 9200.1-85, EPA 540-R-08-005, United States Environmental Protection Agency, 13 January 2009.

USEPA, 2012. *Final Draft Guidance on Quality Assurance Project Plans, CIO 2016-G-05 QAPP,* United States Environmental Protection Agency, January 2012.

# Figure Number 1

## Regional Location Map
## Moffett Federal Airfield- Hangar One
## Mountain View, California




Source: Google Earth, 2015

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Scale: None Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

# Figure Number 2A

**Pilot Study Location Map**
**Moffett Federal Airfield- Hangar One**
**Mountain View, California**

**Source: Google Earth, 2015**

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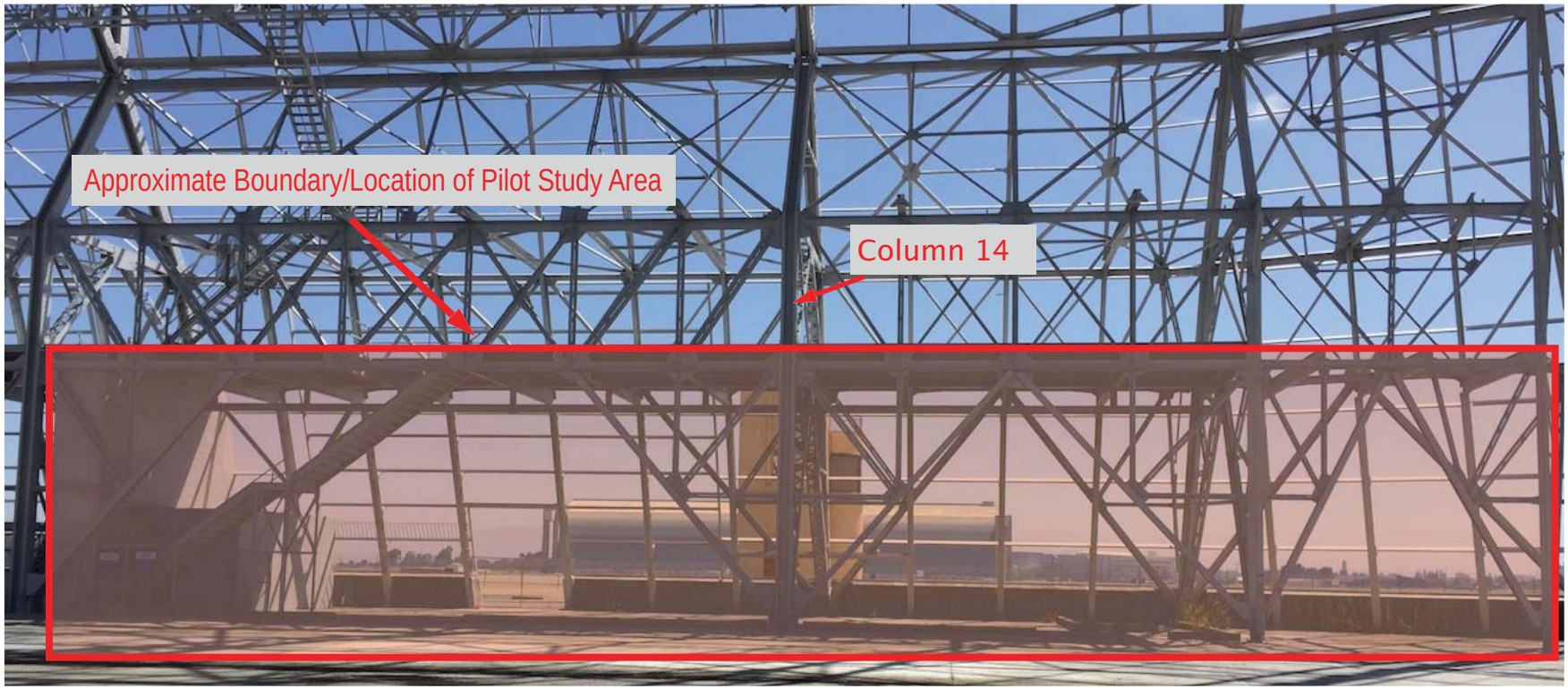
Scale: None Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

# Figure Number 2B

**Pilot Study Location Map**
**Moffett Federal Airfield- Hangar One**
**Mountain View, California**

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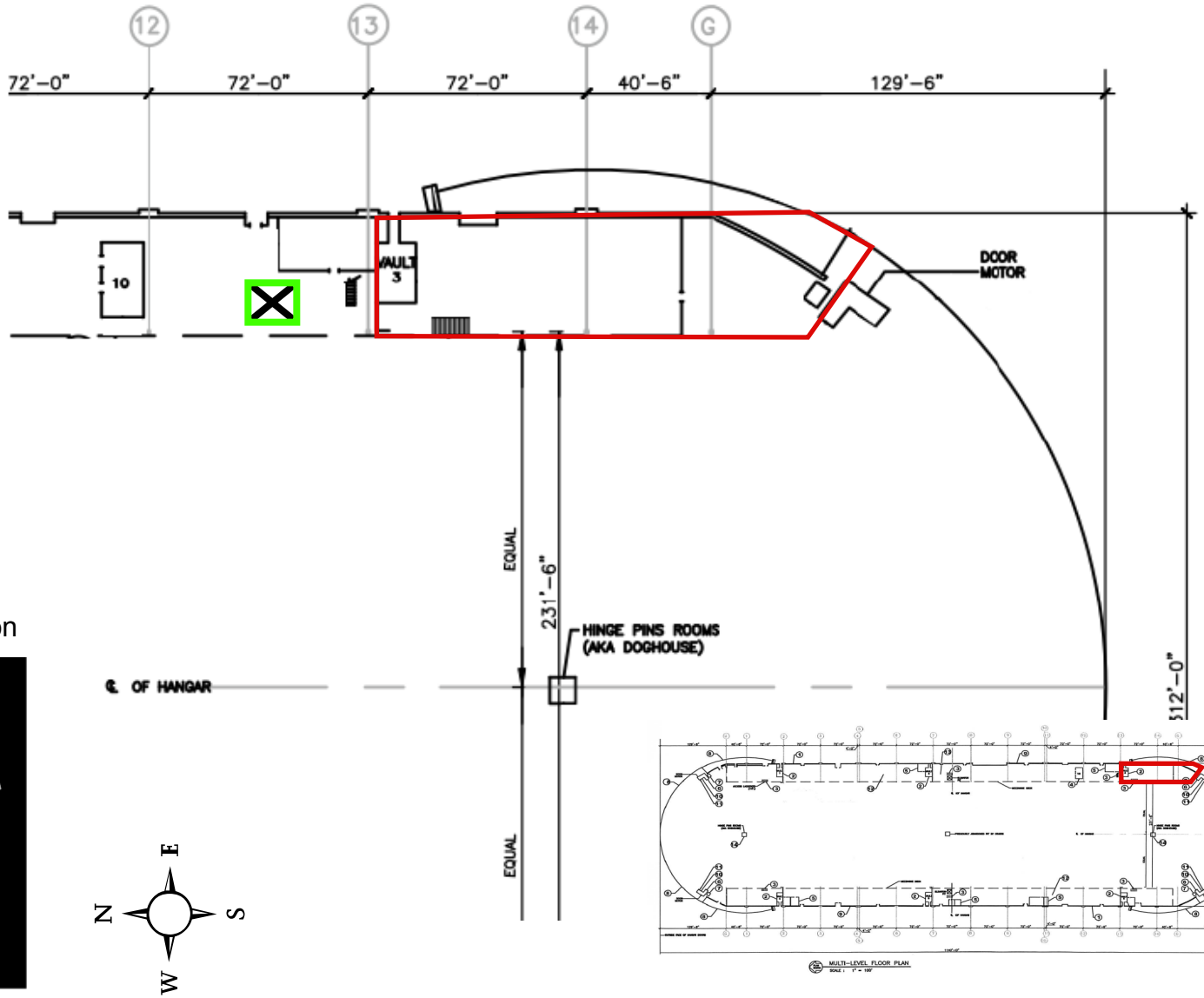
Scale: None Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

# Figure Number 2C

## Pilot Study Sample Location Map
## Moffett Federal Airfield- Hangar One
## Mountain View, California




Waste Storage Location

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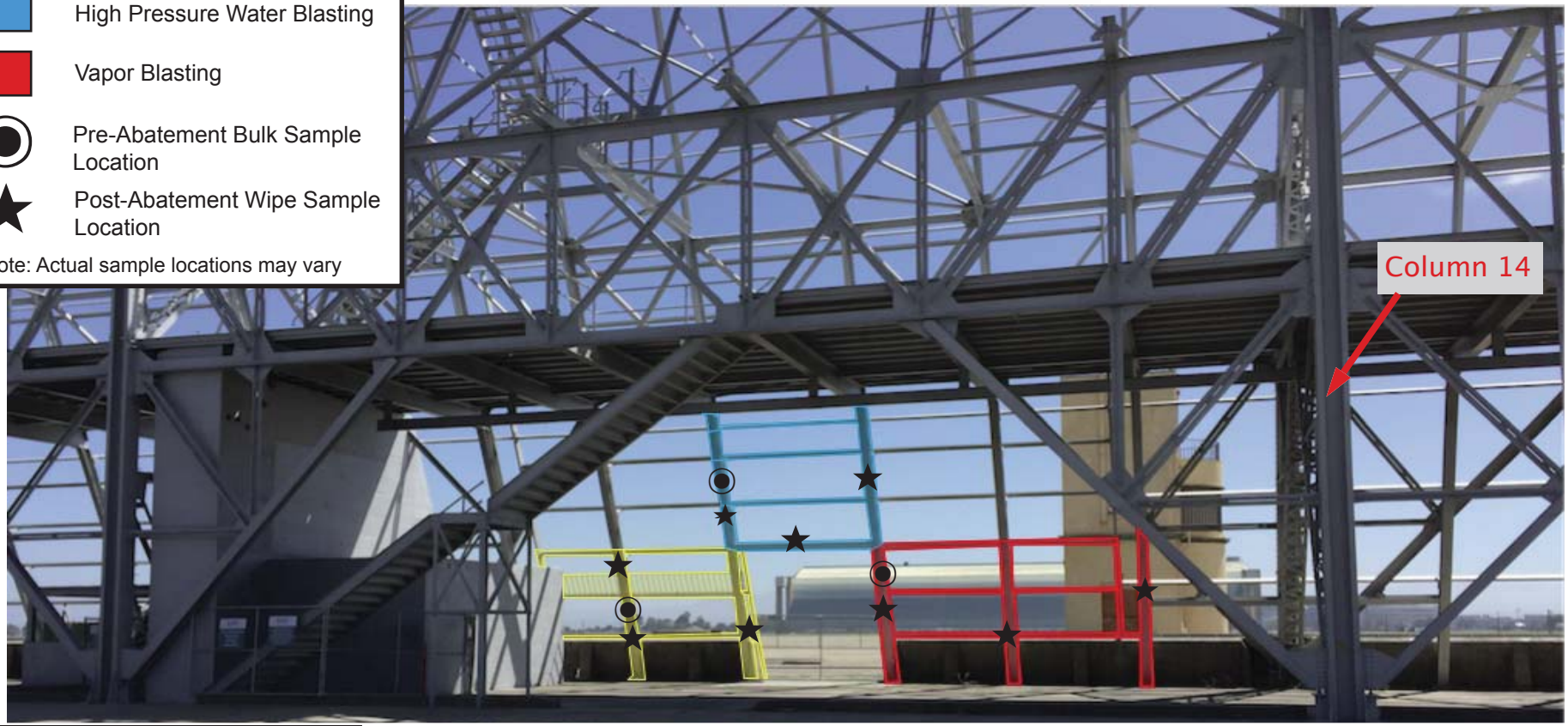
# Figure Number 3A

## Pilot Study Sample Location-Map 3A
## Moffett Federal Airfield- Hangar One
## Mountain View, California

LEGEND

- Media Blasting
- High Pressure Water Blasting
- Vapor Blasting
- Pre-Abatement Bulk Sample Location
- Post-Abatement Wipe Sample Location

*Note: Actual sample locations may vary

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Scale

0' 10' 20'

Drawn By: JMS

Date: 6/03/15

Project Number: 1591-001.02

# Figure Number 3B

## Pilot Study Sample Location-Map 3B
## Moffett Federal Airfield- Hangar One
## Mountain View, California

LEGEND

- Media Blasting
- High Pressure Water Blasting
- Vapor Blasting
- Pre-Abatement Bulk Sample Location
- Post-Abatement Wipe Sample Location

*Note: Actual sample locations may vary

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Scale

0' 5' 10'

Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

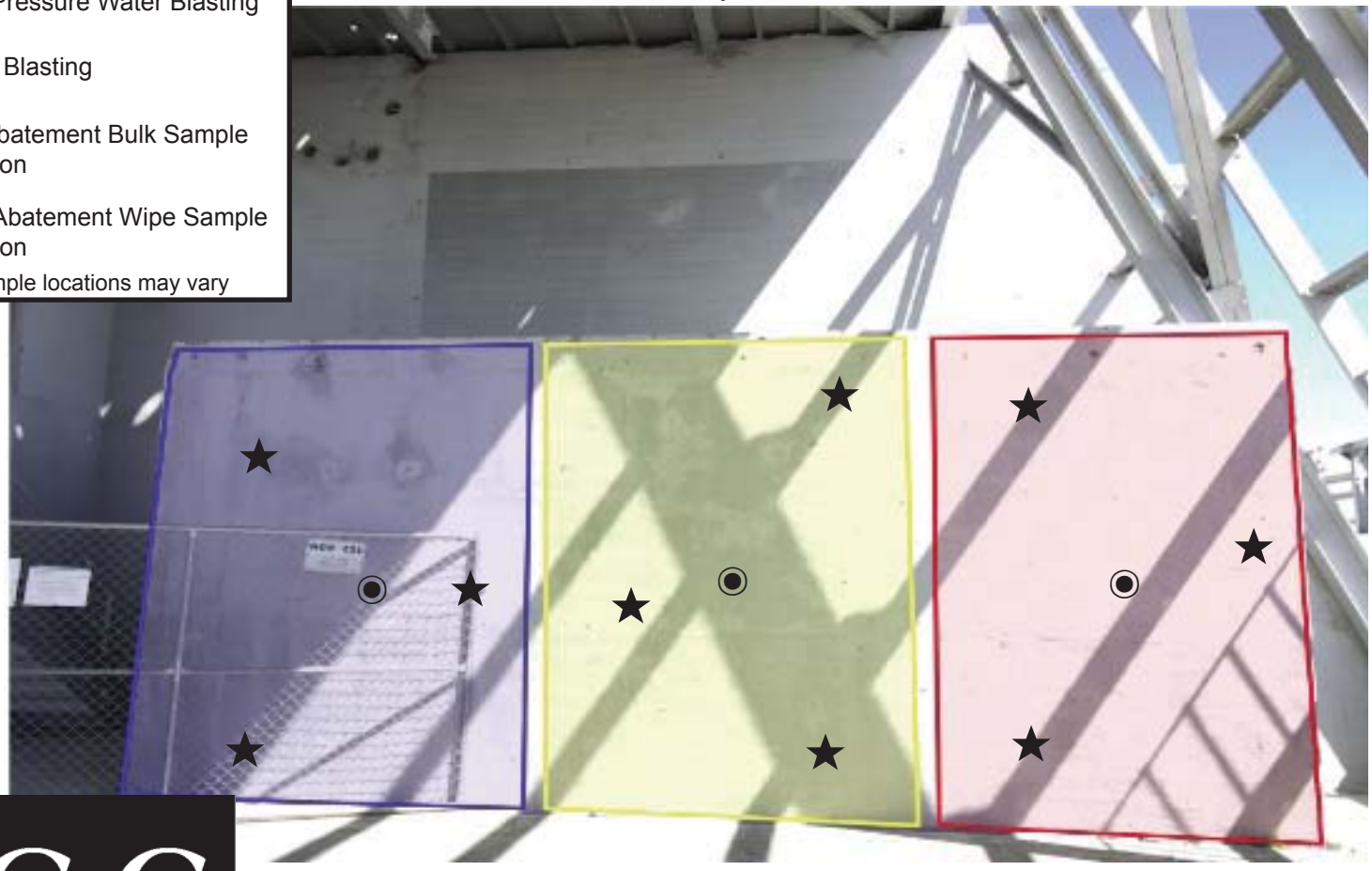
# Figure Number 3C

## Pilot Study Sample Location- Map 3C
## Moffett Federal Airfield- Hangar One
## Mountain View, California

LEGEND

- Media Blasting
- High Pressure Water Blasting
- Vapor Blasting
- Pre-Abatement Bulk Sample Location
- Post-Abatement Wipe Sample Location

*Note: Actual sample locations may vary



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Scale

0' 1.5' 3'

Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

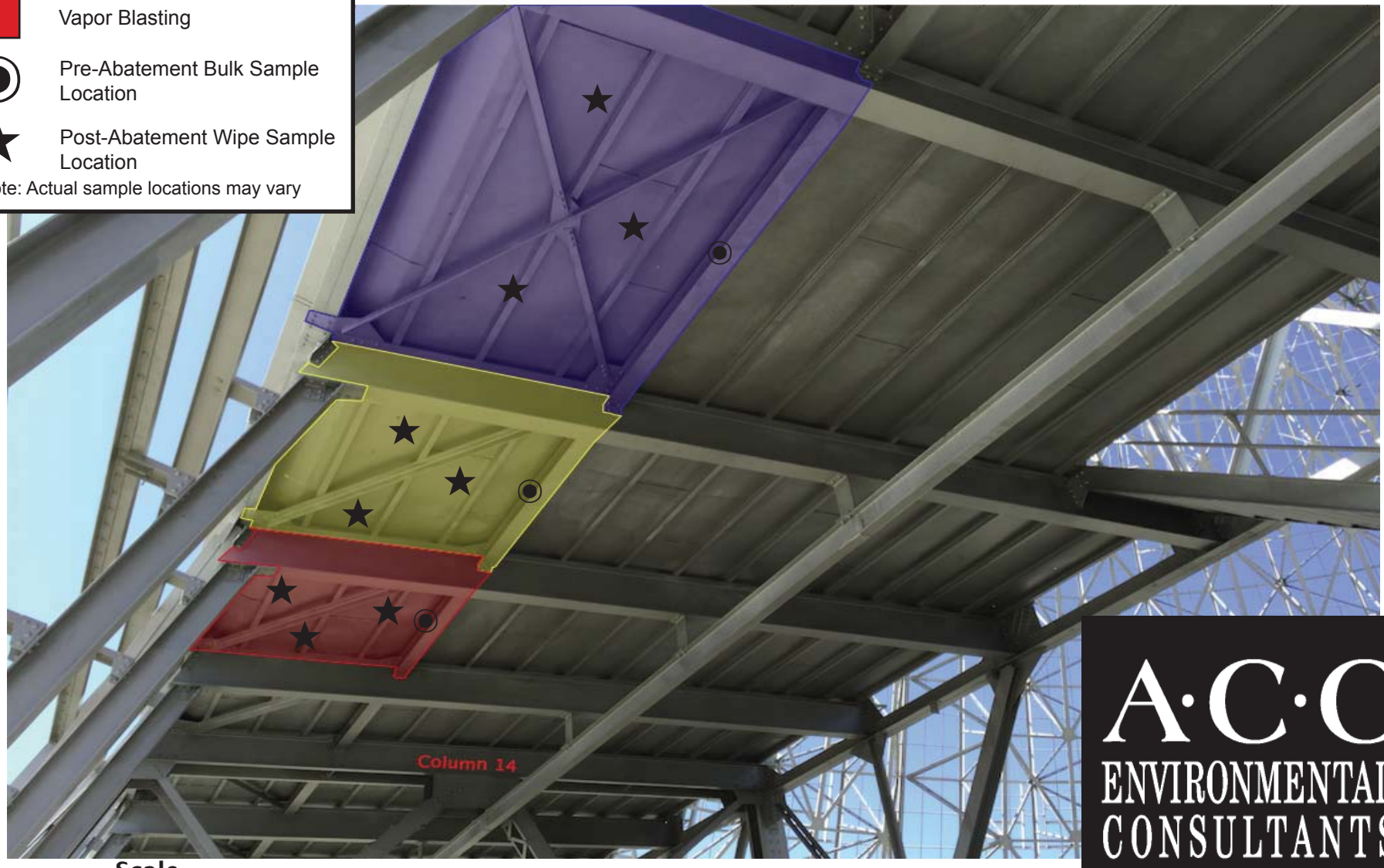
# Figure Number 3D

## Pilot Study Sample Location Map 3D
## Moffett Federal Airfield- Hangar One
## Mountain View, California

LEGEND

- Media Blasting
- High Pressure Water Blasting
- Vapor Blasting
- Pre-Abatement Bulk Sample Location
- Post-Abatement Wipe Sample Location

*Note: Actual sample locations may vary




Scale

0' 3' 6'

Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

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# Figure Number 3E

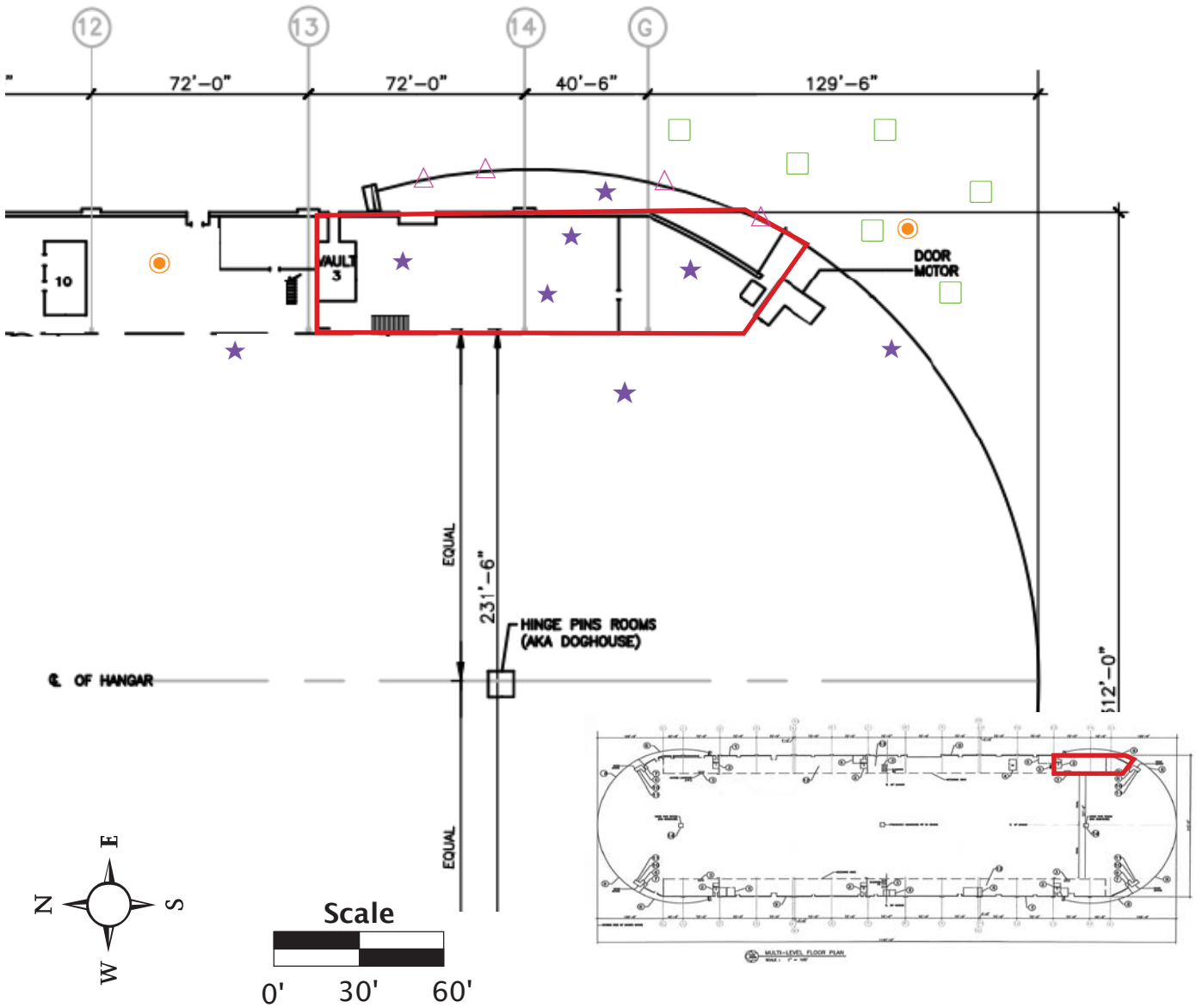
## Pilot Study Sample Location Map 3E
## Moffett Federal Airfield- Hangar One
## Mountain View, California

Legend

- Pre- and Post-Abatement Wipe Sample Location
- Pre-Abatement Soil Sample Location *
- Air Sample Location
- Pre-Abatement Trench Drain Wipe Sample Location

Note: Actual sample locations may vary

* Each of the locations shown in this figure is the approximate location of the center of an approximately 1,000 square foot area from which indicated multi-increment pre-abatement soil sample was collected.

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# Attachment A

# Consultant Site Specific Health and Safety Plan

**Pilot Study Health & Safety Plan**
Planetary Ventures
Hangar One, Moffett Federal Airfield
Mountain View, California

ACC Project Number 1591-001.01

**Prepared for:**

Planetary Ventures, LLC
Andrew W. Meade, P.E., MFA Director
CBRE/Global Corporate Services
600 Amphitheater Parkway, Mountain View CA. 94043
March 3, 2016

Prepared by: _____

Mark Sanchez, Vice President
Certified Asbestos Consultant #92-0082
CDPH Certified Inspector/Assessor, Supervisor, Monitor #5150

Reviewed by: _____ ___ ____

James Wilson
President
Certified Asbestos Consultant #06-4043

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**MOFFETT FEDERAL AIRFIELD**
**PLANETARY VENTURES**
**HANGAR ONE PILOT STUDY**
**SITE SAFETY & HEALTH PLAN**

MARCH 3, 2016

1.0: Site Specific Health and Safety Plan General Planning Requirements

This site safety and health plan is prepared in conformance with Section 6.2 of the Environmental Issues Management Plan ("EIMP") for the MFA Leasehold on the Former Naval Air Station Moffett Field, California, dated 3 March 2015, prepared by EKI and is consistent with State and Federal Occupational Safety and Health Administration (OSHA) standards for hazardous waste operations (California Coder of Regulations, Title 8, Section 5192 and 29 Code of federal Regulations 1910.120 respectively) and any other applicable health and safety standards.

This plan addresses the health and safety procedures to be followed by ACC employees and sub-contractors during field operations of the Planetary Ventures Hangar One Pilot Study. The plan is designed to comply with all applicable standards and regulations governing work performed at the following site:

Location: Hangar 1

Site Address: Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

The work may contact the following contaminants of concern known to be at the site: (Table 2 for details)

Chemical Concerns:

- Lead – in coatings
- PCBs – in coatings
- Asbestos – not known to be present within the work area but present at the site.

Other Physical hazards

- Ultra-High Pressure Water Blasting
- Media Blasting using Copper Slag
  Working at Elevation Fall Protection

Worker Training:

ACC shall staff the project with appropriately licensed and trained workers qualified to collect asbestos, lead/metals and PCBs as promulgated by Federal and State of California regulations. Specifically, ACC employees will have the following current licenses and training:

Table 1: Worker Designation – Qualification and Training Compliance Requirements

| Employee Designation | Qualifications | Training Compliance Requirements |
|---|---|---|
| Project Manager | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor/Monitor<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq<br>Cal-OSHA – 8 CCR 5192 |
| Sr. Site Technician(s): | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq<br>Cal-OSHA – 8 CCR 5192 |
| Safety Officer | DOSH: Certified Asbestos Consultant<br>CDPH: Lead Inspector/Assessor/Supervisor<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq<br>Cal-OSHA – 8 CCR 5192 |
| Site Technician | DOSH: Certified Site Surveillance Technician<br>CDPH: Lead Supervisor or Sampling Technician<br>40 Hour – HAZWOPER | 8 CCR 1529, Chapter 3.2, Article 2.6, Section 341.15<br>CDPH – 17 CCR 35001 et seq<br>Cal-OSHA – 8 CCR 5192 |

Note: No specific certifications for PCB's are established as of this Plan.
Additionally DPR is the qualified Site Safety and Health Authority for the Site. All ACC staff will attend the site-specific general safety orientation for the Hangar 1 Facility provided by DPR.

In addition to the training each person has received as a requirement of their qualifications all ACC personnel will be trained in the specific requirements that govern this work site including but not limited to the following:

DPR – Site Safety training including – working at heights and fall protection.

**Project-Specific Training**

Project-specific training and information will be provided either before traveling to the site or at the site before entry into the exclusion zone. The information and training will be documented and will include the following:

- The contents of this Health and Safety Plan
- A discussion of the health and safety hazards; protective measures and work practices for handling contaminated soil, water or equipment.

2.0: Site Specific Health and Safety Plan

2.1: General Information:

2.1.1 Work Site Location:

The proposed pilot scale abatement study area (Pilot Study Area) is located in the southeastern corner of Hangar 1 and is approximately 120 feet long by 30 feet wide by 20 feet high. Prior to the selection of this area for the proposed Pilot Study, ACC visually inspected the area for potential asbestos-containing materials and did not

observe any. A trench drain is located along the eastern edge of the study area and small area of bare soil is present east/southeast of the Pilot Study Area.

2.1.2 Objective and Scope of Work:

This work addressed by this Plan includes field execution of the proposed abatement Pilot Study which is designed to study multiple proposed methods to perform the removal of lead and PCB containing paints and coatings, from the existing Hangar One structure that will be removed during the proposed Hangar One redevelopment, re-skinning and renovations project. See (Work Plan for the Pilot Scale Abatement Study of Hangar 1, dated March 2016, prepared by ACC) for details.

Note: This project will not disturb soil or any sub-surface elements in the conduct of the planned work. Precautions are in place per the Work Plan to protect subsurface areas from any impact caused by the work. The Pilot Study will gather, analyze and report in-situ performance data that will identify conditions that will be encountered at the site before during and after the proposed abatement work of the entire structure.

2.1.3. ACC Scope of Work:

This Site Health and Safety Plan (HASP) provides health and safety procedure guidelines for field efforts associated with the baseline, perimeter and clearance sampling of lead and PCB-containing materials at the subject property as part of Hangar 1 Pilot Study. See Hangar 1 Pilot Study Abatement Work Plan for more details.

The objective of this HASP is to provide a generally safe working environment for personnel performing fieldwork and the general public at the subject property. Specific health and safety procedures associated with project activities and implementations of this HASP are the sole responsibilities of ACC and/or its subcontractors. All personnel performing fieldwork and onsite subcontractors are expected to be familiar with the requirements of this HASP.

This subject work addresses the planning and execution of the proposed abatement Pilot Study designed to study multiple proposed methods to perform the removal of lead and PCB containing paints and coatings from the existing Hangar One structure that will be removed during the proposed Hangar One redevelopment, re-skinning and renovations project. The Pilot Study will gather, analyze and report in-situ performance data that will identify conditions that will be encountered at the site before during and after the proposed abatement work of the entire structure.

ACC will perform project oversight and will gather data that will measure the effectiveness of proposed methods of removal in order to determine what constitutes the best methods to use to achieve the stated goals of the project. It is the goal of the Pilot Study to gather data through monitoring of contractor activities through observation and the use of various levels of sampling that will assist the design team's effort to determine the best practices, means and methods of abatement, worker and environmental protection, hazardous materials handling and disposal and appropriate monitoring applications that will then be applied to the design and conduct of the proposed wholesale abatement work at the site.

Work subject to this site-specific Health and Safety Plan relates to project oversight, inspections and collection of air, bulk and wipe samples for lead, PCBs, copper and particulates during the conduct of the project. The work will require ACC personnel to work inside of the proposed project work area containment and will also require ACC personnel to sample small amounts of the project related COC's.

The majority of work will take place at the ground level without the need for access to elevated areas above 25 feet. Sampling will be collected from the underside of the mezzanine and from certain elevated ground-floor horizontal structural steel components by use of Class I/IA rated ladders or aerial lifts positioned on the floor surface.

*All work will be conducted in accordance with 1) DPR's corporate IIPP and/or site safety rules, 2) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.* At no time will ACC work outside arms reach of the aerial platforms. ACC will not use aerial platform basket rails or roof-top handrails as a means to climb on/access work areas.

2.1.4 Key Personnel:

Table 2: Projected – Project Roster – Key Personnel

| Project Title | Personnel | Qualification |
|---|---|---|
| Principal: Sr. Project Manager | Mark Sanchez | CAC - 92-082<br>CDPH -Lead IA//M/S 5150<br>HAZWOPER"), 29 Code of<br>Federal Regulations 1910.120 |
| Field Project Manager: | Ben Schulte-Bisping | CDPH-Lead I/A-24564<br>HAZWOPER"), 29 Code of<br>Federal Regulations 1910.120 |
| Field Inspector/Safety Officer | Massoud Navvab | CAC – 98-2531<br>CDPH-I/A/M-8555 |
| Quality Assurance Manager (Sub-Contractor) | Jill B. Henes, | Ph.D., MBA, PMP |

2.1.5 Facility Site Background:

Hangar 1 is a large steel structure measuring approximately 1,133 feet long by 308 feet wide and 198 feet tall. Originally, the steel frame of Hangar 1 was covered with corrugated siding and a built-up asphalt roof, and the interior contained multi-story offices and shops located on both sides of the hangar deck, concrete electrical vaults, and a concrete floor. The area surrounding the hangar is paved, with the exception of several small areas of bare soil located on the eastern side of the hangar. A trench drain that discharges to the storm drain system surrounds the perimeter of Hangar 1.

**Prior Investigations and Remedial Actions**

In 1997, NASA observed the presence of Aroclor 1268, a relatively uncommon polychlorinated biphenyl (PCB) mixture, in a storm water settling basin that receives storm water runoff from the western portion of the former NAS Moffett Field. In 1999, Aroclor 1260 and 1268 were detected in a storm water sample collected from a manhole downstream of Hangar 1. Subsequent investigations, implemented between 1999 and 2002 by NASA, determined that the Hangar 1 siding, commercially known as Robertson Protected Metal, contained PCBs and asbestos and that the lead-based paint used to cover both the siding and steel frame of the hangar also contained PCBs. Bulk samples of the lower (gray) siding were found to contain Aroclor 1260 and 1268 at concentrations as high as 5,500 mg/kg and 35,000 mg/kg, respectively (Benchmark, 2003).

In 2003, NASA and the Navy completed Time-Critical Removal Actions (TCRAs) that consisted of removing contaminated sediments from the storm water collection trench and coating the corrugated siding with an asphalt emulsion to mitigate the migration of PCBs from the exterior surfaces of the hangar. Following the TCRAs, the Navy proposed to perform a Non-Time-Critical Removal Actions (NTCRA) to address the known PCB contamination present in the Hangar 1 siding materials as a more effective long-term remedy than the Navy's TCRA. Thirteen alternatives to mitigate the known PCB contamination at Hangar 1 and reduce the potential negative impacts to human health and the environment from these materials were evaluated in the Engineering Evaluation/Cost Analysis (EE/CA; Navy, 2008) for the NTCRA. The preferred alternative involved the following:

- the complete removal of the Hangar 1 siding;
- the deconstruction of interior structures;
- the removal of debris to appropriate off-site disposal or recycling facilities; and,

- the application of an epoxy coating (Carbomastic® 15; CM15) to the hangar's structural steel frame (AMEC,2013).

Implementation of the NTCRA began in June 2010 and was completed in December 2012. During the NTCRA, with the exception of the (1) structural steel frame; (2) CMU walls surrounding six electrical vaults, the former hazardous materials storage room, and former restrooms; and (3) the door operating mechanisms, all other areas of the hangar (e.g., the surface of the concrete floor, the top and bottom sides of the metal mezzanine decks and I-beams supporting the mezzanine decks, etc.) were remediated completely to remove all PCB contamination. In areas where PCBs remain (i.e., the structural steel frame, CMU walls, and door operating mechanisms), the structures were reportedly over coated with a CM15 epoxy coating (RORE, 2013; AMEC, 2013). The NTCRA completion efforts included a final wash down and decontamination of the Hangar 1 structural steel frame, concrete floor, and storm water conveyance trenches and drains.

Following implementation of the NTCRA, a Focused Feasibility Study (FFS; RORE, 2013) was prepared for the Navy to evaluate short- and long-term options to ensure the protectiveness of the NTCRA. The Navy's Proposed Plan for Hangar 1 (Navy, 2013) summarized the information detailed in the FFS and announced that the Navy's preferred alternative was the implementation of institutional controls.[1]

As paint containing PCBs and lead remain at Hangar 1, the Navy prepared a Long-Term Management Plan (LTMP) for PCB contamination at Hangar 1 (AMEC, 2013) that outlines the coating inspection and maintenance methods and procedures and storm water sediment monitoring program that are required to maintain the viability and effectiveness of the NTCRA; the LTMP has not been approved by the regulatory agencies. The LTMP indicated that these long-term management activities will present various job hazards to workers, including the potential for PCB exposure. In addition, the LTMP indicated that the owner/operator of the Hangar 1 structure would need to develop procedures for controlling the use of the hangar and that modifications to the hangar such as cutting, drilling, grinding, abrasion, welding, fastening, or impact that could damage the CM15 epoxy coating and expose the underlying PCB contamination.

In letters to the USEPA dated 23 October 2015 and 22 December 2015, NASA confirmed to the US EPA that NASA assumed the Navy's obligations with respect to IR Site 29 (defined as including the above ground frame and concrete floor of the Hangar); NASA has not accepted responsibility for any of the contamination that may exist in the soil or groundwater beneath Hangar 1. NASA and the USEPA have agreed to amend the November 2014 NASA FFA Site Management Plan to include the IR Site 29 obligations.

All ACC personnel will be provided a copy of this HASP and are required to follow site specific health and safety precautions at all times; if an unsafe condition is encountered, ACC personnel are empowered to mitigate the hazard if possible, otherwise all personnel are required to 1) vacate the hazard area to a safe distance, 2) notify others in the vicinity of the hazard and 3) notify the designated ACC Project Manager of the hazard for further review and to establish appropriate revised health and safety practices as necessary.

2.1.6 Map of Facility and Work Area:

See attached Figures 1 and 2.

2.1.7 Job Hazards Analysis/Hazards Mitigation:

The following hazard inventory has been reviewed for the site and planned activities; known or anticipated hazards at the site are marked under the "Present" column and related health and safety precautions are discussed within the document.

[1] Select potential institutional controls identified in the Navy's Proposed Plan (Navy, 2013) include: (1) installation and maintenance of signs notifying of the potential exposure hazard, (2) administrative arrangements for access for future monitoring/maintenance, (3) property owner and tenant commitment to inspection and maintenance of the CM15 epoxy coating, (4) sediment sampling, and (5) regulatory agency approval of building modifications that might damage the remedy components.

Table 3: Types of Contaminants and Other Hazards that may be encountered

| *Present* | *Potentially Hazardous Energies* | *Present* | *Falls From Elevated Surfaces (>6 feet)* |
|---|---|---|---|
| **X** | Pressurized Systems (pneumatic, hydraulic) | **X** | Open-sided / Exposed Elevated Work Surfaces |
| | Electrical, Overhead Electrical | | Floor Openings |
| | Kinetic (flywheels, pendulums, rotating shafts, etc.) | **X** | Use of Aerial Lifts / Elevating Work Platforms |
| | Potential (springs under tension, falling objects, etc.) | **X** | Use of Scaffold |
| | Explosives | *Present* | *General Hazards* |
| | Reactives / Corrosives | | High Temperatures / Heat Stroke / Heat Stress |
| | Infrared or UV Radiation | | Low Temperatures |
| | Lasers | | Over-bright Illumination (glare) or Poor Illumination |
| | Thermal (fire, heaters, cryogenic, dry ice, etc.) | **X** | Overhead Work, Utilities, Dropped Items |
| | Other: | **X** | High Noise, High Impact, Etc. (>90 dBA) |
| *Present* | *Environmental & Air Quality Hazards* | **X** | Portable Ladders |
| | Low Oxygen (<19.5%) | **X** | Slips / Trips, Wet / Oily Surfaces |
| **X** | Toxic Gases | | Hot Work (welding, brazing, torch cutting, etc.) |
| **X** | Toxic Liquids | | Confined Spaces |
| **X** | Asbestos Containing Materials (Outside Pilot Study Area) | | General Demolition Activities |
| **X** | Lead -containing Materials | | Engine Exhaust Emissions |
| | Other Regulated Metals: (cadmium, chromium, etc.) | | Excavation / Trenching / Engulfment |
| **X** | PCB Containing Materials | | Earthwork / Heavy Construction Equipment |
| | Mercury Contamination | **X** | Construction / Worksite / Vehicular Traffic |
| | Welding Fumes | | Use of Forklifts, etc. |
| | Flammable Vapors / Combustible Dusts (> 25% of LEL) | **X** | Equipment Pinch-points, etc. |
| | Nuisance Odor | | Impalement Hazards (rebar, etc.) |
| **X** | Nuisance Dust; Silica Dust; Abrasive Blasting | | Hot Pipes / Hot Surfaces |
| | Animal Allergens | | Electrical or Process Piping (LOBO / LOTO needed) |
| **X** | Emissions to Air | | Powder Actuated Tools |
| | Discharge to Storm/Process/Sanitary | | Stinging Insects, Wild Animals, etc. |
| **X** | Generation of Hazardous Wastes | | Biological (BBP, sewage, manure, droppings, etc.) |
| | Other: | | Other: |

Work subject to this site-specific Health and Safety Plan relates to project oversight, inspections and collection of air, bulk and wipe sampling of suspect lead, PCB-containing materials, copper and particulates during the conduct of the project. The work will require ACC personnel to work periodically inside of the proposed project work area containment and will also require ACC personnel to contact and sample small amounts of the project related COC's.

The majority of work will take place at the ground level without the need for access to elevated areas above 25 feet. Sampling will be collected from the underside of the mezzanine and from certain elevated ground-floor horizontal structural steel components by use of Class I/IA rated ladders or aerial lifts positioned on the floor surface. The aerial lift and related safety training will be provided by the Abatement Contractor in accordance with DPR Site HASP requirements.

*All work will be conducted in accordance with:*

1.) *DPR's corporate IIPP and/or site safety rules,*
*2.) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.*

*Note:* At no time will ACC work outside arms reach of the aerial platforms. ACC will not use aerial platform basket rails or roof-top handrails as a means to climb on/access work areas.

Physical and Other Hazards

Physical hazards associated with transportation to and from the site and site activities include those listed in the following table.

Table 4:

| Activity | Hazard | Control(s) |
|---|---|---|
| Driving to, on, and from the site | Striking pedestrians, runaway vehicles, striking structures, overturning vehicles. | Wear seat belts at all times while vehicles are in motion. Use licensed drivers. Define vehicle routes of travel. Obey driving regulations. Do not drive over holes or down sides of improperly sloped depressions. |
| Driving on the site | Runway activities and aircraft traffic | Drive along designated areas as directed by NASA Flight Operations, generally along the airfield side of the Hangar One perimeter fence.<br><br>Keep speeds no more than 15 MPH |
| General site activities | Stinging insects (bees, wasps, spiders) | Use care when sampling near dense vegetation. Be sure individuals allergic to insect bites (if any) have obtained prescription for insect-bite kit. |
| General Site Activities | Bird droppings/manure | Wear protective clothing, and air-purifying respirators as appropriate if the bird manure dust is bothersome. |
| General Site Activities | Slipping on wet or oily surfaces | Use wet methods to collect samples. Spray areas damaged by sampling with adhesive or encapsulant to hold down fibers. |
| General Site Activities | Heat Stress / Heat Stroke | Use of impermeable clothing reduces the cooling ability of the body because of evaporation reduction. This may lead to heat stress.<br><br>Hydrate & rest often<br><br>**See below for more information** |
| Collecting bulk lead and/or PCB samples | Inhalation of contaminated dusts | Use wet methods to collect samples. Spray areas damaged by sampling with adhesive or encapsulant to hold down dusts. |
| Using sharp tools | Cuts and punctures | Use extreme care when cutting or chipping samples with sharp instruments. Retract blades into containers, or hold blades and sharp tools away from body when walking. |
| Working at heights on ladders | Falls from heights, ladders slipping, dropping items | All ladders will be placed at a proper angle equal to 1 length of run for every 4 lengths of rise. Ladders will be tied off before work at heights greater than 20' will be attempted. Only Class I ladders with 300 lb. weight limits will be used. Weight limits on ladders will not be exceeded. Ladder footings will be level and on non-slippery surfaces before climbing. Individuals working under ladders will wear hard hats to protect against dropped objects. |
| Working at heights on elevated walkways, stairs and platforms | Falls from heights, ladders slipping, dropping items | Refer to the DPR Fall Protection HASP |
| Working at heights on elevated aerial lifts, boom lifts, scissor lifts | Falls from heights, ladders slipping, dropping items | Refer to the DPR Fall Protection HASP |
| Open holes in elevated decking; pits | Trips, falls | Refer to the DPR Fall Protection |

**Working at Elevated Heights, from A-frame Ladders, on Aerial Platforms, Near Fall Hazards or Floor Openings**

Refer to the attached DPR HASP requirements related to fall protection and working on elevated platforms.

*All work will be conducted in accordance with 1) DPR's corporate IIPP and/or site safety rules, 2) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.*

All ACC personnel shall have training pursuant to fall protection standards (29 CFR 1926.501-503 and 8 CCR 1669).

ACC Environmental Consultants, Inc. (ACC) staff assigned to the Moffett Field Hangar One PCB & Lead Sampling Activities have received appropriate fall protection training for work on elevated platforms, aerial lifts and similar site conditions pursuant to Federal and California Occupational Health Standards (29 CFR 1926.501-503 and 8 CCR 1669). Training has been completed within the last year and included coverage of the following topics:

- OSHA Fall Protection overview;
- Introduction to California-specific Fall Protection;
- Fall hazard recognition;
- Fall hazard elimination and controls methods;
- Applicable fall protection regulations;
- The responsibilities of designated persons;
- How to use written fall protection procedures;
- Inspection of equipment components and systems before use;

ACC personnel shall use the following fall protection equipment in conjunction with the DPR Fall Protection Plan:

- Body Harness (Protecta Model Number 1191995 or equivalent), capacity 310 pounds
- Fall Arrest Dual Lanyard, 6' Length (Condor Model 19F384), capacity 310 pounds

or

- Fall Arrest Lanyard (Single), 6' length (Protecta Model 1341001 or equivalent), capacity 310 pounds

**Heat Stress**

*Signs of Heat Stress.* "Heat stress" is a term that is used to describe progressively more serious symptoms, as follows:

- An initial rise in skin temperature due to increased blood flow to the skin (skin redness);
- Increase in heart rate, to more than 30 beats/minute above the resting level;
- Collapse, or heat exhaustion, due to inadequate blood flow to the brain;
- Dehydration, due to excessive sweating;
- Hyperventilation, resulting in a reduction of the normal blood carbon dioxide concentrations;
- Tingling around the lips, dizziness, cramping of muscles of hands and feet, and blackout; and
- "Heat stroke," characterized by unconsciousness, hot dry skin, and absence of sweating.

*Control of Heat Stress.* On hot, sunny days (high radiant heat load), if using impermeable work clothing, maintain appropriate work-rest cycles (progressively longer rest breaks in a cool location or the shade as temperature and work tasks increase) and drink water or electrolyte-rich fluids (Gatorade or equivalent) to minimize heat stress

effects. Impermeable clothing will only be worn when absolutely necessary for control of hazardous chemicals.

Also, when ambient temperatures exceed 70 degrees F, employees will conduct monitoring of their heart (pulse) rates, as follows:

- Each employee will check his or her own pulse rate at the beginning of each break period;
- Take the pulse at the wrist for 6 seconds, and multiply by 10; and
- If the pulse rate exceeds 110 beats per minute, then reduce the length of the next work period by one-third.

*Example:* After a one-hour work period at 80 degrees, a worker has a pulse rate of 120 beats per minute. The worker must therefore shorten the next work period by one-third, resulting in a work period of 40 minutes until the next break.

*Treatment of Heat Stress.* Individuals affected by mild forms of heat stress (heat exhaustion, dehydration, or cramping) should take a break in a cool or shaded location, drink liquids, and sit or lay down until feeling better. Shorter work periods should be used until temperature cools off.

Individuals affected by heat stroke are in critical condition. Summon emergency aid immediately, remove clothing, and bathe individual in cool water continually to bring down body temperature.

Pertinent Regulations:

Work of this project will be performed to be consistent with the following, State and Federal standards and regulations:

- Occupational Safety and Health Administration (OSHA) standards for hazardous waste operations (California Coder of Regulations (CCR), Title 8, Section 5192 and 29 Code of Federal Regulations (CFR) 1910.120 respectively).
- Cal-OSHA - 8 CCR 1532.1
- CDPH – Title 17 CCR 35001 et seq
- Cal-OSHA – 8 CCR 5192

2.1.8: Air Monitoring:

The following air samples will be performed during this project by ACC staff:

Volatiles:
ACC does not anticipate exposures to VOC's while at this site due to the nature of the work which will not disturb associated COCs and given the fact that the Hangar One structure is entirely open to air flow and makeup air entering the containment enclosure should not contain any COCs.

Particulate Air Sampling:
During active blasting activities (estimated to occur on a total of three days), perimeter air particulate concentrations will be monitored at two locations (one upwind and one downwind of the Pilot Study Area) using direct-read aerosol monitors equipped with PM10 impactor inlets (TSI AM510 or equivalent). On these days, ACC will routinely visit the perimeter air monitoring stations to monitor for the presence of visible dust and evaluate if abatement activities are resulting in elevated particulate concentrations around the site. If visible dust is observed or if PM10 Dust concentrations exceed 0.11 $mg/m^3$ for 5-minutes or more at either of the perimeter air monitoring stations, abatement activities will be temporarily terminated until the source of the particulates is identified and has been controlled. At the end of each day, data from the particulate aerosol monitors will downloaded and reviewed; average time-weighted concentrations will be compared against the PM10 Action Level.

In addition, during active blasting activities, air samples for PCBs, lead, and copper will be collected at these locations during the same period using personal air sampling pumps and filters as described in Section. Air samples for copper will only be collected on days where copper slag is used as the blast media. One duplicate sample will be collected from the downwind sampling location on the first day of active blasting activities.

The perimeter air samples will be submitted to a certified analytical laboratory under standard chain-of-custody procedures. At the analytical laboratory, the samples will be analyzed on an accelerated turn around time (TAT) and results will be provided to ACC within 24 hours of sample receipt by the analytical laboratory. Abatement activities will be suspended until the laboratory data has been received and the measured concentrations in the perimeter air samples have been compared to their respective Action Levels (see Section 3.7.6.1 of the Pilot Study Abatement Work Plan) to determine if additional mitigation measures are necessary.

2.1.9 Sampling Exposure Risk Analysis

Sampling of Lead-containing Coatings & Materials

As part of the Pilot Study ACC personnel will collect samples of Lead-containing Materials in accordance with Section. 3.7.3 of the Pilot Study Abatement Work Plan).

*Health effects from short-term overexposure to lead*

Lead poisoning can happen if a person is exposed to very high levels of lead over a short period of time. When this happens, a person may feel:

- Abdominal pain
- Constipated
- Tired
- Headachy
- Irritable
- Loss of appetite
- Memory loss
- Pain or tingling in the hands and/or feet
- Weak

Because these symptoms may occur slowly or may be caused by other things, lead poisoning can be easily overlooked. Exposure to high levels of lead may cause anemia, weakness, and kidney and brain damage. Very high lead exposure can cause death.

Lead can cross the placental barrier, which means pregnant women who are exposed to lead also expose their unborn child. Lead can damage a developing baby's nervous system. Even low-level lead exposures in developing babies have been found to affect behavior and intelligence. Lead exposure can cause miscarriage, stillbirths, and infertility (in both men and women).

Generally, lead affects children more than it does adults. Children tend to show signs of severe lead toxicity at lower levels than adults. Lead poisoning has occurred in children whose parent(s) accidentally brought home lead dust on their clothing. Neurological effects and mental retardation have also occurred in children whose parent(s) may have job-related lead exposure.

*Health effects from prolonged exposure to lead*

A person who is exposed to lead over time may feel:

- Abdominal pain

- Constipated
- Depressed
- Distracted
- Forgetful
- Irritable
- Nauseous/Sick

People with prolonged exposure to lead may also be at risk for high blood pressure, heart disease, kidney disease, and reduced fertility.

The Department of Health and Human Services (DHHS), Environmental Protection Agency (EPA), and the International Agency for Research on Cancer (IARC) have determined that lead is probably cancer-causing in humans.

If lead exposure is a possibility in your job, it is important that you understand how exposure occurs. This way you can take steps to lower your chances of being exposed.

*You can be exposed by breathing-in lead fumes or lead dust.*

Lead fumes are produced during metal processing, when metal is being heated or soldered. Lead dust is produced when metal is being cut or when lead paint is sanded or removed with a heat gun. Lead fumes and lead dust do not have an odor, so you may not know you are being exposed.

*You can be exposed by ingesting lead dust.*

Lead dust can settle on food, water, clothes, and other objects. If you eat, drink, or smoke in areas where lead is being processed or stored, you could ingest lead dust. Not washing your hands before you eat or touch your mouth are also ways you could ingest lead.
Though not always the case, ingested lead may leave a metallic taste in your mouth.

*You can be exposed by coming in contact with lead dust.*

Some studies have found lead can be absorbed through skin. If you handle lead and then touch your eyes, nose, or mouth, you could be exposed. Lead dust can also get on your clothes and your hair. If this happens, it's possible that you may track home some of the lead dust, which may also expose your family.

**Inhalation.** Exposure via this route could occur if large amounts of lead-containing dusts are encountered during sampling activities.

**Skin Contact.** Skin contact with lead is not considered a primary route of entry of those how handle lead.

**Ingestion.** Exposure via this route could occur if individuals eat, drink or perform other hand-to-mouth contact while conducting sampling.

*Lead Hazard Exposure Guidelines*

| **Substances** | **CAL OSHA PEL** |
|---|---|
| Lead (airborne) | 30 µg/m3 3039(Action Level)<br>50 µg/m3 (PEL) |

*Contamination Characterization*

It is not anticipated that baseline and clearance sampling activities will typically involve substantial disturbance of site contaminants. The highest potential for exposure to site contaminants is when ACC personnel periodically enter the containment work area during abatement activities. Personal protective equipment listed in Section 2.1.10 will be used.

Sampling of PCB containing Materials

Polychlorinated Biphenyls (PCBs) are poisonous to human health by three routes of exposure: inhalation, ingestion and skin contact. PCBs are a suspected carcinogen. The toxicity of the different types of PCBs increases as the content of chlorine increases. Workers shall wear appropriate personal protective clothing and equipment to protect themselves from potential exposure to all PCBs at all times during removal and disposal of PCB ballasts.

**Inhalation.** Exposure via this route could occur if large amounts of PCB containing dusts are encountered during sampling activities.

**Skin Contact.** Skin contact with PCBs are significant routes of entry of those materials.

**Ingestion.** Exposure via this route could occur if individuals eat, drink or perform other hand-to-mouth contact while conducting sampling.

*PCB Hazard Exposure Guidelines*

| **Substances** | **CAL OSHA PEL** |
|---|---|
| PCBs | 0.5 mg/m3 |

*Contamination Characterization*

It is not anticipated that baseline and clearance sampling activities will typically involve substantial disturbance of site contaminants. The highest potential for exposure to site contaminants is when ACC personnel periodically enter the containment work area during abatement activities. Personal protective equipment listed in Section 2.1.10 will be used.

2.1.10 Personal Protective Equipment (PPE)

The following personal protective equipment will be required AT ALL TIMES:

- Hard Hat
- Steel-toed shoes
- Safety glasses
- Gloves during sampling operations
- Shoe protective booties

The following additional personal protective equipment may be used during baseline, blasting activities and clearance sampling:

- Half –Face Negative Pressure Respirator, PAPR, Supplied Air Respirator
- Disposable Coveralls (e.g.Tyvek)
- Nitrile gloves
- Hearing Protection

Personnel performing sample collection and handling and inspections that require presence within the containment enclosure will be required to wear appropriate personal protective equipment as defined by the hazards present and project site requirements.

Persons collecting bulk and wipe samples of materials suspected of containing one of the known COCs at Hangar One will be protected, by wearing a half-face, air-purifying respirator or PAPR depending on the activity performed and conditions within the containment. All respirators will be equipped with High Efficiency Particulate Air (HEPA) filter cartridges. A higher level of respiratory protection (e.g., full-face respirator, PAPR, Supplied Air Respirator) may be chosen at the safety officer's discretion.

Employees expected to wear air-purifying respiratory protection will have current medical clearance (i.e. within one year) and will be fit-tested for the brand and model respirator they will be wearing during work. All use of respirators will be done in accordance with the Cal-OSHA Respiratory Protection Standard 8 CCR 5144.

2.1.11 Work Zones and Site Security Measures

**Site Access**

ACC Employees:
All employees and/or visitors to the project will be required to sign in and out with DPR's on-site foreman or superintendent responsible for the overall site Health & Safety Plan.

Authorized ACC visitors:
Visitors of ACC's should check in immediately upon arrival. Each visitor will be required to provide and wear the necessary protective equipment during visits and shall be escorted by supervisory personnel while onsite. All visitors, subcontractors and other personnel will be required to sign a safety HASP acknowledgment sheet certifying that they have read and will comply with the HASP. Failure to comply with this site entry procedure will result in expulsion from the Project.

2.1.12: Decontamination Measures:

Personnel Hygiene and Decontamination

*Respirators*
All respirators, shall be provided and maintained by the individual employer (ACC and subcontractors) for their own employees and shall be cleaned per regulatory requirements and inspected daily (if used) by the individual user.

*Disposable Materials*
All disposable clothing, gloves, expendable protective wear, used respirator cartridges, and other disposable material generated during site activities shall be placed in suitable plastic bags and disposed of at an appropriate off-site waste disposal receptacle.

*Personal Hygiene*
All site workers will thoroughly wash hands and face before eating, drinking, or performing other actions with a hand-to-mouth component. All workers entering the containment area where blasting work is occurring will use the three-stage decontamination system installed as part of the containment.

*Minimization of Contamination*
Do not kneel on contaminated ground, stir up unnecessary dust, or perform any practice that increases the probability of hand- to-mouth transfer of contaminated materials. Use plastic drop cloths and equipment covers where appropriate. Eating, drinking, chewing gum, smoking or using smokeless tobacco are forbidden while

collecting samples. Coordination with other site workers will be accomplished, as necessary, to avoid cross-contamination.

Equipment Decontamination

All sampling tools and non-disposable supplies shall be thoroughly washed and/or rinsed prior to storage after use.

2.1.13 General Safe Work Practices:

Personal Injury

In case of a minor personal injury, general first aid procedures should be implemented. A first aid kit will be available at the site at all times. More serious injuries may require assistance from paramedics.

The Project Manager or another designated person will contact the appropriate emergency personnel by dialing emergency numbers listed at the front of this document. The Project Manager or another designated person will contact ACC Management immediately following contact of medical personnel.

General Safety Equipment

The following equipment must be available and easily accessible for use
First Aid Kit, Fire extinguishers (Foam, dry chemical or carbon dioxide)

Waste Handling and Disposal

The waste handling procedures discussed in the work plan will be followed. Waste generated by implementation of this Health and Safety program may include spent protective clothing such as Tyvek suits, gloves and wash and rinse solutions. Protective clothing will be collected in a sealable plastic bag or other appropriate container. All waste materials will be included and disposed of with the abatement contractor's waste materials.

2.1.14 Medical Surveillance

All workers entering the site will have current medicals in accordance with the following regulations:

- Cal-OSHA Respiratory Protection Standard 8 CCR 5144
- Cal-OSHA Lead In Construction Standard 8 CCR 1532.1
- Cal-OSHA Hazwoper 8 CCR 5192

## EMERGENCY RESPONSE CONTACTS AND TELEPHONE NUMBERS

### Local Emergency Contacts

NASA Ames Police, Fire & Medical Emergencies ..... (650) 604-5555
NASA Ames Police, Fire & Medical Non-Emergency ..... (650) 604-5416
El Camino Hospital – Mountain View..... (650) 940-7000

### Hazardous Materials Information

NASA Ames Police, Fire & Medical Non-Emergency ..... (650) 604-5416
NASA Emergency Operations Center ..... (650) 604-2373
EHA-INFO..... (800) 342-4636
Toxline ..... (301) 496-1131
CHEMTREC (24-hour, emergency only) ..... (800) 424-9300
ORNL, Toxicology Information Response Center..... (615) 576-1743
National Response Center..... (800) 424-8802
Poison Control Center ..... (800) 682-9211

### ACC Environmental Consultants

ACC Environmental Oakland Office ..... (510) 638-8400
Mr. Jim Wilson .....(510) 638-8400 x106 -or- cell: (510) 773-7301
Mr. Mark Sanchez (Sr. Project Manager) .....(510) 638-8400 x 104 -or- cell: (510) 773-7303
Mr. Massoud Navvab (Safety Officer)……. (510) 638-8400 –or - cell: (510) 773-7282
Mr. Ben Schulte-Bisping (Field Supervisor)…….. (510) 638-8400 x 105 –or-cell: (510) 773-0708

### Client Contact

Mr. Andrew W. Meade, CBRE/ Planetary Ventures MFA Director ..... (408) 592-2899

### IN EMERGENCY, CALL FOR HELP AS SOON AS POSSIBLE

Give the following information:

- **Where you are** (cross streets or landmarks)
- **Phone number** you are calling from
- **What happened** - type of injury, accident
- **How many persons** need help
- **What is being done** for the victim(s)
- **You hang up last** - let whomever you called hang up first

Emergency Information
*IN CASE OF EMERGENCY, USE THIS SHEET*

Emergency Phone Number: (650) 604-5555 or 911

Site Address: Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

*accessible via the*

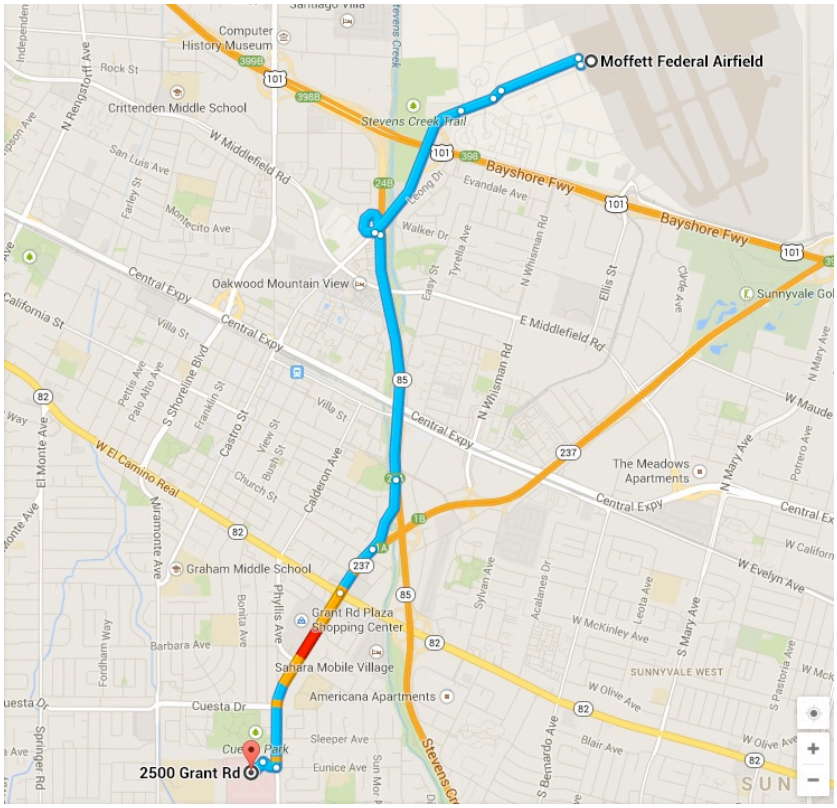
Moffett Field Airfield Operations Building
Cody Road & Edquiba Road
(650) 604-0685

**Directions to Nearest Emergency Hospital:**

El Camino Hospital – Mountain View
2500 Grant Road *(Emergency Department is accessible via South Drive)*
Mountain View, CA 94040
*(4 miles, 10 minutes)*

1. Take north Akron Road and Clark Road to Moffett Blvd (0.6 miles)
2. Take CA 85 South and Grant Road to South Drive (3.3 mi)
3. Turn Right onto South Drive (0.1 mi) into Hospital Emergency Parkibg

**EMERGENCY HOSPITAL ROUTE AND LOCATION MAP**



**Appendices**

Appendix A: DPR Site-specific Fall Protection Program
Appendix B: NASA Ames Health & Safety – Construction Safety Management (Chapter 27)
Appendix C: NASA Ames Health & Safety – Fall Protection (Chapter 40)

**SITE SAFETY REVIEW**
**Baseline, Perimeter and Clearance Lead/Metals & PCB Sampling as Part of the Pilot Study**

**General Information**

Date: _____ Time: _____

**Project:** **HANAGR ONE – ABATEMENT PILOT STUDY**

*Site Address:* Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

**Client Contact:** Mr. Andrew W. Meade, CBRE/ Planetary Ventures MFA Director ..... (408) 592-2899

**Objectives:** Baseline, Perimeter and Clearance Lead/Metals & PCB Sampling

**Topics Discussed:**

*Physical Hazards:* Typical hazards associated with scope of work activities and how to prevent potential employee exposure.

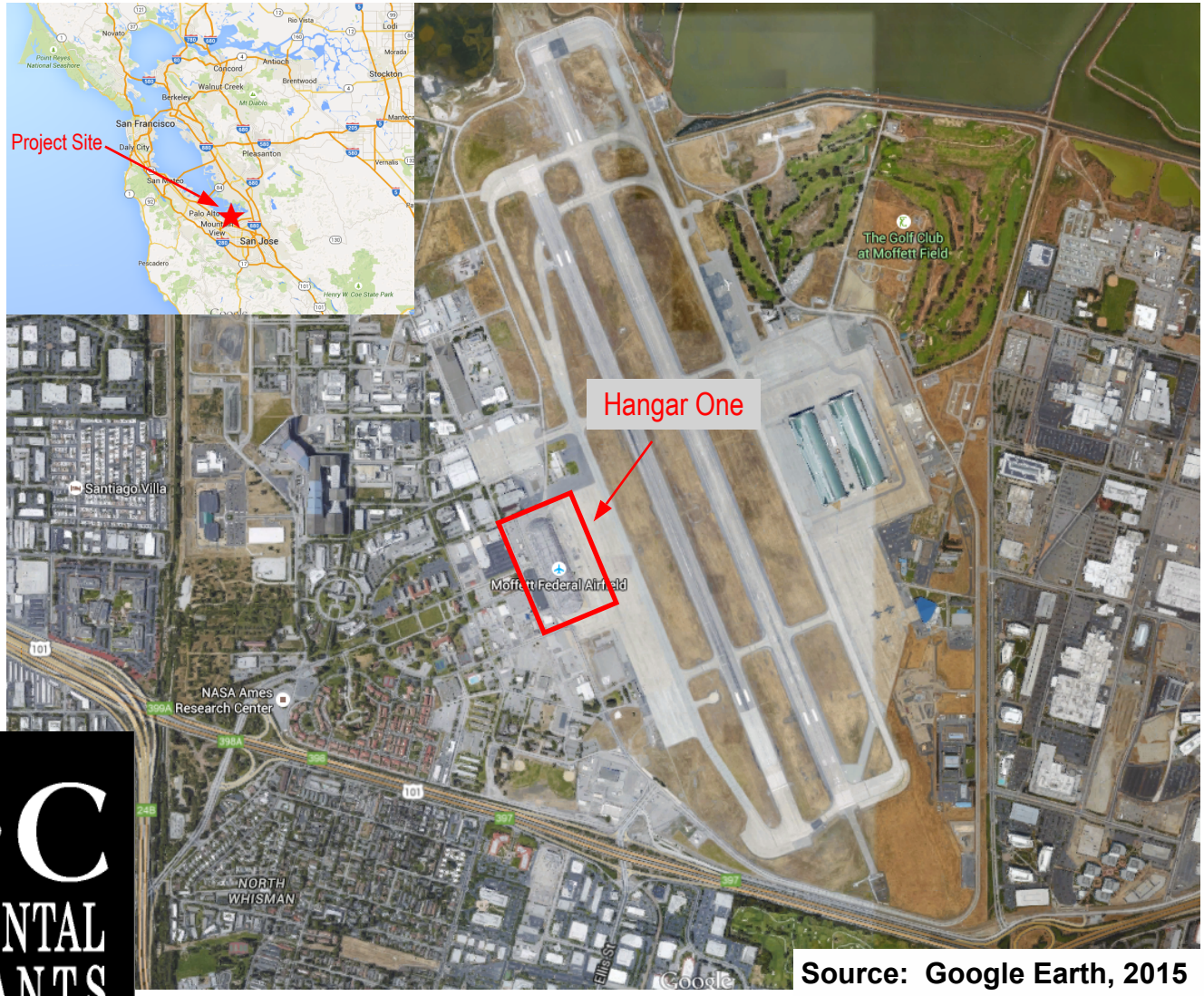
*Special Site Considerations:*
Potential for traffic hazards and physical hazards
Sampling Safety & Hygiene
Fall Protection
Working on Elevated Platforms

ATTENDEES

| Name (please print) | Signature |
|---|---|
| --- | --- |
| --- | --- |
| --- | --- |
| --- | --- |
| --- | --- |
| --- | --- |
| --- | --- |

# Figure Number 1

## Regional Location Map
## Moffett Federal Airfield- Hangar One
## Mountain View, California




Source: Google Earth, 2015

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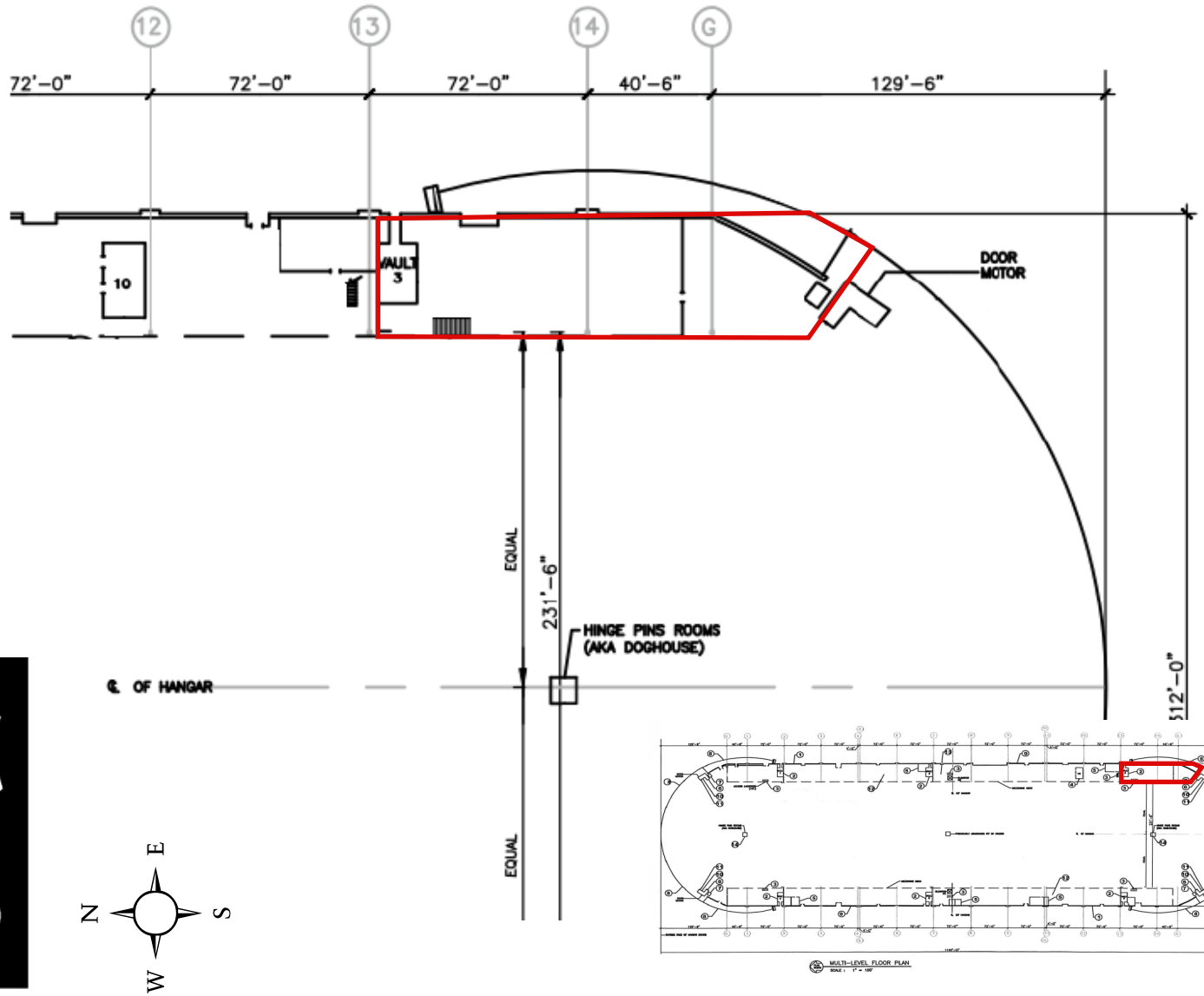
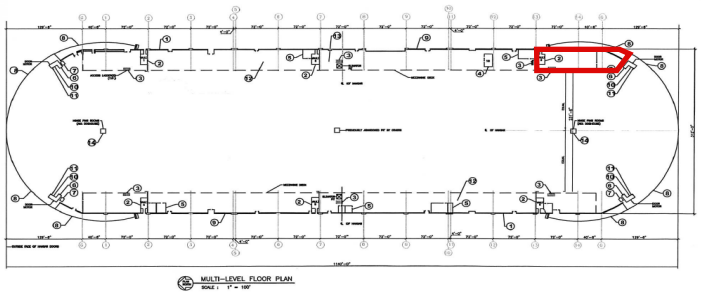
Scale: None Drawn By: JMS Date: 6/03/15 Project Number: 1591-001.02

# Figure Number 2

## Pilot Study Location Map
## Moffett Federal Airfield- Hangar One
## Mountain View, California

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# Attachment B

# Contractor Site Specific Health and Safety Plan

ECOBAY
SERVICES, INC

**Site Specific Health & Safety Plan**
Planetary Ventures
Hangar One, Moffett Federal Airfield
Mountain View, California

**Prepared for:**

Planetary Ventures, LLC
John-Michael Phelps, Project Manager
CBRE/Global Corporate Services
600 Amphitheater Parkway, Mountain View CA. 94043

January 27, 2016

**Prepared By:**

Bill Deadman
Eco Bay Services Inc.
1501 Minnesota St., San Francisco, Ca. 94107

Prepared by: _____
William Deadman / Project Manager

Reviewed by: _____
Padraic Ryan / COO

Reviewed by: _____
Trent Michels / President

1501 Minnesota Street * San Francisco, CA 94107 * Ph. 415- 643-7777 * Fax 415-643-7703

**MOFFETT FEDERAL AIRFIELD**
**PLANETARY VENTURES**
**HANGAR ONE PILOT STUDY**
**SITE SAFETY & HEALTH PLAN**

MARCH 3, 2016

This site safety and health plan addresses the safety and health procedures that will be followed during field operations at the following site and is in compliance with and subordinate to ACC Environmental Work Plan for the Pilot Study of Hanger 1, dated March 2016.

Site Address: Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

Description of Work:

Work subject to this site-specific Health and Safety Plan relates to select abatement of existing coatings containing lead/metals and PCB-materials associated with the Hangar One Pilot Study. Limited amount of coatings will be removed using:

- Ultra Hi Pressure Water Blasting – This process involves the removal of coatings using solely hi-pressure (20,000 to 30,000 PSI)
- Vapor / Media Blasting – New removal technology using water vapor combined with small amounts of copper blast media.
- Media Blasting – This method is very much like sand blasting using other media (typically copper media) solely. Eco Bay may use sand or walnut shell media with the same equipment to better determine most effective means and methods.

This work in no way will disturb soils or concrete floor.

The majority of work will take place at the ground level without the need for access to elevated areas. Certain removal will be from the accessible under deck of the mezzanine areas or from certain elevated ground-floor horizontal structural steel components by Class I/IA rated 6' ladders positioned on concrete ground floor surfaces. Additionally, limited setup and removal will be performed, using aerial lift platforms to approximately 25'. At no time will Eco Bay work outside arms reach of the aerial platforms or existing handrails. Eco Bay will not use aerial platform basket rails or roof-top handrails as a means to climb on/access work areas. *Abatement is expected to take up to 26 working days. Not including waste characterization and hauling.*

All work will be conducted in accordance with:

1) EKI's "Environmental Issues Management Plan (EIMP) for the MFA Leasehold on the Former Naval Air Station"
2) ACC's "Work Plan for the Pilot Scale Abatement Study of Hanger 1", Dated March 2016.
3) Eco Bay's corporate Health and Safety Manual (Applicable Sections Are Attached)
4) DPR's site specific safety plan and IIPP.

5) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.
6) Eco Bay's HASP is supplemental and complementary to the DPR HASP for the site. Where conflicting health and safety requirements are identified in the governing HASP documents, the more stringent will apply for the planned activities.

The primary PPE for this work will be Level D sturdy work boots, hard hats and reflective safety vest will be worn in the work area. During abatement activities, full body disposable Tyvek suits, and disposable gloves.

When there is a potential to disturb Lead / PCB containing materials, Eco Bay Employees will wear respiratory protection as stipulated (see attached Respiratory Protection Plan).

## Site Hazard Summary

The following hazard inventory has been reviewed for the site and planned activities; known or anticipated hazards at the site are marked under the "Present" column and related health and safety precautions are discussed within the document.

All **Eco Bay** personnel are required to follow site specific health and safety precautions at all times; if an unsafe condition is encountered, **Eco Bay** personnel are empowered to mitigate the hazard if possible, otherwise all personnel are required to 1) vacate the hazard area to a safe distance, 2) notify others in the vicinity of the hazard and 3) notify the designated **Eco Bay** Project Manager of the hazard for further review and to establish appropriate revised health and safety practices as necessary.

**Site Name:** **MFA Hangar One**
**Scope of Work:** **Limited Removal of Coatings Containing PCBs & Lead**

**[Check appropriate box related to your work – add anything not covered in Other]**

| *Present* | *Potentially Hazardous Energies* | *Present* | *Falls From Elevated Surfaces (>6 feet)* |
|---|---|---|---|
| | Pressurized Systems (pneumatic, hydraulic) | | Open-sided / Exposed Elevated Work Surfaces |
| x | Electrical, Overhead Electrical | | Floor Openings |
| | Kinetic (flywheels, pendulums, rotating shafts, etc.) | x | Use of Aerial Lifts / Elevating Work Platforms |
| | Potential (springs under tension, falling objects, etc.) | | Use of Scaffold |
| | Explosives | *Present* | *General Hazards* |
| | Reactives / Corrosives | | High Temperatures / Heat Stroke / Heat Stress |
| | Infrared or UV Radiation | | Low Temperatures |
| | Lasers | | Over-bright Illumination (glare) or Poor Illumination |
| | Thermal (fire, heaters, cryogenic, dry ice, etc.) | | Overhead Work, Utilities, Dropped Items |
| | Other: | | High Noise, High Impact, Etc. (>90 dBA) |
| *Present* | *Environmental & Air Quality Hazards* | x | Portable Ladders |
| | Low Oxygen (<19.5%) | X | Slips / Trips, Wet / Oily Surfaces |
| | Toxic Gases | | Hot Work (welding, brazing, torch cutting, etc.) |

| | | | |
|---|---|---|---|
| | Toxic Liquids | | Confined Spaces |
| | Asbestos Containing Materials | | General Demolition Activities |
| x | Lead -containing Materials | | Engine Exhaust Emissions |
| | Other Regulated Metals: (cadmium, chromium, etc.) | | Excavation / Trenching / Engulfment |
| x | PCB Containing Materials | | Earthwork / Heavy Construction Equipment |
| | Mercury Contamination | x | Construction / Worksite / Vehicular Traffic |
| | Welding Fumes | x | Use of Forklifts, etc. |
| | Flammable Vapors / Combustible Dusts (> 25% of LEL) | x | Equipment Pinch-points, etc. |
| | Nuisance Odor | | Impalement Hazards (rebar, etc.) |
| | Nuisance Dust; Silica Dust; Abrasive Blasting | | Hot Pipes / Hot Surfaces |
| | Animal Allergens | | Electrical or Process Piping (LOBO / LOTO needed) |
| x | Emissions to Air | | Powder Actuated Tools |
| | Discharge to Storm/Process/Sanitary | | Stinging Insects, Wild Animals, etc. |
| X | Generation of Hazardous Wastes | | Biological (BBP, sewage, manure, droppings, etc.) |
| X | Other: Hi Pressure Water & Media | | Other: |

## EMERGENCY RESPONSE CONTACTS AND TELEPHONE NUMBERS

### Local Emergency Contacts

NASA Ames Police, Fire & Medical Emergencies.....(650) 604-5555
NASA Ames Police, Fire & Medical Non-Emergency.....(650) 604-5416
El Camino Hospital – Mountain View ..... (650) 940-7000

### Hazardous Materials Information

NASA Ames Police, Fire & Medical Non-Emergency.....(650) 604-5416
NASA Emergency Operations Center.....(650) 604-2373
EHA-INFO .....(800) 342-4636
Toxline.....(301) 496-1131
CHEMTREC (24-hour, emergency only).....(800) 424-9300
ORNL, Toxicology Information Response Center.....(615) 576-1743
National Response Center.....(800) 424-8802
Poison Control Center.....(800) 682-9211

### *Eco Bay Services Inc.*

Office .....(415) 643-7777
Principal in Charge (Bill Deadman) ..... (Office Ext 108)-or- cell: (925) 808-8901
Bea Reynolds (Safety Officer)..... (Office Ext 115) - or- cell: (707) 372-3591
Mauricio Parra (Field Supervisor)…….. ..... -cell: (415) 730-6155

### Client Contact

John-Michael Phelps, CBRE/ Planetary Ventures Project Manager.....

## IN EMERGENCY, CALL FOR HELP AS SOON AS POSSIBLE

Give the following information:

- **Where you are** (cross streets or landmarks)
- **Phone number** you are calling from
- **What happened** - type of injury, accident
- **How many persons** need help
- **What is being done** for the victim(s)
- **You hang up last** - let whomever you called hang up first

## Emergency Information

*IN CASE OF EMERGENCY, USE THIS SHEET*

Emergency Phone Number: (650) 604-5555 or 911

Site Address: Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

*accessible via the*

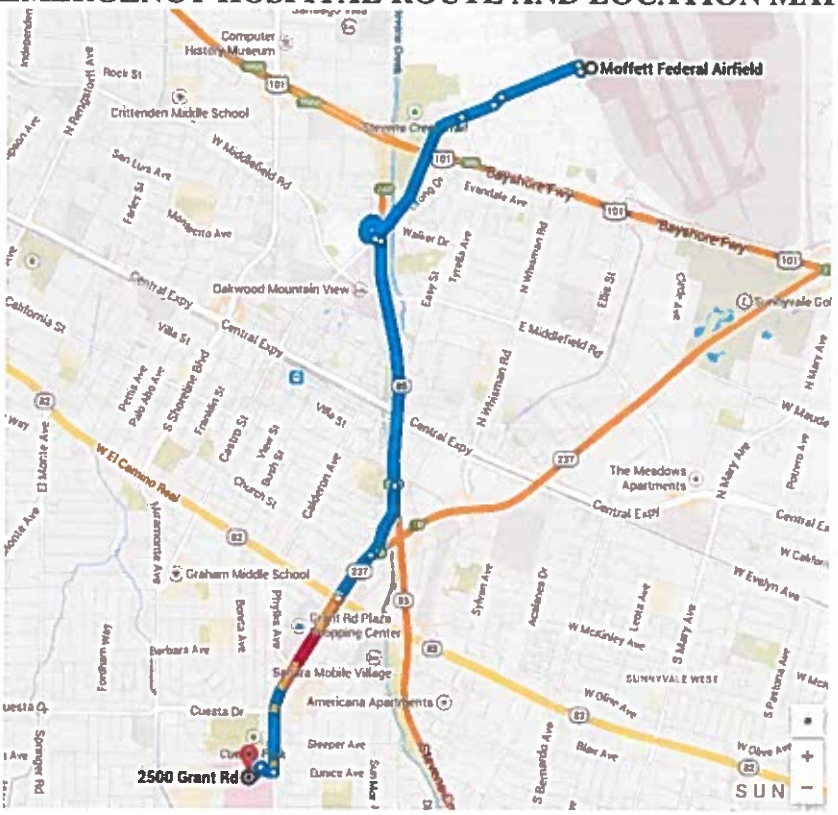
Moffett Field Airfield Operations Building
Cody Road & Edquiba Road
(650) 604-0685

**Directions to Nearest Emergency Hospital:**

El Camino Hospital – Mountain View
2500 Grant Road *(Emergency Department is accessible via South Drive)*
Mountain View, CA 94040
*(4 miles, 10 minutes)*

1. Take north Akron Road and Clark Road to Moffett Blvd (0.6 miles)
2. Take CA 85 South and Grant Road to South Drive (3.3 mi)
3. Turn Right onto South Drive (0.1 mi) into Hospital Emergency Parkibg

**EMERGENCY HOSPITAL ROUTE AND LOCATION MAP**



## 1.0 Introduction

This Site Health and Safety Plan (HASP) provides health and safety procedure guidelines for field efforts associated with Hanger One Pilot Study

The objective of this HASP is to provide a generally safe working environment for personnel procedures associated with project activities and implementations of this HASP are the sole responsibilities of Eco Bay Services and/or its subcontractors. All personnel performing fieldwork and onsite subcontractors are expected to be familiar with the requirements of this HASP.

Work subject to this site-specific Health and Safety Plan relates to abatement of existing coatings of lead/metals and PCB-containing materials associated with the Hangar One Pilot Study. The majority of work will take place at the ground level without the need for access to elevated areas. Some removal will be from underside of deck of the mezzanine areas and from elevated ground-floor horizontal structural steel components. In some areas, access by Class I/IA rated 6' ladders positioned on concrete ground floor surfaces will be needed. Additionally, limited setup and removal will be performed, using aerial lift platforms to approximately 25'. At no time will Eco Bay work outside arms reach of the aerial platforms or existing handrails. Eco Bay will not use aerial platform basket rails or roof-top handrails as a means to climb on/access work areas. All work will be in compliance with Eco Bay fall protection plan (attached)

*Abatement related activities are expected to take up to 25 working days.*

*All work will be conducted in accordance with 1) Eco Bay's corporate Health & Safety Plan, 2) DPR site safety rules, 3) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements 4) EKI EIMP.*

*Eco Bay HASP is supplemental and complementary to the DPR HASP for the site. Where conflicting health and safety requirements are identified in the governing HASP documents, the more stringent will apply for the planned activities.*

### 1.1 Qualification and Related Training for Eco Bay and its Subcontractors

Strict Safety and Security requirements apply at this job site. All Eco Bay employees and its subcontractors shall attend site-specific safety training provided by the General Contractor (DPR). DPR manages access and orientation to the site to all site users and has responsibility for monitoring all visitors to the site. Eco Bay does not anticipate visitors to the site who are not presently oriented and qualified to be present. Eco Bay bears the responsibility for ensuring that all its staff as well as its subcontractor's staff are appropriately trained and equipped to perform the work described in this work plan.

Table 1 and Table 2 provide details regarding the qualifications for Eco Bay, its employees and our subcontractors. In addition to these stated requirements all personnel shall be members in good standing of appropriate union.

**Table 1: List of Contractors with Qualifications by Division of Work**

| Division of Work | Contractor | Qualifications | License / Certification Number |
|---|---|---|---|
| Abatement Work | Eco Bay Services Inc. | A General Engineering<br>B General Contracting<br>C-2 Insulation<br>C-21 Demolition<br>ASB Asbestos Abatement<br>HAZ Hazardous Waste<br>DOSH Certificate | CA State Lic. Board No. 912328<br><br>Dosh #933 |
| Scaffold | Safway Services LLC | D-39 Scaffold | Ca. State Lic Board No. 972968 |
| Re-Coating | C & O Painting | C – 33 Painting | Ca. State Lic Board No. 501260 |
| Coating Inspector | CTS (Construction Testing Services) | N/A | N/A |
| Waste Hauler | World Environmental & Energy | N/A | EPA ID # CAR000037283 |
| Waste Disposal Site #1 CAL HAZ & RCRA | Clean Harbors Buttonwillow Landfill | N/A | EPA ID # CAD980675276 |
| Waste Disposal Site #2 – NON HAZ | Recology Hay Road | N/A | EPA ID# CAD982042475 |

**Table 2: Abatement Contractor Personnel Qualifications and Requirements**

| Employee Description | Qualification | Compliance Requirements | Est. No per category |
|---|---|---|---|
| Project Supervisor | AHERA Asbestos Contractor/Supervisor<br><br>CDPH Lead Supervisor<br><br>40 HR. Hazwopper | 8 CCR 1529<br>CDPH – 17 CCR 35001 et seq and Cal OSHA 8 CCR 1532.1<br>Cal OSHA 8 CCR 5192 | 1 to 3 |
| Laborer | AHERA Asbestos Worker<br><br>CDPH Lead Worker<br><br>40 HR. Hazwopper | 8 CCR 1529<br>CDPH – 17 CCR 35001 et seq and Cal OSHA 8 CCR 1532.1<br>Cal OSHA 8 CCR 5192 | 2 to 8 |

## 2.0 Pilot Study Abatement Activities

Activities include; 1)Pre-Abatement work include; pre-cleaning and erection of negative pressure containment with attached decontamination facilities, 2) removal of coatings using three different methods, 3) final cleaning of; substrate, containment and equipment 4) subcontractors. All work will be done in co-ordination and compliance with DPR, ACC, EKI and all Regulatory Agencies.

### 2.1 Pre – Abatement Activities

Prior to conducting abatement activities the entire work area will need to be cleaned. Debris and surficial sediments on the concrete slab, around the structural steel members and within the trench drains adjacent to the Pilot Study Work Area will be removed using vacuums equipped with high-efficiency particulate air (HEPA) filters. HEPA vacuuming of the concrete floor below work area will be performed to remove debris that could puncture the plastic/membrane and/or containment floor and cause water leaks. Any pre-cleaning that may disturb the surface being cleaned will be subject to the worker protection, containment and waste handling and disposal requirements.

Upon completion of HEPA vacuuming the concrete floors within the Pilot Study Area and the performance of pre-abatement sampling (by ACC) of the concrete floor, a fully encapsulating enclosure will be constructed around the Pilot Study Area as described in Section 2.1.1. The enclosure will be sealed at floor connections in such a manner that no residual waste water will be allowed to migrate from within.

#### 2.1.1 Enclosure Set-up

A fully encapsulated negative pressure enclosure will be installed around the Pilot Study Area by Eco Bay. This enclosure will be constructed of shrink wrap polyethylene and polyvinyl chloride (PVC). The polyethylene will be a fire-retardant reinforced material capable of handling expected wind loads, interior media blasting activities, exposure to the elements (e.g. 12 mil polyethylene) and will be used to enclose all areas above the concrete floor. Seams will be staggered, overlapped, tapped and heat sealed. The PVC floor of the enclosure will be installed in such a way that there is adequate vertical rise to capture and contain all anticipated water and/or media wastes that may accumulate during abatement activities.

A Three Stage decontamination chamber with a shower and a water tight pan will be connected to the enclosure for the decontamination of personnel and a two-stage decontamination chamber will be connected to the enclosure for the decontamination of equipment and to serve as a waste out.

## 2.2 Removal of Coatings

The following sections outline Eco Bay's approach to removal of coatings including layers that include Lead and PCB containing materials. Eco Bay will use three methods to remove coatings; Media Blasting, Ultra Hi-Pressure Water Blasting and Vapor Media Blasting. Each method will be used in several different locations on differing types of still and thickness of coating to determine which method is a) Most Effective, b) Safest and c) Produces the least impact to landfills and environment in general. Attached to this document we have included applicable sections of Eco Bay's Corporate Health and Safety Manual including the Section entitled "Lead and Asbestos Abatement Criteria".

### 2.2.1 Media Blasting

This method involves use of compressor, media pot and hoses. Working in three-person team(s) with one person filling the pot and managing the compressor outside of containment, one directing the nozzle at the area to be abated typically working from 4" to 12" away from the affected area and one person in containment with the person directing the nozzle, this person's role is support with moving hoses and equipment as well as watch for potential hazards or concerns being able to stop work as soon as they see something of concern. The person on the nozzle will typically use enhanced face and respiratory PPE such as a blasting hood attached to supplied air or a PAPR respirator used in conjunction with head and full face protection. Upon completion, spent media and removed coatings will be vacuumed up using HEPA equipped vacuums. Waste produced from this activity will be placed in 55 gallon drums and sealed awaiting characterization testing (ACC will provide).

### 2.2.2 Ultra Hi-Pressure Water Blasting

For this removal method, Eco Bay has employed KR Surface Industries to provide equipment and one equipment operator. Equipment consist of one NLB pump with attached compressor (NLB Cut Sheets are attached) which remain outside of containment. The pump is capable of producing pressure of from 20,000 to 40,000 psi using 4 to 8 gallons per minute. Total water use for pilot study is anticipated to be approximately 500 gallons. Working in a three-person team, including KRSI operator manning the pump, one directing the nozzle at the area to be abated typically working from 4" to 12" away from the affected area and one person in containment with the person directing the nozzle, this person's role is support with moving hoses and equipment as well as watch for potential hazards or concerns being able to stop work as soon as they see something of concern. The person on the nozzle will typically use enhanced face and full body Kevlar protective equipment. Upon completion, water and removed coatings will be vacuumed up using "wet" vacuums. Waste produced from this activity will be placed in 55 gallon drums and sealed awaiting characterization testing (ACC will provide).

### 2.2.3 Vapor . Media Blasting

This removal technique uses a new type of equipment joining both media and water for effective removal. The benefit is the water pressure and flow are significantly reduced thereby being much safer and using less water (< 20 gallons per day). The amount of media (typical copper slag) is also reduced to approximately 5% of what is used in typical media blasting This method does still require a three-person team but no special PPE is required.

### 2.3 Cleaning of; Substrate, Containment and Equipment

After removal of coatings, Eco Bay Employees will wet wipe the structural steel in preparation for visual and wipe sampling by ACC.

After all removal work has been completed and passed testing by ACC, Eco Bay will; Pick up and bag any remaining debris including; suits, poly, tape and misc debris. Containment and all steel and exposed concrete inside containment will be rinsed using low pressure water the resulting waste water will be collected in drums.

All equipment used inside containment will at this time be rinsed and wet wiped and removed from the containment.

A light encapsulant that is compatible with the new coatings will be sprayed on all remaining surfaces.

Eco Bay will turn containment over to ACC for final testing.

### 2.4 Subcontractors

Eco Bay has three subcontractors to be used on this project; Safway Scaffold, KR Surface Industries and C & O Painting. All three subcontractors are properly licensed and insured and have worked with Eco Bay and / or DPR on many occasions. Site specific Certificates of Insurance meeting PV / NASA and DPR requirements will be provided in advance of work.

Safway Scaffold will provide limited scaffold (see attached drawing) to support the containment and to provide access to some of the higher elevations (+/- 20') of work.

KR Surface Industries will supply ultra hi-pressure water equipment with one operator for one day only. Equipment cut sheets information is attached.

C & O Painting will re-apply pre-approved coating in locations where coatings were removed. Material cut sheet and MSDS are forthcoming.

CTS will provide inspection and certification of C&O application.

**Table 3: Per Task Schedule**

| Task | Description | Start Date | End Date | Approximate Duration |
|---|---|---|---|---|
| **Pre Abatement Activities** | | | | |
| Pre-Construction Meeting | Attendees: DPR, EKI, ACC & Eco Bay | 1 | 1 | 1 Day |
| Mobilization | Eco Bay will mobilize; personnel, equipment and materials | 2 | 3 | 1-2 Days |
| Pre-Cleaning | Eco Bay will remove debris and surficial sediments from study area | 3 | 4 | 1-2 Days |
| Erect Enclosure | Erect enclosure using shrink wrap and pvc. | 3 | 8 | 4-6 Days |
| Decontamination Facilities | Erect human, waste and equipment decontamination facilities | 6 | 8 | 2 Days |
| **Abatement Activities** | | | | |
| Enclosure | Install final seals and negative air machines | 9 | 11 | 3 Days |
| Media Blast | Remove select areas using media blast & clean area, | 12 | 13 | 2 Days |
| Ultra Hi-Pressure Water | Remove select areas using ultra hi-pressure water & clean area | 14 | 15 | 2 Days |
| Vapor Blast | Remove select areas using, Vapor Blast Equipment & clean area. | 16 | 18 | 2 Days |
| Additional Removal or Testing as Requested | Remove select areas using means and methods requested by owner, clean and assist ACC Testing* | 19 | 20 | 2 Days |
| **Post Abatement Activities** | | | | |
| Cleaning and Decontamination | Final decontamination of enclosure and all equipment within enclosure | 21 | 22 | 2 Days |
| Removal of Enclosure | All Poly and other materials will be taken down and placed in bins for waste testing and disposal. Equipment will be removed from site | 23 | 25 | 3 Days |
| Waste Removal | After waste characterization, manifest approval and proper containerization, Eco Bay will return to Site and Load out waste to appropriate waste hauler / disposal site. | Ph 2 1 | 1 | 1 Day |
| **Final De-Mobilization** | Final removal of all equipment related to pilot study | Ph 2 2 | 2 | 1 Day |

**Note: Each removal method will only take one day. Two days are included for all methods as that includes ACC testing and out setup and equipment decontamination.**

## 3.0 Health and Safety Hazards

Health and safety concerns on site may consist of:

- Chemical hazards:
  Lead / Metals
  PCBs

- Other Physical hazards (see Section 4.2 Table)
  Working at Elevation
  Fall Protection
  Hi pressure water / media

## 3.1 Removal of Lead-containing Coatings & Materials

*Health effects from short-term overexposure to lead*

Lead poisoning can happen if a person is exposed to very high levels of lead over a short period of time. When this happens, a person may feel:

- Abdominal pain
- Constipated
- Tired
- Headachy
- Irritable
- Loss of appetite
- Memory loss
- Pain or tingling in the hands and/or feet
- Weak

Because these symptoms may occur slowly or may be caused by other things, lead poisoning can be easily overlooked. Exposure to high levels of lead may cause anemia, weakness, and kidney and brain damage. Very high lead exposure can cause death.

Lead can cross the placental barrier, which means pregnant women who are exposed to lead also expose their unborn child. Lead can damage a developing baby's nervous system. Even low-level lead exposures in developing babies have been found to affect behavior and intelligence. Lead exposure can cause miscarriage, stillbirths, and infertility (in both men and women).

Generally, lead affects children more than it does adults. Children tend to show signs of severe lead toxicity at lower levels than adults. Lead poisoning has occurred in children whose parent(s) accidentally brought home lead dust on their clothing. Neurological effects and mental retardation have also occurred in children whose parent(s) may have job-related lead exposure.

*Health effects from prolonged exposure to lead*

A person who is exposed to lead over time may feel:

- Abdominal pain
- Constipated
- Depressed
- Distracted
- Forgetful
- Irritable
- Nauseous/Sick

People with prolonged exposure to lead may also be at risk for high blood pressure, heart disease, kidney disease, and reduced fertility.

The Department of Health and Human Services (DHHS), Environmental Protection Agency (EPA), and the International Agency for Research on Cancer (IARC) have determined that lead is probably cancer-causing in humans.

If lead exposure is a possibility in your job, it is important that you understand how exposure occurs. This way you can take steps to lower your chances of being exposed.

*You can be exposed by breathing-in lead fumes or lead dust.*

Lead fumes are produced during metal processing, when metal is being heated or soldered. Lead dust is produced when metal is being cut or when lead paint is sanded or removed with a heat gun. Lead fumes and lead dust do not have an odor, so you may not know you are being exposed.

*You can be exposed by ingesting lead dust.*

Lead dust can settle on food, water, clothes, and other objects. If you eat, drink, or smoke in areas where lead is being processed or stored, you could ingest lead dust. Not washing your hands before you eat or touch your mouth are also ways you could ingest lead.
Though not always the case, ingested lead may leave a metallic taste in your mouth.

*You can be exposed by coming in contact with lead dust.*

Some studies have found lead can be absorbed through skin. If you handle lead and then touch your eyes, nose, or mouth, you could be exposed. Lead dust can also get on your clothes and your hair. If this happens, it's possible that you may track home some of the lead dust, which may also expose your family.

**Inhalation.** Exposure via this route could occur if large amounts of lead-containing dusts are encountered during sampling activities.

**Skin Contact.** Skin contact with lead is not considered a primary route of entry of those how handle lead.

**Ingestion.** Exposure via this route could occur if individuals eat, drink or perform other hand-to-mouth contact while conducting sampling.

*Lead Hazard Exposure Guidelines*

| Substances | CAL OSHA PEL |
|---|---|
| Lead (airborne) | 30 µg/m3 (Action Level)<br>50 µg/m3 (PEL) |

***Contamination Characterization***

It is not anticipated that lead survey activities will typically involve substantial disturbance of other site contaminants. However, if inspection personnel have the potential for exposure to other site contaminants, protective clothing listed elsewhere in this plan (paragraph 3.6) will be followed.

## 3.2 Abatement of PCB containing Materials

Polychlorinated Biphenyls (PCBs) are poisonous to human health by three routes of exposure: inhalation, ingestion and skin contact. PCBs are a suspected carcinogen. The toxicity of the different types of PCBs increases as the content of chlorine increases. Workers shall wear appropriate personal protective clothing and equipment to protect themselves from potential exposure to all PCBs.

**Inhalation.** Exposure via this route could occur if large amounts of PCB containing dusts are encountered during sampling activities.

**Skin Contact.** Skin contact with PCBs are significant routes of entry of those materials.

**Ingestion.** Exposure via this route could occur if individuals eat, drink or perform other hand-to-mouth contact during abatement activities..

*PCB Hazard Exposure Guidelines*

| Substances | CAL OSHA PEL |
|---|---|
| PCBs | 0.5 mg/m3 |

## 3.3 Physical and Other Hazards

Physical hazards associated with transportation to and from the site, and site activities include those listed in the following Activity Hazard Analysis Table.

| Activity | Hazard | Control(s) |
|---|---|---|
| Driving to, on, and from the site | Striking pedestrians, runaway vehicles, striking structures, overturning vehicles. | Wear seat belts at all times while vehicles are in motion. Use licensed drivers. Define vehicle routes of travel. Obey driving regulations. Do not drive over holes or down sides of improperly sloped depressions. |
| Using Hi pressure Water / Media | Contusions, cuts | Equipment operators and others working in immediate are to wear Kevlar protection on arms and legs, face protection. All employees to be trained in associated hazards and use of equipment. Barrier tape within existing containment will be used to keep all untrained, non-essential people from danger zone. |
| General site activities | Stinging insects (bees, wasps, spiders) | Use care when working near dense vegetation. Be sure individuals allergic to insect bites (if any) have obtained prescription for insect-bite kit. |
| General Site Activities | Bird droppings/manure | Wear protective clothing, and air-purifying respirators as appropriate if the bird manure dust is bothersome. |
| General Site Activities | Slipping on wet or oily surfaces | Use wet methods to collect samples. Spray areas damaged by sampling with adhesive or encapsulant to hold down fibers. |
| General Site Activities | Heat Stress / Heat Stroke | Use of impermeable clothing reduces the cooling ability of the body because of evaporation reduction. This may lead to heat stress.<br>Hydrate & rest often<br>**See below for more information** |
| Removal of bulk suspect asbestos, lead and/or PCB samples | Inhalation of asbestos/ lead or PCB's in dusts | All work will be performed in a negative pressure containment using wet methods to limit dust. All employees will wear appropriate respiratory protection. After removal, an encapsulant will be used to hold down fibers. |

| | | |
|---|---|---|
| Using sharp tools | Cuts and punctures | Use extreme care when cutting or chipping samples with sharp instruments. Retract blades into containers, or hold blades and sharp tools away from body when walking. |
| Working at heights on ladders | Falls from heights, ladders slipping, dropping items | All ladders will be placed at a proper angle equal to 1 length of run for every 4 lengths of rise. Ladders will be tied off before work at heights greater than 20' will be attempted. Only Class I ladders with 300 lb. weight limits will be used. Weight limits on ladders will not be exceeded. Ladder footings will be level and on non-slippery surfaces before climbing. Individuals working under ladders will wear hard hats to protect against dropped objects. |
| Working at heights on elevated walkways, stairs and platforms | Falls from heights, ladders slipping, dropping items | Refer to the DPR Fall Protection HASP |
| Working at heights on elevated aerial lifts, boom lifts, scissor lifts | Falls from heights, ladders slipping, dropping items | Refer to the DPR Fall Protection HASP |
| Open holes in elevated decking; pits and recesses in floors | Trips, falls | Refer to the DPR Fall Protection HASP |

### 3.4 Working with Hi Pressure Water / Media Blast Equipment.

Refer to the attached DPR HASP requirements related to fall equipment us and working on elevated platforms.

*All work will be conducted in accordance with 1) DPR's corporate IIPP and/or site safety rules, 2) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.*

All **Eco Bay** personnel shall have training pursuant to manufacturer's recommendation for use of hi-pressure water by NLB and Vapor Blast Equipment.

**Eco Bay** employees assigned to the direct use and support of this equipment will wear Kevlar protection of arms, chest and legs. All non-essential personnel will be segregated from danger zone by use of barrier tape and containments. Danger zones is the immediate area (5') surrounding the "nozzle of the gun".

In addition to additional PPE referenced above all Eco Bay Personnel have had specialized training related to use of this equipment.

Daily HASP focused specifically on dangers of this equipment will be provided and discussed in daily safety discussions.

## 3.5 Working at Elevated Heights, from A-frame Ladders, on Aerial Platforms, Near Fall Hazards or Floor Openings

Refer to the attached Eco Bay HASP requirements related to fall protection and working on elevated platforms.

*All work will be conducted in accordance with 1) Eco Bays corporate IIPP and/or site safety rules, 2) NASA Health & Safety Construction Safety Management (Chapter 27) requirements and NASA Health & Safety Fall Protection (Chapter 40) requirements.*

All **Eco Bay** personnel shall have training pursuant to fall protection standards (29 CFR 1926.501-503 and 8 CCR 1669).

**Eco Bay** employees assigned to the Moffett Field Hangar One Pilot Study have received appropriate fall protection training for work on elevated platforms, aerial lifts and similar site conditions pursuant to Federal and California Occupational Health Standards (29 CFR 1926.501-503 and 8 CCR 1669). Training has been completed within the last year and included coverage of the following topics:

- OSHA Fall Protection overview;
- Introduction to California-specific Fall Protection;
- Fall hazard recognition;
- Fall hazard elimination and controls methods;
- Applicable fall protection regulations;
- The responsibilities of designated persons;
- How to use written fall protection procedures;
- Inspection of equipment components and systems before use;

Eco Bay personnel shall use the following fall protection equipment in conjunction with the DPR Fall Protection Plan:

- Body Harness (Protecta Model Number 1191995 or equivalent), capacity 310 pounds
- Fall Arrest Dual Lanyard, 6' Length (Condor Model 19F384), capacity 310 pounds

or

- Fall Arrest Lanyard (Single), 6' length (Protecta Model 1341001 or equivalent), capacity 310 pounds

## 3.6 Heat Stress

*Signs of Heat Stress.* "Heat stress" is a term that is used to describe progressively more serious symptoms, as follows:

- An initial rise in skin temperature due to increased blood flow to the skin (skin redness);
- Increase in heart rate, to more than 30 beats/minute above the resting level;
- Collapse, or heat exhaustion, due to inadequate blood flow to the brain;
- Dehydration, due to excessive sweating;
- Hyperventilation, resulting in a reduction of the normal blood carbon dioxide concentrations;
- Tingling around the lips, dizziness, cramping of muscles of hands and feet, and blackout; and
- "Heat stroke," characterized by unconsciousness, hot dry skin, and absence of sweating.

*Control of Heat Stress.* On hot, sunny days (high radiant heat load), if using impermeable work clothing, maintain appropriate work-rest cycles (progressively longer rest breaks in a cool location or the shade as temperature and work tasks increase) and drink water or electrolyte-rich fluids (Gatorade or equivalent) to minimize heat stress effects. Impermeable clothing will only be worn when absolutely necessary for control of hazardous chemicals.

Also, when ambient temperatures exceed 70 degrees F, employees will conduct monitoring of their heart (pulse) rates, as follows:

- Each employee will check his or her own pulse rate at the beginning of each break period;
- Take the pulse at the wrist for 6 seconds, and multiply by 10; and
- If the pulse rate exceeds 110 beats per minute, then reduce the length of the next work period by one-third.

*Example:* After a one-hour work period at 80 degrees, a worker has a pulse rate of 120 beats per minute. The worker must therefore shorten the next work period by one-third, resulting in a work period of 40 minutes until the next break.

*Treatment of Heat Stress.* Individuals affected by mild forms of heat stress (heat exhaustion, dehydration, or cramping) should take a break in a cool or shaded location, drink liquids, and sit or lay down until feeling better. Shorter work periods should be used until temperature cools off.

Individuals affected by heat stroke are in critical condition. Summon emergency aid immediately, remove clothing, and bathe individual in cool water continually to bring down body temperature.

## 4.0 Personnel Protection Equipment

The following personal protective equipment will be required AT ALL TIMES:

- Hard Hat
- Steel-toed shoes
- Safety glasses

The following personal protective equipment will be optional or required as the need arises:

- Hearing protection equipment
- Coveralls – disposable (Tyvek), or fabric (any chemical protective needs)
- Kevlar Body protection when applicable
- Respiratory Protection per attached plan

All Eco Bay personnel will be required to wear the appropriate personal protective equipment. The selection of equipment will be based on that days' activities and included as an integral part of that days HASP.

Eco Bay Employees while performing tasks that may disturb the coating or any material deemed to contain be contaminated by Lead and/or PCB's shall wear at a minimum a half-face, air-purifying respirator. All respirators will be equipped with high- efficiency particulate air (HEPA) filter cartridges. A higher level of respiratory protection (e.g., full-face respirator) may be chosen at employee's discretion. When performing abatement activities respiratory protection based on personal exposure assessment will be minimum required respiratory protecive equipment..

During all activities employees shall wear full body coveralls, with protective booties and hoods.

Hard Hats and Hard Toed Boots shall be required at all times. Other safety gear such as safety vests and gloves shall be worn when deemed appropriate.

Employees expected to wear air-purifying respiratory protection must be fit tested for the brand and model respirator they will be wearing during sampling. Fit tests are required to be performed in accordance with the OSHA standard every 12 months.

### 4.1 General Safety Equipment

The following equipment must be available and easily accessible for use:

- First Aid Kit
- Fire extinguishers (Foam, dry chemical or carbon dioxide)

## 5.0 Training

All personnel who may be exposed to onsite contaminants must provide documentation of the following:

- Current certification for Asbestos Removal (Minimum 40 hr. worker)
- Current certification for Lead Abatement Technician (minimum)
- 40 Hr. Hazwopper Certification

## 6.0 Project-Specific Training

Project-specific training and information will be provided either before traveling to the site or at the site before entry into the exclusion zone. The information and training will be documented and will include the following:

- DPR's /NASA Site Specific Training
- The contents of this Health and Safety Plan
- A discussion of the health and safety hazards; protective measures and work practices for handling contaminated soil, water or equipment.

## 7.0 Waste Handling and Disposal

The waste handling procedures discussed in the work plan will be followed. Waste generated by implementation of this Health and Safety program may include lead / metals and PCB materials, spent protective clothing such as Tyvek suits, gloves and wash and rinse solutions.

All debris will be placed in properly labeled and lined metal drums, tested for waste profiling, properly manifested and hauled to an EPA approved off-site waste disposal site.

## 8.0 Personnel Hygiene and Decontamination

*Respirators (see attached respiratory protection plan)*
All respirators, shall be provided and maintained by the trained user. All Eco Bay employees shall clean their respirator per regulatory requirements and inspected daily (if used) by the individual user.

*Disposable Materials*
All disposable clothing, gloves, expendable protective wear, used respirator cartridges, and other disposable material generated during site activities shall be placed in suitable plastic bags and disposed of at an appropriate off-site waste disposal receptacle.

*Personal Hygiene*
All site workers will thoroughly wash hands and face before eating, drinking, or performing other actions with a hand-to-mouth component.

*Minimization of Contamination*
Do not kneel on contaminated ground, stir up unnecessary dust, or perform any practice that increases the probability of hand- to-mouth transfer of contaminated materials. Use plastic drop cloths and equipment covers where appropriate. Eating, drinking, chewing gum, smoking or using smokeless tobacco are forbidden while collecting samples. Coordination with other site workers will be accomplished, as necessary, to avoid cross-contamination.

## 9.0 Equipment Decontamination

All tools and non-disposable supplies shall be thoroughly washed and/or wet wiped prior to storage after use. All water and rags used in equipment decontamination will be placed in leak tight barrels and characterized for waste profiling to be hauled and disposed of off-site in an appropriate land fill.

## 10.0 Personal Injury

In case of a minor personal injury, general first aid procedures should be implemented. A first aid kit will be available at the site at all times. More serious injuries may require assistance from paramedics.

The Project Manager or another designated person will contact the appropriate emergency personnel by dialing emergency numbers listed at the front of this document. The Project Manager or another designated person will contact DPR immediately following contact of medical personnel.

## 11.0 Attachments

Attachment A: Eco Bay Corporate Health & Safety Manual, Section A- Administration, Subsections:

1. Accident Investigation
2. Code of Safe Practices
3. Emergency Response Plan
4. General Safety Rules
5. Record Keeping
6. Roles and Responsibilities
7. Safety Policy Statement
8. Safety Training
9. Site Control Procedures
10. Substance Abuse
11. Tool Box Talks
12. OSHA Inspections

Attachment B: Eco Bay Corporate Health & Safety Manual, Section B – Hazard Control, Subsections:

1. Accidental Spill Procedures
2. Aerial Work Platforms
3. Asbestos Management Policy
4. Decontamination Program
5. Electrical Hazards
6. Fall Protection
7. Fire Prevention
8. Hand & Power Tools
9. Hazard Communication
10. Heat Illness Prevention Plant
11. Housekeeping
12. IIPP
13. JHA for Abatement
14. Lead and Asbestos Abatement Policy
15. Personal Protective Equipment
16. Project Activity Hazard Analysis
17. Respiratory Protection Plan
18. Scaffolding
19. Hazard Control
20. Signs, Signals & Barricades
21. Stairway and Ladder

Attachment C: Eco Bay Respiratory Protection Table
Attachment D: Pictures and Drawings Showing Area for Pilot Study

Included by Reference:

- NASA Ames Health & Safety – Fall Protection (Chapter 40)
- EKI Environmental Issues Management Plan ("EIMP")
- ACC Work Plan for the Pilot Study of Hanger 1, March 1st Revision

*EXAMPLE*
**SITE SAFETY REVIEW**
**Asbestos, Lead/Metals & PCB**

**General Information**

Date: _____ Time: _____

**Project:** **HANAGR ONE PCB, LEAD/METALS & ASBESTOS ABATEMENT**

*Site Address:* Hangar 1
Moffett Federal Airfield
NASA Ames Research Center
Moffett Field, CA 94035

**Client Contact:** Mr. Kevin Antonelli, CBRE/ Planetary Venture Project Manager (408) 402-2061

**Objectives:** Define Objective of project such as Lead and PCB abatement

**Topics Discussed:**

*Physical Hazards:* Typical hazards associated with scope of work activities and how to prevent potential employee exposure.

*Special Site Considerations:*

Potential for traffic hazards and physical hazards
Sampling Safety & Hygiene
Fall Protection
Working on Elevated Platforms

ATTENDEES

| Name (please print) | Signature |
|---|---|
| --- | --- |
| --- | --- |
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| --- | --- |
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# Attachment C

# Calculation of Risk-Based Airborne Action Level for PCBs

7 September 2015

MEMORANDUM

To: Kevin Antonelli (Google)
Ed Baylosis (Google)
Sallie Lim, Esq. (Google)

Cc: Pam Andes, Esq. (Allen Matkins)

From: Michelle K. King, Ph.D., Claudia Cuadrado, P.E., and John Montgomery-Brown, Ph.D. (EKI)

Subject: Development of a Risk-Based Airborne Action Level for Polychlorinated Biphenyls
MFA Leasehold, Former NAS Moffett Field, California
(EKI B20019.19 T1)

On behalf of Planetary Ventures LLC, Erler & Kalinowski, Inc. (EKI) has prepared this memorandum describing the development of a risk-based airborne action level for polychlorinated biphenyls (PCBs) to be used during a pilot study of PCB and lead abatement options for the Hangar 1 structure located within the MFA Leasehold at the Former Naval Air Station Moffett Field (Airborne Action Level). More specifically, ACC Environmental Consultants (ACC) has prepared a Work Plan for the Pilot Scale Abatement Study of Hangar 1 (Pilot Study) to determine the feasibility of reducing PCB and lead concentrations in accessible structural steel elements and concrete masonry unit (CMU) walls within the Hangar 1 structure. The Pilot Study proposes to remove the PCB- and lead-contaminated coatings from the accessible Hangar 1 structural elements using three different media blasting technologies. During abatement activities, a fully encapsulated negative-pressure enclosure will be installed around the Pilot Study Area (Figure 2 of the Pilot Study) to minimize the potential for PCB- and lead-impacted fugitive dust to be released to the air and migrating outside of the Pilot Study Area. During active media activities (estimated to occur on a total of three days), potential exposure to non-volatile chemicals of concern (COCs) will be assessed through real-time dust monitoring and chemical-specific monitoring at two perimeter locations, one upwind and one downwind of the Pilot Study; samples collected for chemical-specific monitoring will be analyzed for PCBs by United States Environmental Protection Agency (USEPA) Method 8082 and lead by USEPA Method 6020.

Airborne Action Levels for Total Dust, respirable dust (PM10 or particulate matter with an effective diameter of 10 microns or less), and lead were developed in the Pilot Study based on California Ambient Air Quality Standards (CAAQS) or Bay Area Air Quality Management District (BAAQMD) regulatory limits. As there are no CAAQS or BAAQMD regulatory limits for airborne PCBs, this memorandum presents the derivation of a risk-based Airborne Action Level for the protection of nearby workers from potential exposure to PCBs during the Pilot Study.

## DEVELOPMENT OF AN AIRBORNE ACTION LEVEL FOR PCBS

As noted above, the objective of the perimeter air monitoring during abatement activities is to demonstrate that the nearby workers are protected from exposure to PCBs and lead[1] associated with fugitive dust emissions from the abatement enclosure. This section describes the methodology used to develop a risk-based Airborne Action Level for PCBs in the absence of regulatory limits for PCBs.

It is anticipated that abatement activities in which potential PCB emissions could occur will be performed for up to ten days of active work. This assumption does not include setup or take down of the fully encapsulated negative-pressure enclosure or final verification sampling. For risk calculation purposes, it is conservatively assumed that abatement activities will occur over a period of 8 hours a day, 5 days a week, for 2 weeks.

### *Potentially Exposed Populations*

In terms of assessing risk, the USEPA defines "exposure" as the contact of a human with a chemical (USEPA, 1989). In order to assess exposure, each group of people that could potentially be exposed to chemicals (the "potentially exposed populations") must be defined. For the purpose of the Pilot Study, only On-Site Commercial/Industrial Workers,[2] will be considered.

The on-Site Commercial/Industrial Worker population includes hypothetical office and industrial workers in the area surrounding the Pilot Study Area (Figure 2 of the Pilot Study). For the purposes of the risk-based calculations, on-Site Commercial/Industrial Workers are assumed to work primarily outdoors, immediately adjacent to the Pilot Study Area (i.e., the risk-based Action Levels do not account for attenuation that would occur between the source area and the receptor). It is expected that exposure levels to airborne COCs from the Pilot Study Area would be lower for indoor workers.

### *Exposure Parameters*

The assumed exposure parameters for the hypothetical potentially exposed population are discussed below. These exposure parameters were selected based on available Department of Toxic Substances Control (DTSC; 2014) and USEPA (2015) guidance or based on professional judgment regarding project-specific parameters.

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[1] BAAQMD regulates airborne lead concentrations at ground level at a limit of 0.001 mg/m$^3$, measured as a 24-hour time weighted average (TWA; BAAQMD Rule 11-1-302); BAAQMD Rule 11-1-302 does not require any foreknowledge of background lead concentrations at the site. To be conservative, an 8-hour TWA concentration of 0.001 mg/m$^3$ will be used as the Action Level for airborne lead during the Pilot Study.

[2] It is assumed that workers directly involved with Hangar 1 abatement activities will be adequately protected against potential exposure to COCs in air using engineering controls and personal protective equipment (PPE) in accordance with project-specific health and safety plans.

*Exposure Frequency*

The assumed exposure frequency (EF) for On-Site Commercial/Industrial Workers is 10 days/year (i.e., 10 days during which PCB emissions could occur), which is based on ACC's conservative estimate of the anticipated duration of the abatement activities in the Pilot Study Area.

*Exposure Time*

For the inhalation exposure pathways a key parameter is the assumed exposure time (ET), expressed as hours per day (hrs/day). The ET indicates the amount of time a member of the hypothetical population may spend each day in an area where PCB emissions could occur in ambient air.

For On-Site Commercial/Industrial Workers, the ET is assumed to be 8 hrs/day during the length of the abatement activities in which PCBs emissions could occur (i.e., 10 days).

*Averaging Time and Exposure Duration*

For On-Site Commercial/Industrial Workers, the exposure duration is one year. For estimating non-carcinogenic adverse health effects, the averaging time (AT) is assumed to be equal to the exposure duration (ED) in units of days. For estimating cancer risk, averaging time for all hypothetical populations is assumed to be a lifetime of 70 years (USEPA, 1989).

***PCBs Toxicity Assessment***

The two broad measures of adverse human health effects recognized in a risk assessment are carcinogenic risk and non-carcinogenic hazard. Carcinogenic risk, as used herein, is an upper-bound estimate of the incremental risk of developing cancer from exposure to a chemical over a 70-year average lifetime. The carcinogenicity of a chemical is expressed as an inhalation unit risk (IUR) for the inhalation pathway, which represents the estimated incremental lifetime risk of developing cancer per unit of chemical exposure.

Non-carcinogenic adverse health effects are generally assumed to have some threshold dose of the chemical below which adverse health effects are not expected. The non-carcinogenic hazard index is a ratio of the calculated exposure level of a chemical to the maximum "safe" exposure level considering adverse health effects other than cancer.

The Office of Environmental Health and Hazard Assessment (OEHHA) and USEPA are the primary sources of published toxicity data for various chemicals used in risk assessments for sites within California. Toxicity criteria developed by both OEHHA and USEPA are generally derived for only two exposure routes, ingestion (oral) and inhalation, by which the chemical enters the body.

Non-carcinogenic inhalation toxicity data are not available for PCBs. The only available non-carcinogenic oral toxicity data for PCBs is an oral reference dose of 0.02 micrograms per kilogram per day (ug/kg-day) that has been published by USEPA in their Integrated Risk Information System (IRIS) for PCB 1254. This value was, however, was not included in the risk calculations because (1) the main PCB present in structural steel elements and CMU walls within the Hangar 1 structure is PCB 1260 (ACC, 2015) and not PCB 1254 and (2) USEPA no longer recommends using route-to-route extrapolation between the oral and inhalation exposure pathways in risk assessments (U.S. EPA, 2009). Therefore, the Action Level for PCBs during the Pilot Abatement Study will be based on carcinogenic effects. An IUR of 0.00057 cubic meters per microgram (i.e., $(ug/m^3)^{-1}$), which was obtained from OEHHA, was used to estimate the risk-based Action Level for PCBs.

***Action Level for PCBs Corresponding to a $10^{-6}$ Incremental Lifetime Cancer Risk***

The incremental lifetime cancer risk due to inhalation of a chemical can be estimated using the following equation (USEPA, 2015). A target risk level of $10^{-6}$, which is at the low end of the USEPA risk range of $10^{-6}$ to $10^{-4}$ was used to calculate the Airborne Action Level.

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**Equation [1]** Risk-Based Action Levels for Carcinogenic COCs (USEPA, 2015)

$$AL = \frac{CR \times AT_c}{EF \times ED \times ET \times \frac{1\,day}{24\,hours} \times IUR}$$

where:

| | |
|---|---|
| AL | Action Level (micrograms per cubic meter; $ug/m^3$) |
| $AT_c$ | Averaging Time (70 years * 365 days/year; DTSC, 2014 and USEPA, 2015) |
| CR | Target lifetime incremental cancer risk of $10^{-6}$ (unitless) |
| ED | Assumed exposure duration (1 year) |
| EF | Assumed exposure frequency (10 days/year) |
| ET | Exposure time (8 hours/day; DTSC, 2014 and USEPA, 2015) |
| IUR | Inhalation unit risk (0.00057 $(ug/m^3)^{-1}$; OEHHA, 2015) |

The risk-based Airborne Action Level for PCBs was calculated to be approximately 13 $ug/m^3$ using Equation 1 and the exposure parameters discussed above. This Airborne Action Level is health protective because this concentration would have to be sustained at the location of the exposed receptors for the entire duration of the project to yield an incremental cancer risk of $10^{-6}$, which is highly unlikely.

## REFERENCES

ACC, 2015. *PCB, Lead, and Asbestos Sampling Report, Hangar One, Moffett Federal Air Field, Mountain View, California,* ACC Environmental Consultants, February 24, 2015.

DTSC, 2014. *Human Health Risk Assessment (HHRA), HERO HHRA Note Number 1: Recommended DTSC Default Exposure Factors for Use in Risk Assessment at California Hazardous Waste Sites and Permitted Facilities,* California Department of Toxic Substances Control, Office of Human and Ecological Risk, September 30, 2014.

OEHHA, 2015. *Toxicity Criteria Database,* California Environmental Protection Agency, Office of Environmental Health Hazard Assessment, [http://www.oehha.ca.gov/tcdb/index.asp].

USEPA, 1989. *Risk Assessment Guidance for Superfund, Volume 1 – Human Health Evaluation Manual (Part A),* U.S. Environmental Protection Agency, OERR, EPA/540/1-89/002, December 1989.

USEPA, 2009. *Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation Manual (Part F, Supplemental Guidance for Inhalation Risk Assessment),* Final, EPA-540-R-070-002, January 2009.

USEPA, 2015. *Regional Screening Table - User's Guide for Mid-Atlantic Risk Assessment,* U.S. Environmental Protection Agency, June 2015.

Attachment D

Carbomastic® 15 Epoxy Coating: Product Information and Application Instructions

product data carboline®

# Carbomastic® 15

## Selection & Specification Data

**Generic Type** Epoxy mastic

**Description** Aluminum-pigmented, low-stress, high-solids mastic with a proven field history. Carbomastic 15 was the pioneer mastic coating in a number of industrial markets and today still provides unmatched levels of barrier protection and corrosion resistance over existing finishes and rusted or SSPC-SP2 or SP3-cleaned steel.

**Features**
- Excellent performance over minimal surface preparation of steel substrates
- Suitable as a topcoat for most tightly adhered existing coatings
- Excellent choice for field touch-up of zinc-rich primers and galvanized steel
- Unique formulation with aluminum flakes provides exceptional barrier protection
- May be applied at 35°F (2°C) when CM 15 FC's part B is utilized
- Suitable for use under insulation on hot surfaces operating up to 300°F(150°C)
- VOC compliant to current AIM regulations

**Color** Aluminum (C901); Red (M500)

Color variations within a batch and from batch to batch may occur due to the metallic pigments and variations in application techniques and conditions. Neither product is color matched, nor will they match each other. (15 FC may have a greenish appearance.) *Red (M500) is available for use as a contrasting primer in multiple coat applications, but should always be topcoated.

**Primers** Self-priming. May be applied over most tightly adhering coatings as well as inorganic zinc primers.

**Dry Film Thickness** 3.0 - 5.0 mils (76 - 127 microns) over existing coatings
7.0 - 10.0 mils (178 - 254 microns) in one or two coats in severe exposures

**Do not exceed 10.0 mils (250 microns) in a single coat.**

**Solids Content** By Volume 90% +/- 2%

**HAPs Values** As supplied: 0.70 lbs/solid gal

**Theoretical Coverage Rate**
1444 $ft^2$ at 1 mil (35 $m^2$/l at 25 microns)
481 $ft^2$ at 3 mils (12 $m^2$/l at 75 microns)
144 $ft^2$ at 10 mils (4 $m^2$/l at 250 microns)

Allow for loss in mixing and application.

**Severe Exposures** Temperature resistance under insulation: Up to 300°F(150°C)

Discoloration is observed above 180°F(82°C) but does not affect performance.

**VOC Values**
Thinner 10 32 oz/gal: 2.0 lbs/gal (242 g/l)
Thinner 236 E 32 oz/gal: 0.7 lbs/gal (88 g/l)
Thinner 72 32 oz/gal: 2.07 lbs/gal (248 g/l)
Thinner 76 32 oz/gal: 1.9 lbs/gal (231 g/l)
As Supplied 0.7 lbs/gal (88 g/l)

These are nominal values.

**Topcoats** May be coated with Acrylics, Epoxies, Alkyds, or Polyurethanes depending on exposure and need.

## Substrates & Surface Preparation

**General** Surfaces must be clean and dry. Employ adequate methods to remove dirt, dust, oil and all other contaminants that could interfere with adhesion of the coating.

**Steel** Immersion: SSPC-SP10 with a 2.0-3.0 mil (50-75 micron) surface profile.
Non-Immersion: SSPC-SP6 with a 2.0-3.0 mil (50-75 micron) surface profile for maximum protection. SSPC-SP2, SP3, SP7, SP12,or SP14 are also acceptable methods.

**Galvanized Steel** For optimum performance sweep blast cleaning is recommended. Consult your Carboline Sales Representative for specific recommendations.

**Previously Painted Surfaces** Lightly sand or abrade to roughen and degloss the surface. Existing paint must attain a minimum 3A rating in accordance with ASTM D3359 “X-Scribe” adhesion test.

## Performance Data

| Test Method | System | Results |
|---|---|---|
| ASTM 4060 Taber Abrasion | 1 ct. CM15 | 130 mg loss; 1000 cycles using CS 17 wheel and 1000 gm load, |
| ASTM B117 Salt Spray | Rusted Steel 1 ct. CM 15 | No blistering, rusting, or softening No rust creep from scribe |
| ASTM D1735 Water Fog | Rusted Steel 1ct CM 15 | No blistering or softening, No creep from scribe |
| ASTM D522 Flexibility | Blasted steel 1 ct. CM15 | A) Conical - crack 0.38", actual elongation 48.57% B) Cylindrical-no cracking observed |
| ASTM G 14 Impact Resistance | A) Blasted Steel 1 ct. CM 15, B) Rusted Steel 1 ct. CM 15 | Area Damaged A) 1/4 inch (0.25") B) 1/4 - 9/16 inch (0.44") |

Test reports and additional data available upon written request.

## Mixing & Thinning

**Mixing** Power mix separately, then combine and power mix. DO NOT MIX PARTIAL KITS.

**Thinning** May be thinned up to 32 oz/gal (25%) with Thinner 10 for normal conditions. Thinner 72 may be used for hot or windy conditions. Use Thinner 76 for nonphotochemically reactive thinner or Thinner 236E for exempt thinner. Use of thinners other than those supplied by Carboline may adversely affect product performance and void product warranty, whether expressed or implied.

**Ratio** 1:1 Ratio (A to B)

**Pot Life** 2 hours @75°F(24°C) unthinned.
1 hour @90°F(32°C) unthinned

Pot life ends when coating becomes too viscous to use



To the best of our knowledge the technical data contained herein is true and accurate on the date of publication and is subject to change without prior notice. User must contact Carboline Company to verify correctness before specifying or ordering. No guarantee of accuracy is given or implied. We guarantee our products to conform to Carboline quality control. We assume no responsibility for coverage, performance or injuries resulting from use. Liability, if any, is limited to replacement of products. NO OTHER WARRANTY OR GUARANTEE OF ANY KIND IS MADE BY CARBOLINE, EXPRESS OR IMPLIED, STATUTORY, BY OPERATION OF LAW, OR OTHERWISE, INCLUDING MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. Carboline® and Carboguard® are registered trademarks of Carboline Company.

# Carbomastic® 15

## Application Equipment Guidelines

Listed below are general equipment guidelines for the application of this product. Job site conditions may require modifications to these guidelines to achieve the desired results.

**Spray Application (General)** The following spray equipment has been found suitable and is available from manufacturers such as Binks, DeVilbiss and Graco.

**Conventional Spray** Pressure pot equipped with dual regulators, 3/8" I.D. minimum material hose, .086" I.D. fluid tip and appropriate air cap.

**Airless Spray** Pump Ratio: 30:1 (min.)*
GPM Output: 3.0 (min.)
Material Hose: 3/8" I.D. (min.)
Tip Size: .019-.025"
Output PSI: 1900-2100
Filter Size: 60 mesh
*Teflon packings are recommended and available from the pump manufacturer.

**Plural Component** May be applied by plural component spray equipment. Contact Carboline Technical Service for specific recommendations.

**Brush & Roller (General)** Multiple coats may be required to obtain desired appearance, recommended dry film thickness and adequate hiding. Avoid excessive re-brushing or rerolling. Use clean natural bristle brush or medium nap phenolic core roller. Work coating into all irregularities.

## Application Conditions

| Condition | Material | Surface | Ambient | Humidity |
|---|---|---|---|---|
| Minimum | 50 °F (10 °C) | 50 °F (10 °C) | 50 °F (10 °C) | 0% |
| Maximum | 90 °F (32 °C) | 130 °F (54 °C) | 100 °F (38 °C) | 95% |

This product simply requires the substrate temperature to be above the dew point. Condensation due to substrate temperatures below the dew point can cause flash rusting on prepared steel and interfere with proper adhesion to the substrate. Special application techniques may be required above or below normal application conditions.

## Curing Schedule

| Surface Temp. & 50% Relative Humidity | Final Cure Immersion | Dry to Recoat or Topcoat |
|---|---|---|
| 50 °F (10 °C) | 15 Days | 5 Days |
| 60 °F (16 °C) | 10 Days | 3 Days |
| 75 °F (24 °C) | 5 Days | 24 Hours |
| 90 °F (32 °C) | 3 Days | 18 Hours |

**For CM 15 Dry to Touch is 5 hours at 75°F (24°C). Maximum re-coat/topcoat times are 30 days for epoxies and 90 days for polyurethanes at 75°F (24°C).**
These times are based on a 5.0-7.0 mil (125-175 micron) dry film thickness. Higher film thickness, insufficient ventilation or cooler temperatures will require longer cure times and could result in solvent entrapment and premature failure. Excessive humidity or condensation on the surface during curing can interfere with the cure, can cause discoloration and may result in a surface haze. Any haze or blush must be removed by water washing before recoating. If the maximum recoat time is exceeded, the surface must be abraded by sweep blasting prior to the application of additional coats.
**Note:** This product contains conductive pigments and cannot be holiday tested.

## Cleanup & Safety

**Cleanup** Use Thinner #2 or Acetone. In case of spillage, absorb and dispose of in accordance with local applicable regulations.

**Safety** Read and follow all caution statements on this product data sheet and on the MSDS for this product. Employ normal workmanlike safety precautions. Hypersensitive persons should wear protective clothing, gloves and use protective cream on face, hands and all exposed areas.

**Ventilation** When used as a tank lining or in enclosed areas, thorough air circulation must be used during and after application until the coating is cured. The ventilation system should be capable of preventing the solvent vapor concentration from reaching the lower explosion limit for the solvents used. In addition to ensuring proper ventilation, appropriate respirators must be used by all application personnel.

## Packaging, Handling & Storage

**Shelf Life** Part A & B: Min. 36 months at 75°F (24°C)
*Shelf Life : (actual stated shelf life) when kept at recommended storage conditions and in original unopened containers.

**Shipping Weight (Approximate)** 2 Gallon Kit - 25 lbs (11 kg)
10 Gallon Kit - 124 lbs (56 kg)

**Storage Temperature & Humidity** 45° - 110°F (7-43°C)
0-90% Relative Humidity

**Flash Point (Setaflash)** Part A: >200°F (93°C)
Part B: 76°F (24°C)

**Storage** Store Indoors.

carboline®
Coatings - Linings - Fireproofing
2150 Schuetz Rd., St. Louis, MO 63146
PH: 314-644-1000 Toll-Free: 800-848-4645
www.carboline.com

An RPM Company

July 2013
0185

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# Material Safety Data Sheet

**CHEMTREC Transportation Emergency Phone: 800-424-9300**
**Pittsburgh Poison Control Center Health Emergency No.: 412-681-6669**
NOTE: The CHEMTREC Transportation Emergency Phone is to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure, or accident involving chemicals

## 1. Identification

| | | | |
|---|---|---|---|
| **Product Name:** | CARBOMASTIC 15 EPOXYMASTIC PART B | **Revision Date:** | 8/9/2013 |
| **Identification Number:** | 1036B1NL | **Supercedes Date:** | 3/3/2010 |
| **Product Use/Class:** | Epoxy Mastic - FOR INDUSTRIAL USE ONLY | | |
| **Manufacturer:** | Carboline Company<br>2150 Schuetz Road<br>St. Louis, MO 63146<br>800-848-4645 | **Preparer:** | Regulatory Department |

## 2. Hazard Identification

**EMERGENCY OVERVIEW:** Avoid contact with skin, eyes and clothing. Wash thoroughly after handling. Harmful if swallowed. Risk of serious damage to the lungs (by aspiration). Keep container closed. Contains SILICA which can cause cancer. Risk of Cancer depends on duration and level of exposure. Irritating to eyes and skin. WARNING! - FLAMMABLE LIQUID AND VAPOUR. Keep away from heat and sources of ignition. Harmful if inhaled. Use with adequate ventilation. Vapours may cause drowsiness and dizziness.

**EFFECTS OF OVEREXPOSURE - EYE CONTACT:** Causes eye burns.

**EFFECTS OF OVEREXPOSURE - SKIN CONTACT:** Causes skin burns. May cause allergic skin reaction. May be harmful if absorbed through skin.

**EFFECTS OF OVEREXPOSURE - INHALATION:** May cause allergic respiratory reaction. Harmful if inhaled. Symptoms of overexposure may be headache, dizziness, tiredness, nausea and vomiting. Vapours may be irritating to eyes, nose, throat, and lungs.

**EFFECTS OF OVEREXPOSURE - INGESTION:** Harmful if swallowed.

**EFFECTS OF OVEREXPOSURE - CHRONIC HAZARDS:** Crystalline silica is known to cause silicosis. Crystalline silica (Quartz) is classified as a known human carcinogen (Group 1) by IARC. Exposure is by route of inhalation. If material is in a liquid matrix it is unlikely to be inhaled. When sanding or grinding the finished product, there may be potential for crystalline silica to become airborne. Repeated and prolonged exposure to solvents may cause brain and nervous system damage.

**MEDICAL CONDITIONS PRONE TO AGGRAVATION:** No information available.

**PRIMARY ROUTE(S) OF ENTRY:** Inhalation, Skin Absorption, Skin Contact

## 3. Composition/Information On Ingredients

**Hazardous Ingredients**

| Chemical Name | CAS-No. | Weight % Less Than | ACGIH TLV-TWA | ACGIH TLV-STEL | OSHA PEL-TWA | OSHA PEL-CEILING |
|---|---|---|---|---|---|---|
| MICROCRYSTALLINE SILICA | 14808-60-7 | 45.0 | 0.025 MG/M3 (respirable) | N/E | 0.1 MG/M3 (respirable) | N/E |
| MICA | 12001-26-2 | 15.0 | 3 MGM3 | N/E | 3 MGM3 | N/E |
| BENZYL ALCOHOL | 100-51-6 | 10.0 | N/E | N/E | N/E | N/E |
| ALIPHATIC POLYAMINE | TRADE SECRET | 10.0 | NE | NE | NE | NE |

| | | | | | | |
|---|---|---|---|---|---|---|
| DIAMINOCYCLOHEXANE | 694-83-7 | 10.0 | N/E | N/E | N/E | N/E |
| TOLUENE | 108-88-3 | 5.0 | 20 PPM | N/E | 375 MGM3 | N/E |
| META-XYLENE | 108-38-3 | 5.0 | 100 PPM | 150 PPM | 435 MG/M3 | N/E |
| 1-METHOXY-2-PROPANOL ACETATE | 108-65-6 | 5.0 | N/E | N/E | N/E | N/E |
| PARA-XYLENE | 106-42-3 | 5.0 | 100 PPM | 150 PPM | 435 MGM3 | N/E |
| ETHYL BENZENE | 100-41-4 | 5.0 | 20 PPM | N/E | 435 MGM3 | N/E |
| POLYSTYRENE | 9003-53-6 | 5.0 | N/E | N/E | N/E | N/E |

## 4. First-aid Measures

**AFTER EYE CONTACT:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

**AFTER SKIN CONTACT:** In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. If skin irritation persists, call a physician.

**AFTER INHALATION:** Give oxygen or artificial respiration if needed. Remove person to fresh air. If signs/symptoms continue, get medical attention.

**AFTER INGESTION:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. If swallowed, call a poison control centre or doctor immediately.

## 5. Fire-fighting Measures

**Flash Point, °F:** 76 F (24 C) (Setaflash)

**Lower Explosive Limit, %:** 0.9

**Upper Explosive Limit, %:** 12.7

**Extinguishing Media:** Carbon Dioxide, Dry Chemical, Foam, Water Fog

**UNUSUAL FIRE AND EXPLOSION HAZARDS:** Flammable liquid. Vapours are heavier than air and may spread along floors. Vapours may form explosive mixtures with air. Vapors may travel to areas away from work site before igniting/flashing back to vapor source.*Provide adequate ventilation. Prevent the creation of flammable or explosive concentrations of vapour in air and avoid vapour concentration higher than the occupational exposure limits. Keep away from heat/sparks/open flames/hot surfaces. - No smoking. Electrical installations / working materials must comply with the technological safety standards. Wear shoes with conductive soles.

**SPECIAL FIREFIGHTING PROCEDURES:** Cool containers / tanks with water spray. Flammable. Evacuate personnel to safe areas. Use NIOSH approved respiratory protection. Use water spray to cool unopened containers. In the event of fire, wear self-contained breathing apparatus.

## 6. Accidental Release Measures

**PERSONAL SAFETY MEASURES/ENVIRONMENTAL MEASURES/METHOD OF CLEANING/CONTAINMENT:** Remove all sources of ignition. To avoid ignition of vapours by static electricity discharge, all metal parts of the equipment must be grounded. Evacuate personnel to safe areas. Wear personal protective equipment. For personal protection see section 8. Contain spillage, soak up with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and transfer to a container for disposal according to local / national regulations (see section 13). Do not allow material to contaminate ground water system. Prevent product from entering drains.

## 7. Handling and Storage

**INSTRUCTIONS FOR SAFE HANDLING :** Do not breathe vapours or spray mist. Do not get in eyes, on skin, or on clothing. Keep containers dry and tightly closed to avoid moisture absorption and contamination. Use only with adequate ventilation/personal protection. Wash thoroughly after handling. Ensure all equipment is electrically grounded before beginning transfer operations. Do not use sparking tools. Prepare the working solution as given on the label(s) and/or the user instructions.

**STORAGE CONDITIONS:** Keep container closed when not in use. Store in a dry, well ventilated place away from sources of heat, ignition and direct sunlight.

## 8. Exposure Controls/Personal Protection

**ENGINEERING CONTROLS:** Avoid contact with skin, eyes and clothing. Ensure adequate ventilation, especially in confined areas.

**RESPIRATORY PROTECTION:** In order to avoid inhalation of spray-mist and sanding dust, all spraying and sanding must be done wearing adequate respirator. Use only with ventilation to keep levels below exposure guidelines reported in this document. User should test and monitor exposure levels to ensure all personnel are below guidelines. If not sure, or not able to monitor, use State or federally approved supplied air respirator. For silica containing coatings in a liquid state, and/or if no exposure limits are established above, air-supplied respirators are generally not required.

**SKIN PROTECTION:** Lightweight protective clothing. Impervious gloves. Gloves should be discarded and replaced if there is any indication of degradation or chemical breakthrough. Request information on glove permeation properties from the glove supplier.

**EYE PROTECTION:** Safety glasses with side-shields

**OTHER PROTECTIVE EQUIPMENT:** Ensure that eyewash stations and safety showers are close to the workstation location.

**PROTECTION AND HYGIENE MEASURES :** Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday. When using, do not eat, drink or smoke.

## 9. Physical and Chemical Properties

| | | | |
|---|---|---|---|
| **Boiling Range:** | 176 F (80 C) - 554 F (290 C) | **Vapor Density:** | Heavier than Air |
| **Odor:** | Epoxy | **Odor Threshold:** | N/D |
| **Appearance:** | Viscous Liquid | **Evaporation Rate:** | Slower Than Ether |
| **Solubility in Water:** | N/D | **Specific Gravity:** | 1.56 |
| **Freeze Point:** | N/D | **pH:** | N/D |
| **Physical State:** | Liquid | **Vapor Pressure:** | No Information |

(See section 16 for abbreviation legend)

## 10. Stability and Reactivity

**CONDITIONS TO AVOID:** Heat, flames and sparks.

**MATERIALS TO AVOID:** Strong oxidizing agents

**HAZARDOUS DECOMPOSITION PRODUCTS:** Carbon dioxide (CO2), carbon monoxide (CO), oxides of nitrogen (NOx), dense black smoke.

**HAZARDOUS POLYMERIZATION:** Hazardous polymerisation does not occur.

**STABILITY:** Stable under normal conditions.

## 11. Toxicological Information

| Chemical Name | CAS-No. | LD50 | LC50 |
|---|---|---|---|
| MICROCRYSTALLINE SILICA | 14808-60-7 | Not Available | Not Available |
| MICA | 12001-26-2 | Not Available | Not Available |
| BENZYL ALCOHOL | 100-51-6 | 1230 mg/kg rat, oral | 1000 ppm / 8 hrs rat, inhalation |
| ALIPHATIC POLYAMINE | TRADE SECRET | >2300 MG/KG ORAL RAT EST | >10 MG/L / 1 HR EST |
| DIAMINOCYCLOHEXANE | 694-83-7 | 1752 mg/kg, rat, oral | Not Available |
| TOLUENE | 108-88-3 | 5000 mg/kg rat oral, 14000 mg/kg rabbit dermal | 8000 ppm/4 hrs, rat, inhalation |

| | | | |
|---|---|---|---|
| META-XYLENE | 108-38-3 | Not Available | Not Available |
| 1-METHOXY-2-PROPANOL ACETATE | 108-65-6 | 8532 mg/kg, oral (rat) | 101 ppm/4 hr, rat, inh |
| PARA-XYLENE | 106-42-3 | Not Available | Not Available |
| ETHYL BENZENE | 100-41-4 | 3500 mg/kg rat, oral | 17.2 mg/L Inh, Rat, 4Hr |
| POLYSTYRENE | 9003-53-6 | Not Available | Not Available |

## 12. Ecological Information

**ECOLOGICAL INFORMATION:** No information available.

## 13. Disposal Information

**DISPOSAL INFORMATION:** Dispose of in accordance with local regulations.

## 14. Transport Information

| | | | |
|---|---|---|---|
| **DOT Proper Shipping Name:** | Paint | **Packing Group:** | III |
| **DOT Technical Name:** | N/A | **Hazard Subclass:** | N/A |
| **DOT Hazard Class:** | 3 | **Resp. Guide Page:** | 128 |
| **DOT UN/NA Number:** | UN 1263 | | |
| **Additional Notes:** | No Information | | |

## 15. Regulatory Information

### U.S. Federal Regulations:

#### CERCLA - SARA Hazard Category

This product has been reviewed according to the EPA 'Hazard Categories' promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

Fire Hazard, Acute Health Hazard, Chronic Health Hazard

#### Sara Section 313:

This product contains the following substances subject to the reporting requirements of Section 313 of Title III of the Superfund Amendment and Reauthorization Act of 1986 and 40 CFR part 372:

| Chemical Name | CAS-No. |
|---|---|
| TOLUENE | 108-88-3 |
| META-XYLENE | 108-38-3 |
| PARA-XYLENE | 106-42-3 |
| ETHYL BENZENE | 100-41-4 |

#### Toxic Substances Control Act:

This product contains the following chemical substances subject to the reporting requirements of TSCA 12(B) if exported from the United States:

No TSCA 12(b) components exist in this product.

### U.S. State Regulations:

#### New Jersey Right-to-Know:

The following materials are non-hazardous, but are among the top five components in this product.

No NJ Right-To-Know components exist in this product.

### Pennsylvania Right-To-Know:

The following non-hazardous ingredients are present in the product at greater than 3%.

| Chemical Name | CAS-No. |
|---|---|
| HYDROCARBON RESIN | TRADE SECRET |

### CALIFORNIA PROPOSITION 65:

Warning: The following ingredients present in the product are known to the state of California to cause Cancer:

| Chemical Name | CAS-No. |
|---|---|
| MICROCRYSTALLINE SILICA | 14808-60-7 |
| ETHYL BENZENE | 100-41-4 |
| BENZENE | 71-43-2 |

Warning: The following ingredients present in the product are known to the state of California to cause birth defects, or other reproductive hazards.

| Chemical Name | CAS-No. |
|---|---|
| TOLUENE | 108-88-3 |
| BENZENE | 71-43-2 |

## International Regulations:

### CANADIAN WHMIS:

This MSDS has been prepared in compliance with Controlled Product Regulations except for the use of the 16 headings.

Canadian WHMIS Class: B2 D2A D2B

## 16. Other Information

### HMIS Ratings:

**Health:** 2 **Flammability:** 3 **Reactivity:** 0 **Personal Protection:** X

**VOLATILE ORGANIC COMPOUNDS, GR/LTR MIXED (UNTHINNED):** 88

Legend: N.A. - Not Applicable, N.E. - Not Established, N.D. - Not Determined

The information contained herein is, to the best of our knowledge and belief accurate. However, since the conditions of handling and use are beyond our control, we make no guarantee of results, and assume no liability for damages incurred by use of this material. It is the responsibility of the user to comply with all applicable federal, state, and local laws and regulations.

product data

carboline®

# Carboguard® 1340

## Selection & Specification Data

**Generic Type** Polyamido-Amine Epoxy

**Description** Penetrating primer/sealer for use on concrete substrates and Carboline Pyrocrete Fireproofing products. It performs extremely well in sealing cementitious surfaces and is designed to receive a variety of different generic types of finish coats. Some recommended uses of 1340 include the use as a form release agent on plywood or steel forms.

**Features**
- Exceptional wetting characteristics
- Low stress, highly flexible film
- Very high solids
- Low odor
- User-friendly brush & roller application
- VOC compliant to current AIM regulations

**Color** Clear Amber (0910)

**Finish** Gloss

**Primers** Self-priming. May be applied over most generic types of coatings.

**Topcoats** Acrylics, Epoxies, Polyurethanes

**Dry Film Thickness** 1.0 - 2.0 mils (25 - 50 microns) DFT for most applications but can be applied up to 4.0 mils (100 microns) for sealing rough surfaces or shot-blasted concrete. When used as a form release agent, it may be applied up to 10.0 mils (250 microns) wet.

**Solids Content** By Volume: 98% ± 2%

**Theoretical Coverage Rate** 1572 mil $ft^2$ (38.5 $m^2$/l at 25 microns)
Allow for loss in mixing and application. Porous and irregular substrates like concrete/fireproofing coverage rates and should be taken into account.

**VOC Values** Per EPA Method 24: 0.2 lbs/gal (24 g/l)
Thinned:
25 oz/gal w/ #76: 1.3 lbs./gal (156 g/l)
These are nominal values.

**Dry Temp. Resistance** Continuous: 175°F (79°C)
Non-Continuous: 200°F (93°C)

**Limitations**
- Epoxies lose gloss, discolor and eventually chalk in sunlight exposure.
- Do not use for immersion service.

## Substrates & Surface Preparation

**Concrete** Surfaces must be clean and dry. Employ adequate methods to remove dirt, dust, oil and all other contaminants that could interfere with adhesion of the coating.

**General** Concrete must be cured 28 days at 75°F (24°C) and 50% relative humidity or equivalent. Prepare surfaces in accordance with ASTM D4258 Surface Cleaning of Concrete and ASTM D4259 Abrading Concrete. Voids in concrete may require surfacing. Application prior to surfacing assures tight adhesion between concrete and surfacers or membranes. Compatibility with other coatings, surfacers and polyurethane membranes eliminates need for form release oils or curing oils.

**Pyrocrete Fireproofing Products** Contact Carboline Technical Service or your Carboline sales representative for specific applications and requirements.

**Previously Painted Surfaces** Lightly sand or abrade to roughen and degloss the surface. Existing paint must attain a minimum 3A rating in accordance with ASTM D3359 "X-Scribe" adhesion test.



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## Application Equipment

Listed below are general equipment guidelines for the application of this product. Job site conditions may require modifications to these guidelines to achieve the desired results.
General Guidelines:

**Spray Application (General)** Contact Carboline Technical Service for spray equipment and technique.

**Brush & Roller (General)** Avoid excessive re-brushing or re-rolling. Apply only enough material to wet the surface uniformly. Any puddles formed must be brushed out.

**Brush** Use a medium bristle brush.

**Roller** Use a medium or long-nap synthetic roller cover with phenolic core.

## Mixing & Thinning

**Mixing** Power mix separately, then combine and power mix. DO NOT MIX PARTIAL KITS.

**Ratio** 1:1 Ratio (A to B)

**Thinning** Normally not required but may be thinned up to 25 oz/gal (20%) with Thinner #76. Use of thinners other than those supplied or recommended by Carboline may adversely affect product performance and void product warranty, whether expressed or implied.

**Pot Life** 45 minutes at 75°F (24°C). Pot life will be less at higher temperatures.

## Cleanup & Safety

**Cleanup** Use Thinner #2 or Acetone. In case of spillage, absorb and dispose of in accordance with local applicable regulations.

**Safety** Read and follow all caution statements on this product data sheet and on the MSDS for this product. Employ normal workmanlike safety precautions. Hypersensitive persons should wear protective clothing, gloves and use protective cream on face, hands and all exposed areas.

**Ventilation** When used in enclosed areas and product is thinned, thorough air circulation must be used during and after application until the coating is cured. The ventilation system should be capable of preventing the solvent vapor concentration from reaching the lower explosion limit for the solvents used. User should test and monitor exposure levels to insure all personnel are below guidelines. If not sure or if not able to monitor levels, use MSHA/NIOSH approved respirator.

**Caution** This product exotherms at the end of its pot life. Any unused quantities will become extremely hot. The material begins to thicken at the end of its pot life, which is an indication of exotherm. Immediately spread out on an appropriate surface or add sand or other suitable heat sink to the unused material to reduce the severity of exotherm. Take appropriate precautions against breathing fumes. This product when thinned contains flammable solvents. Keep away from sparks and open flames. All electrical equipment and installations should be made and grounded in accordance with the National Electric Code. In areas where explosion hazards exist, workmen should be required to use non-ferrous tools and wear conductive and non-sparking shoes.

## Application Conditions

| Condition | Material | Surface | Ambient | Humidity |
|---|---|---|---|---|
| Normal | 60°-80°F (16°-27°C) | 60°-80°F (16°-27°C) | 60°-80°F (16°-27°C) | 0-80% |
| Minimum | 60°F (16°C) | 50°F (10°C) | 50°F (10°C) | 0% |
| Maximum | 90°F (32°C) | 130°F (54°C) | 100°F (38°C) | 90% |

This product simply requires the substrate temperature to be above the dew point. Condensation due to substrate temperatures below the dew point can cause flash rusting on prepared steel. Special application techniques may be required above or below normal application conditions.

## Curing Schedule

| Surface Temp. & 50% Relative Humidity | Dry to Topcoat or Handle | Maximum Recoat Time w/ Water Borne | Maximum Recoat Time w/ Solvent Borne | Final Cure |
|---|---|---|---|---|
| 50°F (10°C) | 24 Hours | 14 Days | 30 Days | 9 Days |
| 75°F (24°C) | 12 Hours | 14 Days | 30 Days | 6 Days |
| 90°F (32°C) | 6 Hours | 7 Days | 15 Days | 3 Days |

These times are based on a 1.0-2.0 mil (25-50 micron) dry film thickness. Higher film thickness, insufficient ventilation or cooler temperatures will require longer cure times and could result in solvent entrapment and premature failure. Excessive humidity or condensation on the surface during curing can interfere with the cure, can cause discoloration and may result in a surface haze. Any haze or blush must be removed by water washing before recoating. During high humidity conditions, it is recommended that the application be done while temperatures are increasing. If the maximum recoat time is exceeded, the surface must be abraded by sweep blasting or sanding before the application of additional coats.

### Curing Schedule for Curing/Form Release Agent

| Surface Temp. & 50% Relative Humidity | Dry to Topcoat or Handle | Final Cure |
|---|---|---|
| 75°F (24°C) | 5 Hours | 6 Days |

These times are based on 5.0-10.0 (125-250 microns) mils dry film thickness.

## Packaging, Handling & Storage

**Shipping Weight (Approximate)** 0.5 Gallon Kit: 6 lbs (3 kg); 2 Gallon Kit: 22 lbs (10 kg)

**Flash Point (Setaflash)** Part A: >205°F (96°C)
Part B: >205°F (96°C)

**Storage Temperature & Humidity** 40° - 110°F (4°-43°C) Store indoors.
0-90% Relative Humidity

**Shelf Life** Part A & B: Min. 36 months at 75°F (24°C)

***Shelf Life: (actual stated shelf life) when kept at recommended storage conditions and in original unopened containers.**

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An RPM Company

December 2013

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# Safety Data Sheet
# prepared to UN GHS Revision 3

## 1. Identification of the Substance/Mixture and the Company/Undertaking

**1.1 Product Identifier** 0918A1NL

**Product Name:** CARBOGUARD 1340 PART A

**Revision Date:** 01/20/2016

**Supercedes Date:** 07/17/2015

**1.2 Relevant identified uses of the substance or mixture and uses advised against** Component of multicomponent industrial coatings - Industrial use.

**1.3 Details of the supplier of the safety data sheet**

**Manufacturer:**
Carboline Company
2150 Schuetz Road
St. Louis, MO USA 63146

Regulatory / Technical Information:
Contact Carboline Technical Services at
1-800-848-4645

**Datasheet Produced by:** Alotta, Vicki - ehs@stoncor.com

**1.4 Emergency telephone number:**
CHEMTREC 1-800-424-9300 (Inside US)
CHEMTREC +1 703 5273887 (Outside US)
HEALTH - Pittsburgh Poison Control 1-412-681-6669

## 2. Hazard Identification

**2.1 Classification of the substance or mixture**

Hazardous to the aquatic environment, Chronic, category 2
Eye Irritation, category 2
STOT, single exposure, category 3, RTI
Skin Irritation, category 2
Skin Sensitizer, category 1

### 2.2 Label elements

**Symbol(s) of Product**

**Signal Word**

Warning

**Named Chemicals on Label**

EPOXY RESIN

**GHS HAZARD STATEMENTS**

| | | |
|---|---|---|
| Skin Irritation, category 2 | H315 | Causes skin irritation. |
| Skin Sensitizer, category 1 | H317 | May cause an allergic skin reaction. |
| Eye Irritation, category 2 | H319 | Causes serious eye irritation. |
| STOT, single exposure, category 3, RTI | H335 | May cause respiratory irritation. |
| Hazardous to the aquatic environment, Chronic, category 2 | H411 | Toxic to aquatic life with long lasting effects. |

**GHS PRECAUTION PHRASES**

| | |
|---|---|
| P261 | Avoid breathing dust/fume/gas/mist/vapours/spray. |
| P273 | Avoid release to the environment. |
| P280 | Wear protective gloves/protective clothing/eye protection/ face protection. |
| P302+352 | IF ON SKIN: Wash with plenty of soap and water. |
| P304+340 | IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing. |
| P305+351+338 | IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do so. Continue rinsing. |
| P332+313 | If skin irritation occurs: Get medical advice/attention. |
| P333+313 | If skin irritation or rash occurs: Get medical advice/attention. |
| P391 | Collect spillage. |

### 2.3 Other hazards

No Information

**Results of PBT and vPvB assessment:**

The product does not meet the criteria for PBT/VPvB in accordance with Annex XIII.

## 3. Composition/Information On Ingredients

### 3.2 Mixtures

**Hazardous Ingredients**

| CAS-No. | Chemical Name | % |
|---|---|---|
| 25068-38-6 | EPOXY RESIN | 75-100 |
| 13463-67-7 | TITANIUM DIOXIDE | 2.5-10 |
| 1314-13-2 | ZINC OXIDE | 2.5-10 |
| 28553-12-0 | DIISONONYL PHTHALATE | 0.1-1.0 |

| CAS-No. | GHS Symbols | GHS Hazard Statements | M-Factors |
|---|---|---|---|
| 25068-38-6 | GHS07-GHS09 | H315-317-319-335-411 | 0 |
| 13463-67-7 | | | 0 |
| 1314-13-2 | GHS09 | H400-410 | 0 |
| 28553-12-0 | GHS06 | H331 | 0 |

**Additional Information:** The text for GHS Hazard Statements shown above (if any) is given in Section 16.

## 4. First-aid Measures

### 4.1 Description of First Aid Measures

**AFTER INHALATION:** Give oxygen or artificial respiration if needed. Remove person to fresh air. If signs/symptoms continue, get medical attention.
**AFTER SKIN CONTACT:** In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. If skin irritation persists, call a physician.
**AFTER EYE CONTACT:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.
**AFTER INGESTION:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. If swallowed, call a poison control centre or doctor immediately.

### 4.2 Most important symptoms and effects, both acute and delayed

May cause sensitization by skin contact. Irritating to eyes and skin. May be harmful if swallowed.

### 4.3 Indication of any immediate medical attention and special treatment needed

No information available on clinical testing and medical monitoring. Specific toxicological information on substances, if available, can be found in section 11.
When symptoms persist or in all cases of doubt seek medical advice.

## 5. Fire-fighting Measures

### 5.1 Extinguishing Media:

Carbon Dioxide, Dry Chemical, Foam, Water Fog

**UNUSUAL FIRE AND EXPLOSION HAZARDS:** Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas. Vapors may spread long distances and ignite.

### 5.2 Special hazards arising from the substance or mixture

No Information

### 5.3 Advice for firefighters

In the event of fire, wear self-contained breathing apparatus. Evacuate personnel to safe areas. Use NIOSH approved respiratory protection. Use water spray to cool unopened containers.

## 6. Accidental Release Measures

### 6.1 Personal precautions, protective equipment and emergency procedures

For personal protection see section 8. Ensure adequate ventilation. Evacuate personnel to safe areas. Remove all sources of ignition.

### 6.2 Environmental precautions

Do not allow material to contaminate ground water system. Prevent product from entering drains.

### 6.3 Methods and material for containment and cleaning up

Prevent further leakage or spillage if safe to do so. Contain spillage, soak up with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and transfer to a container for disposal according to local / national regulations (see section 13).

### 6.4 Reference to other sections

Please refer to disposal requirements or country specific disposal requirements for this material. See Section 13 for further information.

## 7. Handling and Storage

### 7.1 Precautions for safe handling

**INSTRUCTIONS FOR SAFE HANDLING :** Keep containers dry and tightly closed to avoid moisture absorption and

contamination. Prepare the working solution as given on the label(s) and/or the user instructions. Ensure all equipment is electrically grounded before beginning transfer operations. Do not use sparking tools. Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing. Use only with adequate ventilation/personal protection. Avoid breathing vapors, mist or gas.

**PROTECTION AND HYGIENE MEASURES :** Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday. When using, do not eat, drink or smoke.

### 7.2 Conditions for safe storage, including any incompatibilities

**CONDITIONS TO AVOID:** Heat, flames and sparks.

**STORAGE CONDITIONS:** Keep container closed when not in use. Store in a dry, well ventilated place away from sources of heat, ignition and direct sunlight.

### 7.3 Specific end use(s)

No specific advice for end use available.

## 8. Exposure Controls/Personal Protection

### 8.1 Control parameters

**Ingredients with Occupational Exposure Limits (US)**

| Name | % | ACGIH TLV-TWA | ACGIH TLV-STEL | OSHA PEL-TWA | OSHA PEL-CEILING | OEL Note |
|---|---|---|---|---|---|---|
| EPOXY RESIN | 75-100 | N/E | N/E | N/E | N/E | |
| TITANIUM DIOXIDE | 2.5-10 | 10 MGM3 | N/E | 10 MGM3 | N/E | |
| ZINC OXIDE | 2.5-10 | 2 MGM3 | 10 MGM3 | 5 MGM3 | N/E | |
| DIISONONYL PHTHALATE | 0.1-1.0 | N/E | N/E | N/E | N/E | |

**FURTHER INFORMATION:** Refer to the regulatory exposure limits for the workforce enforced in each country.

### 8.2 Exposure controls

**Personal Protection**

**RESPIRATORY PROTECTION:** In order to avoid inhalation of spray-mist and sanding dust, all spraying and sanding must be done wearing adequate respirator. Use only with ventilation to keep levels below exposure guidelines reported in this document. User should test and monitor exposure levels to ensure all personnel are below guidelines. If not sure, or not able to monitor, use State or federally approved supplied air respirator. For silica containing coatings in a liquid state, and/or if no exposure limits are established above, air-supplied respirators are generally not required.

**EYE PROTECTION:** Safety glasses with side-shields.

**HAND PROTECTION:** Gloves should be discarded and replaced if there is any indication of degradation or chemical breakthrough. Impervious gloves. Request information on glove permeation properties from the glove supplier.

**OTHER PROTECTIVE EQUIPMENT:** Ensure that eyewash stations and safety showers are close to the workstation location. Lightweight protective clothing

**ENGINEERING CONTROLS:** Avoid contact with skin, eyes and clothing. Use only in an area equipped with explosion proof exhaust ventilation. Ensure adequate ventilation, especially in confined areas.

## 9. Physical and Chemical Properties

### 9.1 Information on basic physical and chemical properties

| | |
|---|---|
| **Appearance:** | Viscous Amber Or White |
| **Physical State** | Liquid |
| **Odor** | Epoxy |
| **Odor threshold** | N/D |
| **pH** | N/D |

| | |
|---|---|
| **Melting point / freezing point (°C)** | N/D |
| **Boiling point/range (°C)** | 176 F (80 C) - 513 F (267 C) |
| **Flash Point, (°C)** | 97 |
| **Evaporation rate** | Slower Than Ether |
| **Flammability (solid, gas)** | Not determined |
| **Upper/lower flammability or explosive limits** | 0.9 - 7.1 |
| **Vapour Pressure, mmHg** | N/D |
| **Vapour density** | Heavier than Air |
| **Relative density** | Not determined |
| **Solubility in / Miscibility with water** | N/D |
| **Partition coefficient: n-octanol/water** | Not determined |
| **Auto-ignition temperature (°C)** | Not determined |
| **Decomposition temperature (°C)** | Not determined |
| **Viscosity** | Unknown |
| **Explosive properties** | Not determined |
| **Oxidising properties** | Not determined |

**9.2 Other information**

| | |
|---|---|
| **VOC Content g/l:** | 24 |
| **Specific Gravity (g/cm3)** | app 1.16 |

## 10. Stability and Reactivity

**10.1 Reactivity**
No reactivity hazards known under normal storage and use conditions.

**10.2 Chemical stability**
Stable under normal conditions.

**10.3 Possibility of hazardous reactions**
Hazardous polymerisation does not occur.

**10.4 Conditions to avoid**
Heat, flames and sparks.

**10.5 Incompatible materials**
Strong oxidizing agents.

**10.6 Hazardous decomposition products**
Carbon dioxide ($CO_2$), carbon monoxide (CO), oxides of nitrogen ($NO_x$), dense black smoke.

## 11. Toxicological Information

**11.1 Information on toxicological effects**

**Acute Toxicity:**

**Oral LD50:** N/D

**Inhalation LC50:** N/D

**Irritation:** Unknown

**Corrosivity:** Unknown

**Sensitization:** Unknown

**Repeated dose toxicity:** Unknown

**Carcinogenicity:** Unknown

**Mutagenicity:** Unknown

**Toxicity for reproduction:** Unknown

**If no information is available above under Acute Toxicity then the acute effects of this product have not been tested. Data on individual components are tabulated below:**

| CAS-No. | Chemical Name | Oral LD50 | Dermal LD50 | Vapor LC50 |
|---|---|---|---|---|
| 25068-38-6 | EPOXY RESIN | 11400 mg/kg, rat, oral | 23000 mg/kg, dermal, rabbit | >20 mL/kg skin, sensitizer |
| 13463-67-7 | TITANIUM DIOXIDE | 25000 mg/kg, oral (rat) | | Not Available |
| 1314-13-2 | ZINC OXIDE | 15000 mg/kg, oral, rat | | Not Available |
| 28553-12-0 | DIISONONYL PHTHALATE | 9750 mg/kg, oral, rat | | 9.4 mg/l / 4h, rat inh |

**Additional Information:**

May cause sensitization by skin contact. Irritating to eyes and skin. May be harmful if swallowed.

## 12. Ecological Information

**12.1 Toxicity:**

**EC50 48hr (Daphnia):** Unknown

**IC50 72hr (Algae):** Unknown

**LC50 96hr (fish):** Unknown

**12.2 Persistence and degradability:** Unknown

**12.3 Bioaccumulative potential:** Unknown

**12.4 Mobility in soil:** Unknown

**12.5 Results of PBT and vPvB assessment:** The product does not meet the criteria for PBT/VPvB in accordance with Annex XIII.

**12.6 Other adverse effects:** Unknown

| CAS-No. | Chemical Name | EC50 48hr | IC50 72hr | LC50 96hr |
|---|---|---|---|---|
| 25068-38-6 | EPOXY RESIN | 2.1 mg/l (daphnia) | 11 mg/l (algae) | 1.3 mg/l (fish) |
| 13463-67-7 | TITANIUM DIOXIDE | No information | No information | No information |
| 1314-13-2 | ZINC OXIDE | No information | No information | No information |
| 28553-12-0 | DIISONONYL PHTHALATE | No information | No information | No information |

## 13. Disposal Considerations

**13.1 WASTE TREATMENT METHODS:** Do not burn, or use a cutting torch on, the empty drum. If recycling is not practicable, dispose of in compliance with local regulations. Dispose of in accordance with local regulations. Empty containers should be taken to an approved waste handling site for recycling or disposal.

## 14. Transport Information

| | | |
|---|---|---|
| 14.1 | **UN number** | UN3082 |
| 14.2 | **UN proper shipping name** | Environmentally Hazardous Substance, Liquid, N.O.S. |
| | **Technical name** | (Epoxy Resin) |
| 14.3 | **Transport hazard class(es)** | 9 |
| | **Subsidiary shipping hazard** | N/A |
| 14.4 | **Packing group** | III |
| 14.5 | **Environmental hazards** | Marine Pollutant: Yes (Epoxy Resin) |
| 14.6 | **Special precautions for user** | Unknown |
| | **EmS-No.:** | F-A, S-F |
| 14.7 | **Transport in bulk according to Annex II of MARPOL 73/78 and the IBC code** | Unknown |

## 15. Regulatory Information

**15.1 Safety, health and environmental regulations/legislation for the substance or mixture:**

## U.S. Federal Regulations: As follows -

### CERCLA - Sara Hazard Category

This product has been reviewed according to the EPA 'Hazard Categories' promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

Acute Health Hazard, Chronic Health Hazard

### Sara Section 313:

This product contains the following substances subject to the reporting requirements of Section 313 of Title III of the Superfund Amendment and Reauthorization Act of 1986 and 40 CFR part 372:

| Chemical Name | CAS-No. |
|---|---|
| ZINC OXIDE | 1314-13-2 |

### Toxic Substances Control Act:

All components of this product are either listed on the TSCA Inventory or are exempt.

This product contains the following chemical substances subject to the reporting requirements of TSCA 12(B) if exported from the United States:

No TSCA 12(b) components exist in this product.

## U.S. State Regulations: As follows -

### New Jersey Right-to-Know:

The following materials are non-hazardous, but are among the top five components in this product.

| Chemical Name | CAS-No. |
|---|---|
| ALUMINUM HYDROXIDE | 21645-51-2 |

### Pennsylvania Right-To-Know

The following non-hazardous ingredients are present in the product at greater than 3%.

No PA Right-To-Know components exist in this product.

### California Proposition 65:

Warning: The following ingredients present in the product are known to the state of California to cause Cancer:

| Chemical Name | CAS-No. |
|---|---|
| TITANIUM DIOXIDE | 13463-67-7 |
| DIISONONYL PHTHALATE | 28553-12-0 |
| CARBON BLACK | 1333-86-4 |
| ETHYL BENZENE | 100-41-4 |
| CUMENE | 98-82-8 |

Warning: The following ingredients present in the product are known to the state of California to cause birth defects, or other reproductive hazards.

No Proposition 65 Reproductive Toxins exist in this product.

## International Regulations: As follows -

### * Canadian DSL:

No Information

**15.2 Chemical Safety Assessment:**

No Chemical Safety Assessment has been carried out for this substance/mixture by the supplier.

# 16. Other Information

**Text for GHS Hazard Statements shown in Section 3 describing each ingredient:**

| | |
|---|---|
| H315 | Causes skin irritation. |
| H317 | May cause an allergic skin reaction. |
| H319 | Causes serious eye irritation. |
| H331 | Toxic if inhaled. |
| H335 | May cause respiratory irritation. |
| H400 | Very toxic to aquatic life. |
| H410 | Very toxic to aquatic life with long lasting effects. |
| H411 | Toxic to aquatic life with long lasting effects. |

**Reasons for revision**

No Information

No Information

# Safety Data Sheet
# prepared to UN GHS Revision 3

## 1. Identification of the Substance/Mixture and the Company/Undertaking

**1.1 Product Identifier** 0918B1NL

**Product Name:** CARBOGUARD 1340 PART B

**Revision Date:** 05/30/2015

**Supercedes Date:** 29/05/2015

**1.2 Relevant identified uses of the substance or mixture and uses advised against** Component of multicomponent industrial coatings - Industrial use.

**1.3 Details of the supplier of the safety data sheet**

**Manufacturer:**
Carboline Company
2150 Schuetz Road
St. Louis, MO USA 63146

Regulatory / Technical Information:
Contact Carboline Technical Services at
1-800-848-4645

**Datasheet Produced by:** Schlereth, Ken - ehs@stoncor.com

**1.4 Emergency telephone number:**
CHEMTREC 1-800-424-9300 (Inside US)
CHEMTREC +1 703 5273887 (Outside US)
HEALTH - Pittsburgh Poison Control 1-412-681-6669

## 2. Hazard Identification

**2.1 Classification of the substance or mixture**

Acute Toxicity, Oral, category 4
Acute Toxicity, Inhalation, category 4
Hazardous to the aquatic environment, Chronic, category 2
Reproductive Toxicity, category 2
Skin Corrosion, category 1
Skin Sensitizer, category 1

## 2.2 Label elements

**Symbol(s) of Product**

**Signal Word**

Danger

**Named Chemicals on Label**

BISPHENOL A, BENZYL ALCOHOL, DIETHYLENETRIAMINE, TOFA, REACTION PRODUCTS WITH TEPA, 4-NONYL PHENOL, BRANCHED, NONYL PHENOL

**GHS HAZARD STATEMENTS**

| | | |
|---|---|---|
| Acute Toxicity, Oral, category 4 | H302 | Harmful if swallowed. |
| Skin Corrosion, category 1 | H314-1 | Causes severe skin burns and eye damage. |
| Skin Sensitizer, category 1 | H317 | May cause an allergic skin reaction. |
| Acute Toxicity, Inhalation, category 4 | H332 | Harmful if inhaled. |
| Reproductive Toxicity, category 2 | H361 | Suspected of damaging fertility or the unborn child. |
| Hazardous to the aquatic environment, Chronic, category 2 | H411 | Toxic to aquatic life with long lasting effects. |

**GHS PRECAUTION PHRASES**

| | |
|---|---|
| P260 | Do not breathe dust/fume/gas/mist/vapours/spray. |
| P264 | Wash hands thoroughly after handling. |
| P270 | Do no eat, drink or smoke when using this product. |
| P273 | Avoid release to the environment. |
| P280 | Wear protective gloves/protective clothing/eye protection/ face protection. |
| P284 | Wear respiratory protection. |
| P301+310 | IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician. |
| P302+352 | IF ON SKIN: Wash with plenty of soap and water. |
| P303+361+353 | IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower. |
| P304+340 | IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing. |
| P305+351+338 | IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do so. Continue rinsing. |
| P308+P313 | IF exposed or concerned: Get medical advice/attention |
| P333+313 | If skin irritation or rash occurs: Get medical advice/attention. |
| P391 | Collect spillage. |

## 2.3 Other hazards

Not applicable

**Results of PBT and vPvB assessment:**

The product does not meet the criteria for PBT/VPvB in accordance with Annex XIII.

## 3. Composition/Information On Ingredients

### 3.2 Mixtures

**Hazardous Ingredients**

| CAS-No. | Chemical Name | % |
|---|---|---|
| 100-51-6 | BENZYL ALCOHOL | 50-75 |
| 68953-36-6 | TOFA, REACTION PRODUCTS WITH TEPA | 10-25 |
| 111-40-0 | DIETHYLENETRIAMINE | 2.5-10 |
| 80-05-7 | BISPHENOL A | 2.5-10 |
| 84852-15-3 | 4-NONYL PHENOL, BRANCHED | 1.0-2.5 |
| TRADE SECRET | SILANE | 0.1-1.0 |
| 91672-41-2 | NONYL PHENOL | <0.1 |

| CAS-No. | GHS Symbols | GHS Hazard Statements | M-Factors |
|---|---|---|---|
| 100-51-6 | GHS07 | H302-312-319-332 | 0 |
| 68953-36-6 | GHS05-GHS07 | H314-317 | 0 |
| 111-40-0 | GHS05-GHS07 | H302-312-314-317-331 | 0 |
| 80-05-7 | GHS05-GHS07-GHS08-GHS09 | H317-318-335-361-411 | 0 |
| 84852-15-3 | GHS05-GHS07-GHS08-GHS09 | H302-314-361-400-410 | 0 |
| TRADE SECRET | | | 0 |
| 91672-41-2 | GHS07 | H302 | 0 |

**Additional Information:** The text for GHS Hazard Statements shown above (if any) is given in Section 16.

## 4. First-aid Measures

### 4.1 Description of First Aid Measures

**AFTER INHALATION:** Give oxygen or artificial respiration if needed. Remove person to fresh air. If signs/symptoms continue, get medical attention.
**AFTER SKIN CONTACT:** In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. If skin irritation persists, call a physician.
**AFTER EYE CONTACT:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.
**AFTER INGESTION:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. If swallowed, call a poison control centre or doctor immediately.

### 4.2 Most important symptoms and effects, both acute and delayed

Irritating to eyes and skin. May be harmful if swallowed.

### 4.3 Indication of any immediate medical attention and special treatment needed

No information available on clinical testing and medical monitoring. Specific toxicological information on substances, if available, can be found in section 11.

## 5. Fire-fighting Measures

### 5.1 Extinguishing Media:

Carbon Dioxide, Dry Chemical, Foam, Water Fog

**UNUSUAL FIRE AND EXPLOSION HAZARDS:** Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas. Vapors may spread long distances and ignite.

### 5.2 Special hazards arising from the substance or mixture

No Information

### 5.3 Advice for firefighters

In the event of fire, wear self-contained breathing apparatus. Evacuate personnel to safe areas. Use NIOSH approved

respiratory protection. Use water spray to cool unopened containers.

## 6. Accidental Release Measures

### 6.1 Personal precautions, protective equipment and emergency procedures

Ensure adequate ventilation. Ensure adequate ventilation. Evacuate personnel to safe areas. Remove all sources of ignition.

### 6.2 Environmental precautions

Do not allow material to contaminate ground water system. Prevent product from entering drains.

### 6.3 Methods and material for containment and cleaning up

Contain spillage, soak up with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and transfer to a container for disposal according to local / national regulations (see section 13).

### 6.4 Reference to other sections

Please refer to disposal requirements or country specific disposal requirements for this material. See Section 13 for further information.

## 7. Handling and Storage

### 7.1 Precautions for safe handling

**INSTRUCTIONS FOR SAFE HANDLING :** Keep containers dry and tightly closed to avoid moisture absorption and contamination. Prepare the working solution as given on the label(s) and/or the user instructions. Ensure all equipment is electrically grounded before beginning transfer operations. Do not use sparking tools. Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing. Use only with adequate ventilation/personal protection. Avoid breathing vapors, mist or gas.
**PROTECTION AND HYGIENE MEASURES :** Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday. When using, do not eat, drink or smoke.

### 7.2 Conditions for safe storage, including any incompatibilities

**CONDITIONS TO AVOID:** Heat, flames and sparks.
**STORAGE CONDITIONS:** Keep container closed when not in use. Store in a dry, well ventilated place away from sources of heat, ignition and direct sunlight.

### 7.3 Specific end use(s)

No specific advice for end use available.

## 8. Exposure Controls/Personal Protection

### 8.1 Control parameters

**Ingredients with Occupational Exposure Limits (US)**

| Name | % | ACGIH TLV-TWA | ACGIH TLV-STEL | OSHA PEL-TWA | OSHA PEL-CEILING | OEL Note |
|---|---|---|---|---|---|---|
| BENZYL ALCOHOL | 50-75 | N/E | N/E | N/E | N/E | |
| TOFA, REACTION PRODUCTS WITH TEPA | 10-25 | N/E | N/E | N/E | N/E | |
| DIETHYLENETRIAMINE | 2.5-10 | 1 PPM | N/E | 4 MGM3 | N/E | |
| BISPHENOL A | 2.5-10 | N/E | N/E | N/E | N/E | |
| 4-NONYL PHENOL, BRANCHED | 1.0-2.5 | N/E | N/E | N/E | N/E | |
| SILANE | 0.1-1.0 | 5PPM | NE | 5PPM | NE | |
| NONYL PHENOL | <0.1 | NE | NE | NE | NE | |

**FURTHER INFORMATION:** Refer to the regulatory exposure limits for the workforce enforced in each country.

### 8.2 Exposure controls

**Personal Protection**

**RESPIRATORY PROTECTION:** In order to avoid inhalation of spray-mist and sanding dust, all spraying and sanding must be done wearing adequate respirator. Use only with ventilation to keep levels below exposure guidelines reported in this document. User should test and monitor exposure levels to ensure all personnel are below guidelines. If not sure, or not able to monitor, use State or federally approved supplied air respirator. For silica containing coatings in a liquid state, and/or if no exposure limits are established above, air-supplied respirators are generally not required.

**EYE PROTECTION:** Safety glasses with side-shields.

**HAND PROTECTION:** Gloves should be discarded and replaced if there is any indication of degradation or chemical breakthrough. Impervious glovesRequest information on glove permeation properties from the glove supplier.

**OTHER PROTECTIVE EQUIPMENT:** Ensure that eyewash stations and safety showers are close to the workstation location. Lightweight protective clothing

**ENGINEERING CONTROLS:** Avoid contact with skin, eyes and clothing. Use only in an area equipped with explosion proof exhaust ventilation. Ensure adequate ventilation, especially in confined areas.

## 9. Physical and Chemical Properties

### 9.1 Information on basic physical and chemical properties

| | |
|---|---|
| **Appearance:** | Viscous Brown Liquid |
| **Physical State** | Liquid |
| **Odor** | Faint Amine |
| **Odor threshold** | |
| **pH** | N/D |
| **Melting point / freezing point (°C)** | N/D |
| **Boiling point/range (°C)** | 149 F (65 C) - 500 F (260 C) |
| **Flash Point, (°C)** | 97 |
| **Evaporation rate** | |
| **Flammability (solid, gas)** | |
| **Upper/lower flammability or explosive limits** | Not determined |
| **Vapour Pressure, mmHg** | N/D |
| **Vapour density** | |
| **Relative density** | |
| **Solubility in / Miscibility with water** | N/D |
| **Partition coefficient: n-octanol/water** | |
| **Auto-ignition temperature (°C)** | |
| **Decomposition temperature (°C)** | |
| **Viscosity** | Unknown |
| **Explosive properties** | |
| **Oxidising properties** | |

### 9.2 Other information

| | |
|---|---|
| **VOC Content g/l:** | 24 |
| **Specific Gravity (g/cm3)** | 1.02 |

## 10. Stability and Reactivity

**10.1 Reactivity**
No reactivity hazards known under normal storage and use conditions.

**10.2 Chemical stability**
Stable under normal conditions.

**10.3 Possibility of hazardous reactions**
Hazardous polymerisation does not occur.

**10.4 Conditions to avoid**
Heat, flames and sparks.

**10.5 Incompatible materials**
Strong oxidizing agents.

**10.6 Hazardous decomposition products**
Carbon dioxide (CO2), carbon monoxide (CO), oxides of nitrogen (NOx), dense black smoke.

## 11. Toxicological Information

**11.1 Information on toxicological effects**

**Acute Toxicity:**

**Oral LD50:** N/D

**Inhalation LC50:** N/D

**Irritation:** Unknown

**Corrosivity:** Unknown

**Sensitization:** Unknown

**Repeated dose toxicity:** Unknown

**Carcinogenicity:** Unknown

**Mutagenicity:** Unknown

**Toxicity for reproduction:** Unknown

**If no information is available above under Acute Toxicity then the acute effects of this product have not been tested. Data on individual components are tabulated below:**

| CAS-No. | Chemical Name | Oral LD50 | Dermal LD50 | Vapor LC50 |
|---|---|---|---|---|
| 100-51-6 | BENZYL ALCOHOL | 1230 mg/kg rat, oral | 2000 mg/kg, dermal, rabbit | 1000 ppm / 8 hrs rat, inhalation |
| 68953-36-6 | TOFA, REACTION PRODUCTS WITH TEPA | 4750 mg/kg oral, rat | | Not Available |
| 111-40-0 | DIETHYLENETRIAMINE | 1080 mg/kg, oral, rat | | 10 mg/L / 1 hour, inh, rat |
| 80-05-7 | BISPHENOL A | 11400 mg/kg, oral, rat | | Not Available |

| | | | |
|---|---|---|---|
| 84852-15-3 | 4-NONYL PHENOL, BRANCHED | 1620 mg/kg oral | Not Available |
| TRADE SECRET | SILANE | NOT AVAILABLE | NOT AVAILABLE |
| 91672-41-2 | NONYL PHENOL | 1620MG/KG ORAL<br>2140 MG/KG SKIN | NOT AVAILABLE |

**Additional Information:**

Irritating to eyes and skin. May be harmful if swallowed.

## 12. Ecological Information

**12.1 Toxicity:**

**EC50 48hr (Daphnia):** Unknown

**IC50 72hr (Algae):** Unknown

**LC50 96hr (fish):** Unknown

**12.2 Persistence and degradability:** Unknown

**12.3 Bioaccumulative potential:** Unknown

**12.4 Mobility in soil:** Unknown

**12.5 Results of PBT and vPvB assessment:** The product does not meet the criteria for PBT/VPvB in accordance with Annex XIII.

**12.6 Other adverse effects:** Unknown

| CAS-No. | Chemical Name | EC50 48hr | IC50 72hr | LC50 96hr |
|---|---|---|---|---|
| 100-51-6 | BENZYL ALCOHOL | No information | No information | No information |
| 68953-36-6 | TOFA, REACTION PRODUCTS WITH TEPA | No information | No information | No information |
| 111-40-0 | DIETHYLENETRIAMINE | No information | No information | No information |
| 80-05-7 | BISPHENOL A | No information | No information | No information |
| 84852-15-3 | 4-NONYL PHENOL, BRANCHED | No information | No information | No information |
| TRADE SECRET | SILANE | No information | No information | No information |
| 91672-41-2 | NONYL PHENOL | No information | No information | No information |

## 13. Disposal Considerations

**13.1 WASTE TREATMENT METHODS:** Do not burn, or use a cutting torch on, the empty drum. If recycling is not practicable, dispose of in compliance with local regulations. Dispose of in accordance with local regulations. Empty containers should be taken to an approved waste handling site for recycling or disposal.

## 14. Transport Information

| | | |
|---|---|---|
| 14.1 | UN number | N/A |
| 14.2 | UN proper shipping name | Not Regulated |
| | Technical name | N/A |
| 14.3 | Transport hazard class(es) | None |
| | Subsidiary shipping hazard | N/A |
| 14.4 | Packing group | N/A |
| 14.5 | Environmental hazards | Unknown |
| 14.6 | Special precautions for user | Unknown |
| | EmS-No.: | Unknown |
| 14.7 | Transport in bulk according to Annex II of MARPOL 73/78 and the IBC code | Unknown |

## 15. Regulatory Information

15.1 **Safety, health and environmental regulations/legislation for the substance or mixture:**

### U.S. Federal Regulations: As follows -

**CERCLA - Sara Hazard Category**

This product has been reviewed according to the EPA 'Hazard Categories' promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

Acute Health Hazard, Chronic Health Hazard

**Sara Section 313:**

This product contains the following substances subject to the reporting requirements of Section 313 of Title III of the Superfund Amendment and Reauthorization Act of 1986 and 40 CFR part 372:

| Chemical Name | CAS-No. |
|---|---|
| BISPHENOL A | 80-05-7 |

**Toxic Substances Control Act:**

This product contains the following chemical substances subject to the reporting requirements of TSCA 12(B) if exported from the United States:

No TSCA 12(b) components exist in this product.

**U.S. Clean Air Act:**

EPA Coating Category:
EPA VOC Content Limit (g/l):
Product VOC Content (g/l)
Thinning Recommendations:
Application Recommendations:

May be harmful if swallowed.

## U.S. State Regulations: As follows -

### New Jersey Right-to-Know:

The following materials are non-hazardous, but are among the top five components in this product.

| Chemical Name | CAS-No. |
|---|---|
| No NJ Right-To-Know components exist in this product. | |

### Pennsylvania Right-To-Know

The following non-hazardous ingredients are present in the product at greater than 3%.

No PA Right-To-Know components exist in this product.

### California Proposition 65:

Warning: The following ingredients present in the product are known to the state of California to cause Cancer:

| Chemical Name | CAS-No. |
|---|---|
| FORMALDEHYDE | 50-00-0 |

Warning: The following ingredients present in the product are known to the state of California to cause birth defects, or other reproductive hazards.

| Chemical Name | CAS-No. |
|---|---|
| BISPHENOL A | 80-05-7 |
| METHYL ALCOHOL | 67-56-1 |

## International Regulations: As follows -

### * Canadian DSL:

No Information

**15.2 Chemical Safety Assessment:**

No Chemical Safety Assessment has been carried out for this substance/mixture by the supplier.

## 16. Other Information

### Text for GHS Hazard Statements shown in Section 3 describing each ingredient:

| | |
|---|---|
| H302 | Harmful if swallowed. |
| H312 | Harmful in contact with skin. |
| H314 | Causes severe skin burns and eye damage. |
| H317 | May cause an allergic skin reaction. |
| H318 | Causes serious eye damage. |
| H319 | Causes serious eye irritation. |
| H331 | Toxic if inhaled. |
| H332 | Harmful if inhaled. |
| H335 | May cause respiratory irritation. |
| H361 | Suspected of damaging fertility or the unborn child. |
| H400 | Very toxic to aquatic life. |
| H410 | Very toxic to aquatic life with long lasting effects. |
| H411 | Toxic to aquatic life with long lasting effects. |

### Reasons for revision

No Information

No Information

# Attachment E

# Project Plan for Quality Assurance

# Project Plan for Quality Assurance
# Attachment E to the Work Plan for the Pilot Scale Abatement Study of Hangar 1

Hangar 1, Moffett Federal Airfield
Mountain View, California
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| 3 & 5 | Project Organization and QAPP Distribution | 2.2.3 | Distribution List | PP | Distribution List |
| | | 2.2.4 | Project Organization for Project Plan<br>Communication Pathway for Pilot Study<br>Project Schedule | PP 1.1<br>WP 3.1<br>WP 3.3 | Project Tasks and Schedule |
| 4 , 7 & 8 | Personnel Qualifications and Sign-off Sheet | 2.2.1 | Title, Version, and Approval/Sign-Off | PP<br>WP 3.2 | Title Page<br>Qualifications and Related Training for Contractors and Subcontractors |
| 6 | Communication Pathways | 2.2.7 | Special Training Requirements and Certification | WP 3.1 | Communication Pathways |
| 9 | Project Planning Session Summary | 2.2.5 | Project Background, Overview, and Intended Use of Data | WP 3 | Pilot Scale Abatement Study Planning |
| 10 | Conceptual Site Model | 2.2.5 | Project Background, Overview, and Intended Use of Data | WP 2 | Site Conditions and Background |
| 11 | Project/Data Quality Objectives | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria | PP 1.3<br>PP 1.4 | Data Quality Objectives and Data Quality Indicators<br>Measurement Quality Objectives (MQOs) |
| 12 | Measurement Performance Criteria | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria | PP 1.3<br>PP 1.4 | Data Quality Objectives and Data Quality Indicators<br>Measurement Quality Objectives (MQOs) |
| 13 | Secondary Data Uses and Limitations | Chapter 3 | QAPP Elements for Evaluating Existing Data | WP 1.1<br>WP 1.2<br>WP 2<br>WP 2.1<br>WP 3.7.1.1 | Hangar 1<br>Objectives of Pilot Scale Abatement Study<br>Site Conditions and Background<br>Condition of Hangar 1 and the Implemented NTCRA Remedy<br>Pre-Abatement Sampling |
| 14 & 16 | Project Tasks & Schedule | 2.2.4 | Project Organization and Schedule | WP 3.3 | Project Tasks and Schedule |
| 15 | Project Action Limits and Laboratory-Specific Detection / Quantitation Limits | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria | WP 3.5<br>WP 3.6<br>PP 1.4 | Target Post-Abatement Acceptance Criteria<br>Airborne Action Levels<br>Measurement Quality Objectives (MQOs) |
| 17 | Sampling Design and Rationale | 2.3.1 | Sample Collection Procedure, Experimental Design, and Sampling Tasks | WP 3.7<br>WP 3.7.1 | Planned Sample Collection Activities<br>Sampling Rationale |
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| 22 | Field Equipment Calibration, Maintenance, Testing, and Inspection | 2.3.6 | Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables | PP 3.2<br><br>WP 3.7.4.3<br><br>WP 3.7.4.4 | Field Equipment Calibration, Maintenance, Testing, and Inspection<br>Field Quality Control for XRF Sampling<br>Field Quality Control for Air Monitoring |
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# Acronyms and Abbreviations

ACC: ACC Environmental Consultants
ASTM: Association for Testing and Materials
CCB: Continuing Calibration Blank
CCV: Continuing Calibration Verification
CF: Calibration Factor
CM15: Carbomastic 15 epoxy coating
CMU: Concrete Masonite Unit
COC: Contaminant of Concern
%D: Percent difference
DDT: Dichlorodiphenyltrichloroethane
DMC: Deuterated Monitoring Compounds
DV: Data Validation
DQI: Data Quality Indicators
DQO: Data Quality Objectives
EDD: Electronic Data Deliverable
GC-MS: Gas Chromatography-Mass Spectrometry
GFAAS: Graphite Furnace Atomic Adsorption Spectrometry
HEPA: High Efficiency Particulate Air
ICAL: Initial Calibration
ICB: Initial Calibration Blank
ICP: Inductively Coupled Plasma
ICS: Interference Check Samples
ICV: Initial Calibration Verification
IDQTF: Intergovernmental Data Quality Task Force
LCS: Laboratory Control Samples
LD: Laboratory Duplicate
LOD: Limit of Detection
LTMP: Long-Term Management Plan
$m^3$: cubic meter
MB: Method Blank
MCE: Mixed Cellulose Ester
MDL: Minimum Detection Limit
MFA: Moffett Federal Airfield
$mg/m^3$: milligrams per cubic meter
mg/kg: milligrams per kilogram
MQO: Measurement Quality Objectives
MS: Matrix Spike
MSD: Matrix Spike Duplicate
N/A: Not Applicable
NAS: Naval Air Station
Navy: U.S. Department of the Navy
ND: Not-detected
NIOSH: National Institute of Occupational Safety and Health
NIST: National Institute of Standards and Technology
NPS: Non-porous steel
NTCRA: Non-Time-Critical Removal Actions
PBARCCS: precision, bias, accuracy, representativeness, comparability, completeness, and sensitivity

PCB: Polychlorinated Biphenyls
PCS: Performance Characterization Sheet
PDS: Post-Digestion Spike
PDSD: Post-Digestion Spike Duplicate
PQL: Practical Quantification Limit
Project Plan: Project Plan for Quality Assurance
PV: Planetary Ventures, LLC.
QAM: Quality Assurance Manager
QAPP: Quality Assurance Project Plan
QC: Quality Control
QL: Quantitation Limits
%R: Percent recovery
RPD: Relative Percent Difference
RSD: Relative Standard Deviation
S2BVM: Stage 2B Validation Manual
SOPs: Standard Operating Procedures
TCLP Toxicity Characteristic Leaching Procedure
TCRA: Time-Critical Removal Action
TTLC: Total Threshold Limit Concentration
ug: micrograms
ug/100 $cm^2$: micrograms per 100 square centimeters
ug/$ft^2$: micrograms per square foot
ug/$m^3$: micrograms per cubic meter
U.S.: United States
USEPA: U.S. Environmental Protection Agency
VOA: Volatile Organic Analysis
VOC: Volatile Organic Compounds
WET: Waste Extraction Test
XRF: X-Ray Fluorescence Spectrometer

# 1. Introduction

On behalf of Planetary Ventures, LLC (PV), ACC Environmental Consultants (ACC) has prepared this Project Plan, as Attachment E to the Proposed Final Work Plan for the Pilot Scale Abatement Study of Hangar 1 (Work Plan). The objective of the Pilot Scale Abatement Study (Pilot Study) is to determine the feasibility of reducing PCB and lead concentrations in accessible structural steel elements and concrete masonry unit (CMU) walls in the Hangar 1 structure. In this Attachment, the main text of the Work Plan for the Pilot Study Abatement Study is referred to as the "Work Plan."

This Project Plan has been prepared in response to comments provided by the United States Environmental Protection Agency (USEPA) in December 2015 regarding the *Draft Work Plan for the Pilot Scale Abatement Study at Hangar 1*, dated September 21, 2015. This Project Plan focuses on elements included in the USEPA comment letter related to sample collection and data evaluation; it provides elements of quality assurance project plans (QAPP) as set forth in *Guidance on Quality Assurance Project Plans* (QAPP Guidance), CIO 2106-G-05 QAPP (USEPA, 2012) and Optimized UFP-QAPP Worksheets (QAPP Worksheets; IDQTF, 2012) but is not intended to be a fully complaint QAPP. A crosswalk between the quality elements described in these two documents and the respective sections of this Project Plan and the Work Plan is included in the Table of Contents.

As requested by USEPA, this Project Plan describes the quality assurance and quality control protocols to be followed. It includes data quality objectives (DQOs), proposed sample collection activities, sampling handling, analytical methods, quality control requirements, data verification, validation, and usability. Worker health and safety monitoring protocols are included in Attachments A and B of the Work Plan and not discussed herein.

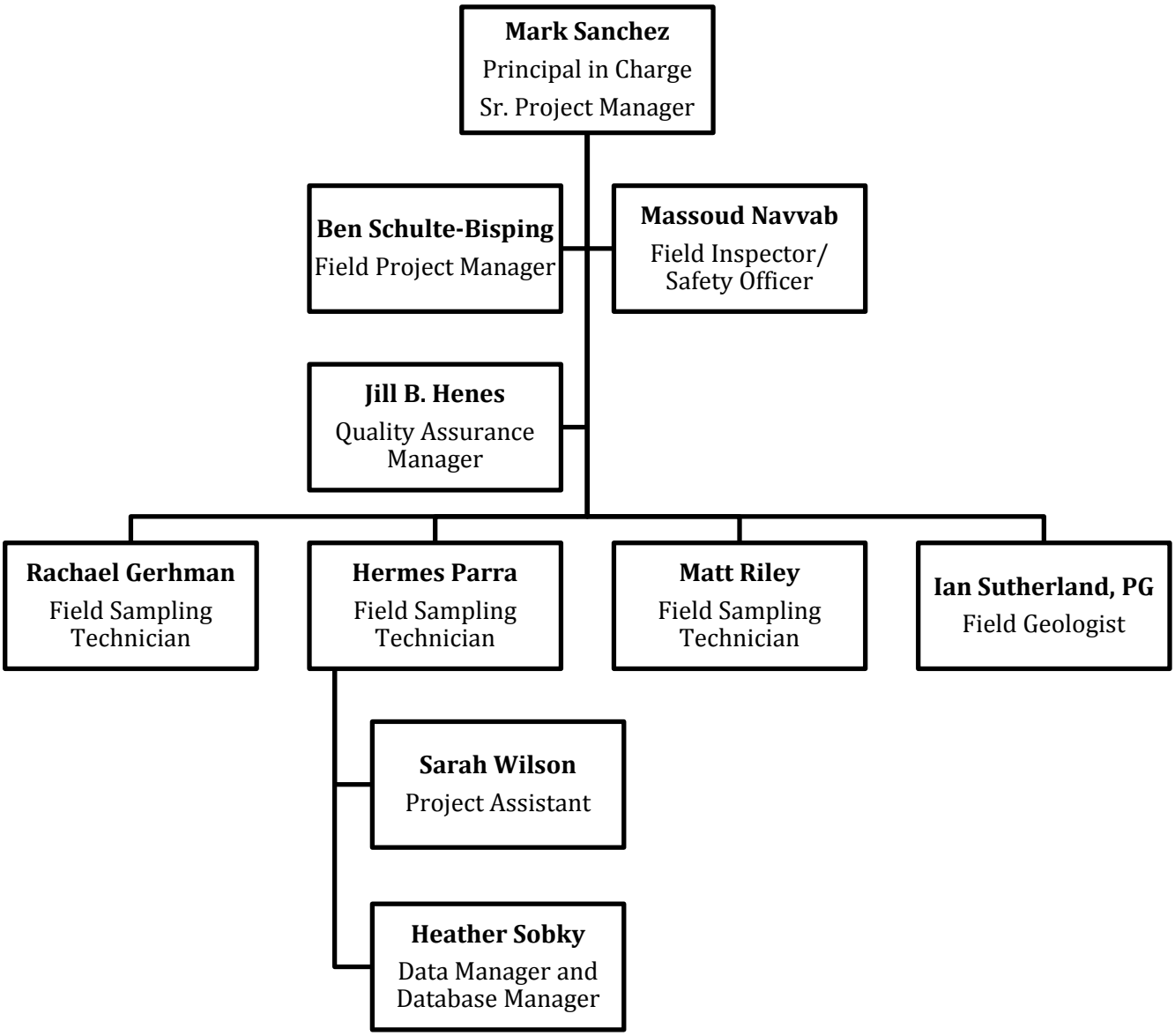
## *1.1 Project Organization*

It is not possible for any one person to complete all of the tasks set forth in the Work Plan. It is necessary for numerous individuals to work together as a team. Each member of team will be assigned a specific piece of the project that may require a different or unique set of skills. Each team member will have a clear understanding of the roles the other team members will play and of the lines of authority within the project team. The project success is dependent on each person performing his or her assigned duties effectively and with mutual cooperation amongst project team members.

The roles, responsibilities, and qualifications of the key project participants are described in Sections 3.1 and 3.2 of the Work Plan.

The Project Organizational Chart for the work specifically described in the Project Plan is presented below:

# Organizational Chart



## *1.2 Hangar 1- Background, Overview and Intended Uses of Data*

Hangar 1 is a large steel structure measuring approximately 1,133 feet long by 308 feet wide and 198 feet tall. Originally, the steel frame of Hangar 1 was covered with corrugated siding and a built-up asphalt roof, and the interior contained multi-story offices and shops located on both sides of the hangar deck, concrete electrical vaults, and a concrete floor. The area surrounding the hangar is paved, with the exception of several small areas of bare soil located on the eastern side of the hangar. A trench drain that discharges to the storm drain system surrounds the perimeter of Hangar 1.

The primary objectives of the Pilot Study are to determine the feasibility of reducing PCB and lead concentrations in accessible structural steel elements and CMU walls within the Hangar 1 structure. Data to be collected during the Pilot Study, described in the Work Plan and in this Project Plan, will be used to assess the effectiveness of different abatement techniques for PCB and lead removal.

## *1.3 Data Quality Objectives and Data Quality Indicators*

As set forth in the QAPP Guidance and QAPP Worksheets, the Data Quality Objective (DQO) process generates performance and acceptance criteria for collecting environmental data, resulting in a set of qualitative and quantitative statements that describes a data collection activity. The DQO process, which aims to facilitate data collection to meet user needs, includes these steps (USEPA, 2012):

- Define Problem Statement.
- Identify the Study Goal.
- Identify Information Inputs.
- Define the Study Boundaries.
- Develop Analytic Approach.
- Specify Performance or Acceptance Criteria.
- Develop the Detailed Plan for Obtaining Data.

### 1.3.1 *Statement of the Problem*

As described in the Work Plan, numerous investigations and remedial actions have been conducted to mitigate PCBs and lead on Hangar 1 and in associated soil and sediments. After completion of the last phase of remediation, the Navy prepared a Long-Term Management Plan (LTMP) for PCB contamination at Hangar 1 (AMEC, 2013) that outlines the coating inspection and maintenance methods and procedures and storm water sediment monitoring program that are required to maintain the viability and effectiveness of the Non-Time-Critical Removal Actions (NTCRA).

The primary objectives of the Pilot Study are to determine the feasibility of reducing PCB and lead concentrations in accessible[1] structural steel elements and CMU walls within the Hangar 1 structure to:

1) Concentrations that could potentially eliminate the need for institutional or engineering controls at Hangar 1;
2) Minimize the potential exposure of future workers to PCBs and lead during long-term operations and maintenance activities; and,
3) Minimize the need for institutional controls such as the installation and maintenance of signage regarding the potential exposure hazard and the necessity of obtaining regulatory agency approval of building modifications that might damage remedy components.

### 1.3.2 *Study Goals and Information Inputs*

The sample collection activities described below have been designed to assess: (1) effective means and methods to remove the coatings of the building superstructure, (2) coating removal technologies and engineering controls that will provide protective levels of airborne Contaminant of Concern (COCs) to workers and at the site perimeter, and (3) evaluate characteristics for disposal of waste streams generated during the pilot study and during full-scale abatement.

Study goals and specific sample collection activities to achieve each goal:

1. Evaluate effectiveness of the three different abatement techniques that are described in the Work Plan. This will be achieved by measurement of pre-abatement baseline concentrations of lead and PCB in bulk samples from the structure (paint/coating chips on structural steel members and CMU walls). Post-abatement lead and PCB concentrations remaining on the structural components will be measured using wipe samples. An X-Ray Fluorescence Spectrometer (XRF) will be used to conduct a survey of lead to better define sampling locations and to evaluate the progress of abatement activities.
2. Evaluate concentrations of fugitive dust and COCs on particulates at the perimeter of the Pilot Study area to aid in establishing engineering controls and an air monitoring program during full-scale abatement and coating removal activities. Upwind and downwind measurements of particulates and COCs on particulates will be used. Potential worker exposures are discussed further in Attachments A and B.
3. Evaluate chemical concentrations in waste streams to enable appropriate disposal of wastes created during the pilot study and to aid in determining the waste classification and quantities that are likely to be generated by the full-scale abatement project.

[1] Accessible surfaces are all surfaces throughout the Hangar 1 structure that are not tight-tolerance mated surfaces (e.g., steel members riveted together) and will include all mated surfaces with greater than a ½" gap. Caulk or other coatings will be removed prior to determining whether a surface is a tight-tolerance mated surface.

### 1.3.3 *Boundaries of the Study*

The proposed pilot scale abatement study area (Pilot Study Area) is located in the southeastern corner of Hangar 1 (see Main Text Figures 2A, 2B, and 2C) and is approximately 120 feet long by 30 feet wide by 20 feet high. Prior to the selection of this area for the proposed Pilot Study, ACC visually inspected the area for potential asbestos-containing materials and did not observe any.[2] A trench drain is located along the eastern edge of the study area and small area of bare soil is present east/southeast of the Pilot Study Area (Work Plan Figure 2A).

The Pilot Study Area was selected as it contains a number of different structural steel elements and CMU walls and is considered representative of the entire Hangar 1 structure. Figures 3A, 3B, and 3D illustrate the different types of interior and exterior structural steel elements and Figure 3C illustrates the subject CMU wall.

### 1.3.4 *Analytic Approach*

Measurement of lead and PCB concentration in various media should seek to report concentrations below Target Post-Abatement Acceptance Criteria (Work Plan Table 5) and Airborne Action Levels (Work Plan Table 6).

Waste streams will be characterized for PCBs, lead and other metals, and analyses requested by off-site disposal facilities.

### 1.3.5 *Performance and Acceptance Criteria*

PCBs and lead concentrations reported for wipe samples, bulk samples, and air samples will guide the assessments of the most effective means and methods to remove lead and PCBs on the structure and engineering controls adequacy for the site perimeter. Measurement errors could affect these assessments in several ways. For example, measurement errors could result in a decision that target post- abatement criteria have not been achieved when they have been.

Data quality indicators (DQIs) can help address these potential issues by assessing precision, bias, accuracy, representativeness, comparability, completeness, and sensitivity, or “PBARCCS”. Measurement Quality Objectives (MQOs) may be set as acceptance criteria for these DQI parameters, described as:

- Precision: the measure of agreement between replicated measurements of the same property under identical or nearly identical conditions.
- Bias: The systematic or persistent distortion of a measurement process that causes errors in one direction.
- Accuracy: the degree to which a measurement agrees with a true value.
- Representativeness: the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

[2] During the Pilot Study, if materials are encountered that are suspected of containing asbestos, work will be stopped and the suspect materials sampled and analyzed for asbestos. If asbestos is determined to be present in these materials, the materials will be removed in accordance with applicable laws and regulations before the continuation of abatement activities within the Pilot Study Area.

- Comparability: the expression of the measure of confidence that one data set can be compared with another and that the two data sets may appropriately be combined.
- Completeness: the amount of valid, usable data compared with the total amount of data collected or planned.
- Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest.

These are described below and summarized in Table A 1.

*Precision*

Precision is a quantitative measure of the variability of a group of data compared with their average value. Precision is usually stated in terms of relative percent difference (RPD) for pairs of data or relative standard deviation (RSD) for larger groups of data.

The measurement of precision depends on the sampling technique and analytical method. Field duplicate and laboratory duplicate samples will measure precision for project samples.

For a pair of measurements, RPD will be used, and RSD for a series of measurements. Equations for RPD and RSD are:

$$\text{RPD} = \frac{|D_1 - D_2|}{(D_1 + D_2) \times 0.5} \times 100$$

Where:

$D_1$ and $D_2$ = the two replicate values;

$$\text{RSD} = \frac{S}{X}; \text{ and,}$$

$$S = \sqrt{\frac{\sum (x_i - X)^2}{n-1}}$$

Where:

| | | |
|---|---|---|
| S | = | standard deviation |
| $x_i$ | = | each observed value |
| X | = | the arithmetic mean of all observed values |
| n | = | total number of values |

The precision of laboratory analysis will be assessed through the analysis of laboratory duplicate samples, field duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) samples. The frequency of field duplicate collection is described in Section 3.1.1.

*Bias*

Bias in a measurement system may result from systematic analytical error. Bias will be evaluated based on sample spiking including routine quality control (QC) samples prepared and analyzed by the primary

analytical laboratory—that is, MS/MSD samples, Laboratory Control Samples (LCS) or blank spikes, and analytical (post-digestion) spikes. Bias for each sample that is analyzed for PCBs will also be evaluated based on surrogate recovery.

*Accuracy*

Accuracy measures the overall agreement of a measurement with a known value. It includes a combination of random error, or "precision" and systematic error, or "bias," with both parameters potentially resulting from sampling or analytical error. Laboratory accuracy will be evaluated based on a program of sample spiking, potentially including analyzing MS/MSD samples, method blanks, LCSs or blank spikes, and analytical (post-digestion) spikes.

The frequency of laboratory quality control samples and data quality indicators and measurement quality objectives are included in Section 3.1.2.

Media blanks as well as MS/MSDs and LCS, will measure accuracy (see Section 3). For MS/MSDs, accuracy is calculated using this equation:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where:

| | | |
|---|---|---|
| %R | = | % recovery |
| SSR | = | spike sample result |
| SR | = | sample result |
| SA | = | amount of spike added to sample |

*Representativeness*

Representativeness is a qualitative expression of the degree to which sample data accurately and precisely represent a characteristic of a population, variations in a parameter at a sampling point, or an environmental condition. Representativeness of data will be promoted by consistently applying field procedures established in the Work Plan and this Project Plan. Sample shipping and storage will adequately preserve the sample for later analysis. Data deemed non-representative after comparison with existing data will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

*Comparability*

Comparability is a qualitative expression of the measure of confidence that one data set can be compared with another and combined for decision-making purposes. The methods presented in the Work Plan and Project Plan follow standard field procedures and laboratory methods that were used previously at the site.

*Completeness*

Completeness is defined as the percentage of data judged valid compared with the amount that was expected to be obtained under anticipated, normal conditions.

The overall completeness goal will be 90 percent, calculated as follows:

$$C\ (\%) = \frac{D}{P \times n} \times 100$$

Where:

| | | |
|---|---|---|
| D | = | number of confident quantifications |
| P | = | number of analytical parameters per sample requested for analysis |
| n | = | number of samples requested for analysis |

The percentage of usable and unusable data will also be calculated through use of the project database by the following equations:

% Usable Data = 100 × (Number of Usable Results)/(Total Number of Results)

% Unusable Data = 100 × (Number of Unusable Results)/(Total Number of Results)

*Sensitivity*

Sensitivity is the capability of a method or instrument to discriminate among measurement responses representing different levels of the variable of interest. The minimum detection limit (MDL – used by USEPA) and limit of detection (LOD -- used by NIOSH) are empirically-derived values used to estimate the lowest concentration that methods can detect in a matrix interference-free environment. The laboratory's Quantitation Limits (QLs) or Reporting Limits (RL), however named, are the lowest concentrations of detected analytes that the instrument is reliably able to quantitate. QLs should ideally be below cleanup goals (see Section 3).

### 1.3.6 *Develop the Detailed Plan for Obtaining Data*

The detailed plan for obtaining data is included in Section 3.7 and Section 4 of the Work Plan.

## *1.4 Measurement Quality Objectives (MQOs)*

MQOs developed for each of the DQI criteria are summarized in Table A 1.

Table A 1: Summary of Data Quality Indicators and Measurement Quality Objectives

| Data Quality Indicator | QC Sample or Measurement Performance Activity | Measurement Quality Objective |
|---|---|---|
| Overall Precision | Field Duplicates | See Section 3.1.1 |
| Analytical Precision | Laboratory Control Sample Duplicates | See Section 3.1.2 |
| Analytical Precision | Matrix Spike/Matrix Spike Duplicate | See Section 3.1.2 |
| Analytical Bias/Accuracy | Laboratory Control Samples | See Section 3.1.2 |
| Representativeness | Comparison of data collected after NTCRA by Navy and by PV.<br>Hold times, preservation, equipment blank, preservative blank. | Determine suitability of pilot test location.<br>Assess whether data collected for pilot test can capture desired information. |
| Comparability | Hold times, preservation, equipment blank, preservative blank. | Assess field QC measures included in Section 3.1.1 |
| Completeness | Useable data relative to total data collected | > 90% |

## 2. Sample Collection Activities

Baseline samples will be collected prior to abatement activates. Perimeter air samples will be collected during abatement activities. Post-abatement samples will be collected to assess the relative effectiveness of each of the abatement methods used during the Pilot Study.

### *2.1 Baseline Sample Collection Activities*

Baseline sample collection will be conducted to determine pre-abatement PCB and lead concentrations in paint/coating chips from the structural steel, CMU walls within the Pilot Study Area, and in soil near the Pilot Study Area. Wipe samples will be collected from the concrete foundation and concrete drainage trench adjacent to the Pilot Study Area to determine pre-Pilot Study concentrations on these surfaces. The baseline soil sample collection activities will include multi-increment surface soil sample from the exposed soil immediately adjacent to the Pilot Study Area to have data to compare pre-Pilot Study PCB and lead concentrations with post-Pilot Study concentrations in shallow soil if there is a breach in containment during Pilot Study activities.

Table 9 of the Work Plan summarizes the proposed sample locations, number of samples, and number of duplicate samples that will be collected during Baseline Sampling. Wipe samples will be collected in accordance with Association for Testing and Materials (ASTM) D6661-10 (PCBs) or ASTM E1728-10 (lead). The sampling team will be supplied with purchased copies of the referenced ASTM standards and instructed in the proper execution of the defined protocols prior to commencement of work. Other details regarding sample collection protocols are included in Section 3.7.3 of the Work Plan.

As part of baseline sample collection, airborne particulate concentrations and concentrations of airborne lead, copper, and PCBs will be monitored at two locations (one upwind and one downwind of the Pilot Study Area) over a three day period to establish background concentrations prior to work.

### *2.2 Abatement and Post-Abatement Sampling Activities*

During abatement activities, particulate concentrations and concentrations of airborne lead and PCBs will be monitored at two locations (one upwind and one downwind of the Pilot Study Area) and compared to Airborne Action Levels and concentrations measured during baseline sample collection.

Following abatement activities with each blasting technology, verification samples will be collected to assess PCB and lead concentrations; the measured concentrations will be compared to the Target Post-Abatement Acceptance Criteria before re-application of the CM15 epoxy coating. Pilot Abatement Study wastes will be characterized for disposal and disposed of at an off-site facility. Additional details on these post-abatement activities are provided below. It is anticipated that decontamination and removal of the abatement enclosure, the recoating and inspection of abated areas, and the final verification sampling will be completed within 5 to 10 working days.

Table 10 of the Work Plan summarizes the proposed post-abatement sample locations, number of samples, and number of duplicate samples that will be collected. Wipe samples will be collected in

accordance with Association for Testing and Materials (ASTM) D6661-10 (PCBs) or ASTM E1728-10 (lead). The sampling team will be provided and trained in the proper execution of the sampling methods as described in Section 3.7 of the Work Plan prior to commencement of work.

## *2.3 Waste Characterization*

Composite samples of each type of waste will be collected (see Section 4.3.3 of the Work Plan); the waste characterization samples will be representative of all construction wastes, including water effluents, dusts, and other solids, generated by the abatement technology. The samples will be analyzed for PCBs and Title 22 Metals; additional samples may be collected and/or additional analyses may be performed at the request the off-site disposal facility. After sampling by ACC, the drums and totes will be sealed, wet wiped, decontaminated, and removed from the enclosure and placed in a secure, bermed, waste staging area with appropriate signage.

## *2.4 Sampling Identification Nomenclature*

Each sample will be designated with a unique sample number based on type of sample analysis (SOIL, AIR, WIPE, BULK, XRF), contaminate of concern (e.g., lead (L), PCBs (P), copper (C)); location of sample (e.g., non-porous steel (NPS)) and other appropriate unique identifiers such as sample date (031516) and sample number. For example, WIPE-L-NPS-031516-01 would represent the first wipe sample that was collected from non-porous steel on 15 March 2016 for the analysis of lead.

## *2.5 Sample Handling, Custody Procedures, and Documentation*

The following sections summarize sample handling and chain-of-custody procedures.

### 2.5.1 *Sample Containers and Preservation*

Samples will be collected in containers and preserved as listed in Table A 2.

### 2.5.2 *Sample Labels*

All samples will be labeled in waterproof, permanent ink with the collector's initials, a unique sample identification number (well identification), date and time of sample collection, location, sample type, analytical method, and preservative used.

### 2.5.3 *Sample Packaging and Shipping*

All samples must be transported to the analytical laboratory in a cooled (0-6$^{o}$C or ice is present) container under chain-of-custody procedures. The shipping containers will be affixed with tamper-indicating custody seals or evidence tape before shipping.

### 2.5.4 *Chain-of-Custody Protocol*

Complete chain-of-custody forms will accompany the samples to the analytical laboratory. Chain-of-custody records will include the following information written in waterproof, permanent ink and placed in the cooler in a waterproof plastic bag:

- Client and project number;
- Site name;

- Name or initials and signature of sampler;
- Name of destination laboratory;
- Field sampling identification number for each sample;
- Date and time of collection for each sample;
- Number and type of containers filled with sample for each sample;
- Analysis requested for each sample;
- Preservatives used, if any, for each sample;
- Sample matrix for each sample;
- Indication of which samples are to be used for laboratory control samples (Matrix Spikes, Laboratory Duplicates, Serial Dilutions, etc.
- Signatures of all persons involved in possession of the samples; that is, “relinquished by” and “received by”;
- Dates and times of transfers of sample possession; and,
- Shipping company tracking number, if applicable.

Samples will always be accompanied by a chain-of-custody record. When transferring samples to the analytical laboratory, the individuals relinquishing and the individuals receiving the samples will sign, date, and note the time on the chain-of-custody record. A separate chain-of-custody record will accompany each transfer of samples. The method of shipment and courier name will be entered on the chain-of-custody record.

Upon completion of the log-in procedure, a copy of the signed chain-of-custody record, receiving documentation, problem reports, and log-in documentation will be forwarded to the Field Project Manager or the Data Manager for accuracy review.

## 3. Analytical Methods and Quality Control

Soil, air, bulk, and wipe samples will be analyzed using methods listed below:

- PCBs by Gas Chromatography (USEPA Method 8082 and NIOSH Method 5503)
- Lead by ICP/MS (USEPA Method 6020) and by Graphite Furnace (NIOSH Method 7105)
- Copper by ICP (NIOSH Method 7303)

Table A 2 lists the Containers, Required Preservation, and Holding Times for each Matrix.

Table A 2: Soil, Air, Bulk and Wipe Laboratory Methods, Containers, Preservation, and Hold Times

| Analytical Group | Matrix | Analytical Method | Containers | Preservation | Holding Times |
|---|---|---|---|---|---|
| PCB | Soil | USEPA Method 8082 | 8 oz. wide-mouth glass jar | Chilled Cooler – no blue ice | Extraction 14 days from collection; Analysis 40 days from extraction |
| | Air | NIOSH 5503 | Florisil containing sorbent tube | Chilled Cooler – no blue ice | Extraction 14 days from collection; Analysis 40 days from extraction |
| | Bulk | USEPA Method 8082 | Glass VOA | Chilled Cooler – no blue ice | Extraction 14 days from collection; Analysis 40 days from extraction |
| | Wipe | USEPA Method 8082 | Glass VOA | Chilled Cooler – no blue ice | Extraction 14 days from collection; Analysis 40 days from extraction |
| Lead | Soil | USEPA Method 3050/6020 | 8 oz. wide-mouth glass jar | Chilled Cooler – no blue ice | 180 days |
| | Air | NIOSH 7303 | 37mm 0.8 micron MCE filter cassette | Chilled Cooler – no blue ice | 180 days |
| | Bulk | USEPA Method 3050/6020 | Sterile Centrifuge Tubes | Chilled Cooler – no blue ice | 180 days |
| | Wipe | USEPA Method 3050/6020 | Sterile Centrifuge Tubes | Chilled Cooler – no blue ice | 180 days |
| Copper | Air | NIOSH 7303 | 37mm 0.8 micron MCE filter cassette | Chilled Cooler – no blue ice | 180 days |

## *3.1 Quality Control Requirements*

The following sections describe these quality control elements:

- Field Quality Control
- Laboratory Quality Control
- Laboratory Internal Quality Control Checks
- Laboratory Method Detection Limits

### 3.1.1 *Field Quality Control*

The field quality control checks monitor the data quality as it is affected by field procedures and conditions. Field quality control samples are listed in Table A 3 and described in Section 3.7.4 of the Work Plan. A summary of minimum quality control criteria and corrective actions for field quality control checks is included in Table A 4.

Table A 3: Field Quality Control Sample Summary

| Sample Media | Sample Location | Analytes | No. of Samples | No. of Field Duplicates | No. of Field-Equipment Blanks | No. of Media Blanks | No. of Equip. Blanks | Anticipated No. of Samples to Lab |
|---|---|---|---|---|---|---|---|---|
| Wipe | Reusable Equipment (Pre- and Post-Pilot Study) | PCBs<br>lead | 12<br>12 | 6<br>6 | 2<br>2 | 0<br>0 | 0<br>0 | 20<br>20 |
| Wipe | Scaffolding (Pre- and Post-Pilot Study) | PCBs<br>lead | 6<br>6 | 2<br>2 | 2<br>2 | 0<br>0 | 0<br>0 | 10<br>10 |
| Wipe | Concrete Floor (Pre- and Post-Pilot Study) | PCBs<br>lead | 16<br>16 | 4<br>4 | 1<br>1 | 0<br>0 | 0<br>0 | 21<br>21 |
| Wipe | Drainage Trench (Pre-Pilot Study) | PCBs<br>lead | 4<br>4 | 1<br>1 | 1<br>1 | 0<br>0 | 0<br>0 | 6<br>6 |
| Wipe | All Steel elements (Post-Abatement) | PCBs<br>lead | 27<br>27 | 9<br>9 | 3<br>3 | 0<br>0 | 0<br>0 | 39<br>39 |
| Wipe | CMU Walls (Post-Abatement) | PCBs<br>lead | 9<br>9 | 3<br>3 | 3<br>3 | 0<br>0 | 0<br>0 | 15<br>15 |
| Bulk | Soil (Pre-Pilot Study) | PCBs and lead | 6 | 2 | 0 | 0 | 2 | 10 |
| Bulk | Coatings (Pre-Abatement; all Steel elements and CMU Walls) | PCBs and lead | 12 | 4 | 0 | 0 | 2 | 18 |
| Bulk | CMU Wall (Pre- and Post-Abatement) | PCBs and lead | 6 | 2 | 0 | 0 | 4 | 12 |
| Air | Perimeter Air | PCBs<br>lead<br>copper | 12<br>12<br>4 | 2<br>2<br>2 | 0<br>0<br>0 | 2<br>2<br>1 | 0<br>0<br>0 | 16<br>16<br>7 |

Table A 4: Summary of Minimum Quality Control Criteria and Corrective Actions for Field Quality Control Checks

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Field-equipment wipe blank | 2 or 10% of samples | • <QL | • Review wipe and solvent source. |
| Media blank | 1 or 10% of samples | • <QL | • Review source of blank. |
| Equipment blank | 2 or 10% of soil, CMU wall, and/or paint/coating samples | • <QL | • Review source of contaminant, review analytical data for potential cross contamination, qualify data as appropriate |
| Field duplicate | Daily | • RPD ≤ 25% if both sample and duplicate results ≥ (5X PQL). RPD ≤ 50% if sample or duplicate results < (5X PQL) | • Evaluate total sample collection and analysis uncertainty, review findings with field sampling team and laboratory, and qualify data as appropriate. |

### 3.1.2 *Laboratory Quality Control*

The laboratory will implement its own quality control program to check that data quality objectives are being met. This section describes the laboratory quality control elements. The frequency and acceptance criteria are included in Table A 5, Table A 6, Table A 7, and Table A 8.

Laboratory Control Elements to be used for the Pilot Study include:

- Sample Preservation and Holding Time: These requirements limit changes in sample condition between the time of collection and the time of analysis.

- Method Blank (MB): This sample is of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures. This sample monitors for laboratory artifacts and contamination in the analytical system.

- Laboratory Control Sample (LCS): This sample monitors the performance of each step during laboratory preparation and analysis. This sample demonstrates laboratory method accuracy and method precision when analyzed in duplicate.

- Laboratory Duplicates (LD): These samples demonstrate laboratory method precision.

- Matrix and Analytical Spikes (MS/MSD): Matrix spikes are project samples that are spiked with a known concentration of a target analyte prior to sample processing; analytical (post-digestion) spikes are known concentrations of a target analyte added to project samples after preparation but prior to analysis. Matrix spikes help gauge the effect of the sample matrix on sample preparation, while analytical spikes assess matrix effects on the measurement methodology.

- Post-Digestion Spikes (PDS/PDSD): A portion of prepared that has been spiked with an analyte is a post digestion spike. This sample helps gauge the effect of matrix on the analysis.

- Serial Dilution Sample (SD): A sample with a concentration of an analyte at least 100 times greater than concentration in the method blank is diluted by a factor of 5. This sample monitors potential interference effects.

Either a MS/MSD pair, or alternatively, an original analysis and a laboratory duplicate (LD) pair will be used by the laboratory for assessing the precision of analyses. One MS/MSD pair or an original sample/LD sample pair are submitted for analysis with every analytical batch of 20 or fewer investigative field samples. Depending on the specific method, the laboratory may require additional sample volume for MS/MSD analysis. If insufficient sample volume is provided to the laboratory, precision and accuracy are measured by the analysis of duplicate LCSs. Prior to field sampling, the Project Manager or QAM will discuss the need for additional sample volume with the laboratory.

Calibration of analytical instruments is required to assess whether equipment is operating properly and functioning at the desired sensitivity to meet the specified data quality objectives and quantitation/method detection limits. In general, reference standards used will "bracket" the expected concentration of the samples. This generally will require the use of two to seven different standard concentration levels that are used to demonstrate the instrument's linear range in quantitation. Calibration of an instrument must be performed prior to the analysis of any sample and then at periodic intervals (continuing calibration) during the sample analyses to verify that the instrument is still calibrated. Sample concentrations are often outside the instrument's linear range and, therefore, need to be diluted and reanalyzed.

The acceptance criteria for initial calibration for the PCB analysis are ≤ 20% RSD for target analytes and ≤ 30% for surrogate compounds; for initial calibration verification standards, ≤ 20% RSD. The frequency, acceptance criteria and corrective actions for Calibrations and Laboratory Quality Control Samples are included in Table A 5 through Table A 8 below.

Table A 5: Summary of Minimum Quality Control Criteria and Corrective Actions for PCBs by Gas Chromatography (NIOSH Method 5503)

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank | One per Batch or (1 per 20 samples minimum) | • < QL | • Review data and identify source. Report samples results that are ND or >10X blank concentration.<br>• Re-prepare and reanalyze all other samples, if possible.<br>• Address in Narrative. |
| Laboratory Control Sample | One per Batch or (1 per 20 samples minimum) | • 50-135% of expected value; ≤30 RPD between MS and MSD | • High %R – Report samples results that are ND; prepare all other samples again and reanalyze, if possible.<br>Low %R – Re-prepare and reanalyze all<br>• samples, if possible.<br>Address in Narrative. |
| Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) | Initially; whenever required, due to failure of CCV | • RSD for CFs ≤20% (≤30% for Surrogate compounds)<br>• Coefficient of Determination $R^2$ ≥ 0.990 | • Terminate analysis<br>• Re-calibrate and verify before sample analysis.<br>• Address in Narrative. |
| Continuing Calibration Verification (CCV) at midpoint of ICAL | Beginning of each day, after every 10 samples, and end of run | • %D between CF of CCV and average CFs from ICAL ≤25% | • Re-calibrate and verify.<br>• Re-analyze samples back to last good CCV.<br>• Address in Narrative. |

Table A 6: Summary of Minimum Quality Control Criteria and Corrective Actions for PCBs by Gas Chromatography (USEPA Method 8082)

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank | One per Batch or (1 per 20 samples minimum) | • < QL | • Review data and identify source. Report samples results that are ND or >10X blank concentration;<br>• Prepare all other samples again and reanalyze, if possible.<br>• Address in Narrative. |
| Laboratory Control Sample | One per Batch or (1 per 20 samples minimum) | • 50-135% of expected value; ≤30 RPD between MS and MSD | • High %R – Report samples results that are ND; re-prepare and reanalyze all other samples, if possible.<br>Low %R – Re-prepare and reanalyze all<br>• samples, if possible.<br>Address in Narrative. |
| Matrix Spike/Matrix Spike Duplicate | One per Batch or (1 per 20 samples minimum) | • 50-135% of expected value; ≤30 RPD between MS and MSD | • Control on LCS.<br>• Address in Narrative. |
| Surrogate | Every standard, sample, method blank, and QC sample | • 60-150% of expected value | • High %R – Report sample if all results are ND, if any positive results, re-prepare and reanalyze sample, if possible.<br>Low %R – Re-prepare and reanalyze<br>• sample, if possible.<br>Address in Narrative. |
| Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) | Initially; whenever required, due to failure of CCV | • RSD for CFs ≤20% (≤30% for Surrogate compounds) | • Terminate analysis.<br>• Re-calibrate and verify before sample analysis.<br>• Address in Narrative. |
| Continuing Calibration Verification (CCV) at midpoint of ICAL | Beginning of each day, after every 10 samples, and end of run | • %D between CF of CCV and average CFs from ICAL ≤25% | • Re-calibrate and verify.<br>• Re-analyze samples back to last good CCV.<br>• Address in Narrative. |

Table A 7: Summary of Minimum Quality Control Criteria and Corrective Actions for Lead by GFAAS (NIOSH Method 7105)

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank | One per Batch or (1 per 20 samples minimum) | • < QL | • Review data and identify source. Report samples results that are ND or >10X blank concentration.<br>• Prepare all other samples again and reanalyze, if possible.<br>• Address in Narrative. |
| Laboratory Control Sample | One per Batch or (1 per 20 samples minimum) | • 80-120% of expected concentration | • High %R – Report samples results that are ND; re-prepare and reanalyze all other samples, if possible.<br>Low %R – Re-prepare and reanalyze all<br>• samples, if possible.<br>Address in Narrative. |
| Laboratory Duplicate (EPA 6020 only) | One per Batch or (1 per 20 samples minimum) | • 75-125% of expected concentration | • Address in Narrative. |
| Matrix Spike/Matrix Spike Duplicate | One per Batch or (1 per 20 samples minimum) | • 75-125% of expected concentration | • Control on LCS.<br>• Address in Narrative. |
| Post Digestion Spike/ Duplicate | One per Batch or (1 per 20 samples minimum) | • 75-125% of expected concentration | • Control on LCS.<br>• Address in Narrative. |
| Internal Standards | Every standard, sample, method blank, and QC sample | • Method Control Limits | • Dilute sample 5-fold and reanalyze.<br>• Recalibrate and reanalyze sample.<br>• Address in Narrative. |

Table A 8: Summary of Minimum Quality Control Criteria and Corrective Actions for Metals by ICP/MS (EPA Method 6020) or Cu by ICP (NIOSH Method 7303)

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Method Blank | One per Batch or (1 per 20 samples minimum) | • < QL | • Review data and identify source. Report samples results that are ND or >10X blank concentration.<br>• Prepare all other samples again and reanalyze, if possible.<br>• Address in Narrative. |
| Laboratory Control Sample | One per Batch or (1 per 20 samples minimum) | • 80-120% of expected concentration | • High %R – Report samples results that are ND; re-prepare and reanalyze all other samples, if possible.<br>Low %R – Prepare all other samples again<br>• and reanalyze, if possible.<br>Address in Narrative. |
| Laboratory Duplicate (EPA 6020 only) | One per Batch or (1 per 20 samples minimum) | • Water: RPD <± 20 for samples >5X QL; ± QL for samples <5X QL<br>• Solids: RPD <± 40 for samples >5X QL; ± 2X QL for samples <5X QL | • Address in Narrative. |
| Matrix Spike/Matrix Spike Duplicate (EPA 6020 only) | One per Batch or (1 per 20 samples minimum) | • 75-125% of expected concentration | • Control on LCS.<br>• Address in Narrative. |
| Post Digestion Spike/Post Digestion Spike Duplicate (EPA 6020 only) | One per Batch or (1 per 20 samples minimum) | • 75-125% of expected concentration | • Address in Narrative. |
| Serial Dilution (EPA 6020 only) | One per Batch or (1 per 20 samples minimum) | • ± 10% difference from original results for analyte concentrations greater than 50 times the MDL | • Address in Narrative. |
| Internal Standards (EPA 6020 only) | Every standard, sample, method blank, and QC sample | • 60-125% of expected Intensity | • Dilute sample 5-fold and reanalyze. Recalibrate and reanalyze sample.<br>• Address in Narrative. |
| Initial Calibration (minimum blank + 1 calibration standard) (ICAL) | Initially, Daily; whenever required, due to failure of CCV | • Acceptable ICV, CRQL check standard, and ICB standards | • Terminate analysis<br>• Re-calibrate and verify before sample analysis. |
| Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards) | Daily, immediately following ICAL and prior to sample analysis | • 90-110% of expected concentration | • Terminate analysis and identify and document problem.<br>• Re-prepare and re-analyze ICV and all associated samples.<br>• Re-calibrate and re-analyze re-prepped ICV and all associated samples. |
| Continuing Calibration Verification (CCV) | Before samples, after every 10 samples, and end of run | • 90-110% of expected concentration | • Re-calibrate and verify.<br>• Re-analyze samples back to last acceptable CCV. |

| QC Element | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Low Concentration Verification Standard | After ICV, but before sample analysis | • 65-135% of expected concentration | • Re-prepare and re-analyze standard.<br>• Re-calibrate and verify. |
| ICP Interference Check Samples (ICS) | Run at start and finish of daily run or twice per 8 hours | • 80-120% of expected concentration | • Re-prepare and re-analyze standard.<br>• Re-calibrate, verify, and re-analyze all associated samples. |

### 3.1.3 *Laboratory Internal Quality Control Checks*

Laboratories selected for the work herein are included in Table A 9. These laboratories are State of California-certified analytical laboratories that have written operating procedures documenting and defining the instrumentation, maintenance, calibration, method detection limits, QC analyses, internal standards, acceptance criteria, etc. for the analytical procedures used. While not all of this information will be reviewed as part of the validation process, all of it will be available if needed. The laboratory will conduct internal QC checks to evaluate whether sampling procedures and laboratory analytical method performance are within acceptable limits of precision, bias, and accuracy, with these objectives:

- Document procedures, including any modifications to established administrative or technical procedures.
- Check that analytical procedures are validated and conducted according to method guidelines and laboratory quality assurance.
- Monitor laboratory performance using a systematic inspection program.
- Properly report and archive data.

Table A 9: Selected Laboratories

| Analytical Group | Matrix | Analytical Method | Analytical Laboratories | Available Sample Turn Around Times |
|---|---|---|---|---|
| PCB | Soil | EPA Method 8082 | • C<br>• T | 1-5 Day |
| | Air | NIOSH 5503 | • C<br>• T<br>• M | 1-5 Day |
| | Bulk | EPA Method 8082 | • C<br>• T<br>• M | 1-5 Day |
| | Wipe | EPA Method 8082 | • C<br>• T<br>• M | 1-5 Day |
| Lead | Soil | EPA Method 6020 | • C<br>• T<br>• M | 1-5 Day |
| | Air | NIOSH 7303 / EPA 6020 | • C<br>• T | 1-5 Day |
| | Bulk | EPA Method 3050/6020 | • C<br>• T<br>• M | 1-5 Day |
| | Wipe | EPA Method 3050/6020 | • C<br>• T<br>• M | 1-5 Day |
| Copper | Air | NIOSH 7303 / EPA 6020 | • C<br>• T<br>• M | 1-5 Day |

Abbreviations:
C: Curtis and Tompkins, Berkeley, CA
M: McCampbell Analytical, Inc., Pittsburg, CA
T: Test America, Sacramento, CA

### 3.1.4 *Laboratory Specific Detection and Quantification Limits*

The method detection limit (MDL) or Limit of Detection as used in USEPA Methods and limit of detection (LOD) as used by NIOSH is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero.

The quantitation limit (QL) or reporting limit (RL), however named, is the lowest concentration reliably achieved within limits of precision and accuracy, typically at the lowest point in the calibration curve during routine operating conditions. The QL is a multiple of the MDL/LOD for each analyte. Laboratory-specific QLs will be evaluated if a new laboratory is chosen. The nominal MDLs/LODs and QLs may not be achievable for all samples due to matrix interferences, qualification by the laboratory's QC program, or multiple dilutions during analysis. In such cases, the laboratory will report the MDLs/LODs and QLs it believes were demonstrably achievable. Table A 10 includes laboratory specific detection and quantification limits. Table A 11 compares these limits with Target Post-Abatement Acceptance Criteria and Air Action Levels.

Table A 10: Laboratory Specific Detection and Quantitation Limits

| Analytical Group | Matrix | Analytical Method | Limit of Detection (LOD) | Method Detection Limit (MDL) | Quantitation Limit (QL) | Analytical Laboratory |
|---|---|---|---|---|---|---|
| PCB | Soil | EPA Method 8082* | N/A | 1.93-7.96 ug/kg | 12-24 ug/kg | • C |
| | | | | 2.7-7.4 ug/kg | 33.0 ug/kg | • T |
| | Air | NIOSH 5503 (1) | N/A | N/A | 0.14-0.27 ug/m$^3$ | • C |
| | | | N/A | N/A | 2.5 ug/m$^3$ | • T |
| | | | 6.25 ug/m$^3$ | N/A | 6.25 ug/m$^3$ | • M |
| | Bulk | EPA Method 8082* | N/A | 0.00193-0.00796 mg/kg | 0.012-0.024 mg/kg | • C |
| | | | | 0.0022-0.0332 mg/kg | 0.05 mg/kg | • M |
| | | | | 0.003-0.007 mg/kg | 0.033 mg/kg | • T |
| | Wipe | EPA Method 8082 | N/A | N/A | 2-4 ug/wipe | • C |
| | | | | 0.025 ug/wipe | 0.05 ug/wipe | • M |
| | | | | 0.250 ug/wipe | 1.00 ug/wipe | • T |
| Lead | Soil | EPA Method 6020 | N/A | 0.0668 mg/kg | 0.250 mg/kg | • C |
| | | | | 0.094 mg/kg | 0.50 mg/kg | • M |
| | | | | 0.0600 mg/kg | 0.100 mg/kg | • T |
| | Air | NIOSH 7303 / EPA 6020 (2) | N/A | N/A | 0.026 mg/kg | • C |
| | | | N/A | N/A | 0.013 mg/kg | • T |
| | Bulk | EPA Method 3050/6020 | N/A | 0.07 mg/kg | 0.25 mg/kg | • C |
| | | | | 0.094 mg/kg | 0.5 mg/kg | • M |
| | | | | 0.06 mg/kg | 0.1 mg/kg | • T |
| | Wipe | EPA Method 3050/6020 | N/A | N/A | 0.25 ug/wipe | • C |
| | | | | 0.5 ug/wipe | 0.5 ug/wipe | • M |
| | | | | 0.06 ug/wipe | 0.2 ug/wipe | • T |
| Copper | Air | NIOSH 7303 (2) | N/A | N/A | 0.026 ug/m$^3$ | • C |
| | | | N/A | N/A | 0.13 ug/m$^3$ | • T |
| | | | 0.360 ug/m$^3$ | N/A | 0.360 ug/m$^3$ | • M |

Notes:

* The quoted LODs, QLs, and MDLs for PCBs represent the range of these values for nine Aroclors.

(1) – Assuming a 40 Liter Sample

(2) – Assuming a 1,920 Liter Sample

Abbreviations:

C: Curtis and Tompkins, Berkeley, CA

M: McCampbell Analytical, Inc., Pittsburg, CA

T: Test America, Sacramento, CA

Table A 11: Target Post-Abatement Acceptance Criteria and Air Action Levels Compared to Lab Detection and Quantitation Limits

| Analytical Group | Matrix | Analytical Method | Quantitation Limit (QL) | Lab | Acceptance Criteria/Action Levels |
|---|---|---|---|---|---|
| PCBs | Soil | EPA Method 8082* | 12-24 ug/kg<br>33.0 ug/kg | • C<br>• T | 1 mg/kg (i.e., 1,000 ug/kg) or no greater than pre-abatement background levels |
| | Air | NIOSH 5503 (1) | 0.14-0.27 ug/m$^3$<br>2.5 ug/m$^3$<br>6.25 ug/m$^3$ | • C<br>• T<br>• M | 0.013 mg/m$^3$ (i.e., 13 ug/m$^3$) |
| | Wipe | EPA Method 8082 | 2-4 ug/wipe<br>0.05 ug/wipe<br>1.00 ug/wipe | • C<br>• M<br>• T | <10 ug/100 cm$^2$ |
| Lead | Soil | EPA Method 6020 | 0.250 mg/kg<br>0.50 mg/kg<br>0.100 mg/kg | • C<br>• M<br>• T | 320 mg/kg or no greater than pre-abatement background levels |
| | Air | NIOSH 7303 / EPA 6020 (2) | 0.026 ug/m$^3$<br>0.013 ug/m$^3$ | • C<br>• T | 0.001 mg/m$^3$ (i.e., 1 ug/m$^3$) |
| | Wipe | EPA Method 3050/6020 | 0.026 ug/m$^3$<br>0.360 ug/m$^3$<br>0.357 ug/m$^3$ | • C<br>• M<br>• T | <40 ug/ft$^2$ (interior floor surfaces/CMU wall);<br><250 ug/ft$^2$ for interior horizontal surfaces |

Notes:
* The quoted LODs, QLs, and MDLs for PCBs represent the range of these values for nine Aroclors.
(1) – Assuming a 40 Liter Sample
(2) – Assuming a 1,920 Liter Sample

Abbreviations:
C: Curtis and Tompkins, Berkeley, CA
M: McCampbell Analytical, Inc., Pittsburg, CA
T: Test America, Sacramento, CA

## *3.2 Field Equipment Calibration, Maintenance, Testing, and Inspection*

This section describes calibration, maintenance, testing and inspection of field equipment including: Air Sampling Pumps, Aerosol Monitors, XRF, and differential manometers.

Air sampling pumps will be calibrated using low flow rotameters in the field. Rotameters will calibrated using a Dri CAL Primary Gas Flow Calibrator prior to use. The calibration is done in the field by disconnecting the

tubing from the unit, turning the unit on and setting it to zero. Once done the tubing is reattached to the unit.

PM10 Particulate air monitors will be calibrated daily or per sampling event. All equipment will be factory calibrated and serviced prior to use on the pilot study. Each instrument in the field will be zeroed out in accordance with manufactures procedures prior to use at the beginning of the shift and after lunch break. The zeroing procedure is accomplished by attaching a High Efficiency Particulate Air (HEPA) filter to the air inlet of the instrument. The instrument is then put into the Zero Calibration mode. After completing the Zero Calibration process the instrument will read "Zero Cal Complete" showing a steady concentration of 0.0 $mg/m^3$. The instrument is now ready for use. If the instrument shows erratic zero readings or a negative concentration is shown which is an indication of Zero Drift, then all hose connections will be checked for tightness and the HEPA filter will be replaced and the unit will be rerun through the calibration procedure. If the instrument continues to fail the Zero Calibration procedure then the unit will be replaced with another unit.

The Niton XRF will be checked by a technician using the Performance Characterization Sheet (PCS) included in Attachment F of the Work Plan to confirm that the instrument meets calibration check tolerances. A minimum of three calibration check readings using the NIST Standards will be conducted before the beginning of the inspection and after every four hours of testing. A set of calibration check readings will also be collected at the end of each workday.

Digital Differential manometers will be used to continuously monitor negative pressure differentials of the containment enclosure.

Additional information regarding the calibration of flow rates is included in Attachment F of the Work Plan.

## *3.3 Data Management*

### 3.3.1 *Field Data*

Daily field activities will be documented in the site field log books and on forms, noting the Project Name, arrival and departure dates and times, location identification, sample identification, descriptions of sampling activities, instrument calibration details, field screening results, challenges that arise during the day and their resolution, weather conditions, site visitors, and other information deemed necessary.

The Field Team Leader will be responsible for managing and forwarding field data in proper format.

### 3.3.2 *Analytical Data*

The analytical data will be reported with Stage 2B data packages. Stage 2B Data Validation is performed on Laboratory-provided Summary Forms; raw data is not evaluated. Stage 2B is appropriate for the Pilot Study because the data will be used to evaluate relative efficiencies of abatement techniques. The data will not be used to establish clean up goals or clearance levels. The laboratory will provide analytical results both in hardcopy format and as an electronic data deliverable (EDD). The electronic file will be converted into database files maintained by the Data Manager (see Section 3.1 of the Work Plan), and the resulting database entries will be printed and compared manually with the hardcopy analytical reports. Any discrepancies will be discussed with the laboratory. Data packages that are to be verified and validated

manually in accordance with Stage 2B guidelines (see Section 4.1 of this document) may be labeled Stage_2B_Validation_Manual (S2BVM).

In order for the Data Validation Consultant (DV Consultant) or QAM to perform Stage 2B checks, it is necessary for the laboratory to provide Inorganic Summary Forms for lead and copper and Organic Summary Forms for PCB analysis including all of the calibration and quality control results associated with each method. In addition to a standard level 2 report, the deliverables listed in Table A 12 and Table A 13 are required:

Table A 12: Stage 2B Laboratory Deliverables for PCBs by Gas Chromatography (USEPA Method 8082 or NIOSH Method 5505)

| | |
|---|---|
| 1. | Sample Data (sort by Laboratory sample #) |
| | 1.1. Results Data Sheet (for both columns, optional for 2nd column) |
| | 1.2. Comparison of both column results with %D calculation. |
| 2. | Run Logs (sort by Instrument & Date) |
| | 2.1. Run Logs from the data system or logbook |
| 3. | Initial Calibration Data (sort by ICAL/ICV Instrument & Date) |
| | 3.1. Initial Calibration QC Summary Form (for both columns) |
| | 3.2. Retention Time Summary Form (for both columns) |
| | 3.3. ICV QC Summary Form (for both columns) - to follow ICAL data |
| 4. | Continuing Calibration Data (sort by Instrument & Date) |
| | 4.1. CCV QC Summary Form (for both columns) |
| 5. | Quality Control Data (sort by Instrument & Date) |
| | 5.1. Surrogate Recoveries Summary Form |
| | 5.2. Method Blank/LCS/LCSD Association Summary |
| | 5.3. Method Blank Results Summary Forms (for both columns) |
| | 5.4. LCS/LSCD QC Summary Form |
| | 5.5. LCS/LCSD Results Summary Forms (for both columns) |
| | 5.6. MS/MSD QC Summary Form |
| | 5.7. MS/MSD Results Summary Forms (for both columns) |
| 6. | Preparation/Extraction Logs (if applicable) |
| | 6.1. Sample Preparation Logbook Pages |
| 7. | Chain-of-Custody Records |
| | 7.1. Chain-of-Custody Records and Receiving Documentation |
| | 7.2. Completed Internal COC (if applicable) |
| 8. | Anomalies Report |
| | 8.1. Completed Anomalies Reports |

Table A 13: Stage 2B Laboratory Deliverables for Lead by ICP/MS (USEPA Method 6020) and by GFAAS (NIOSH Method 7105) and Copper by ICP (NIOSH Method 7303)

| | |
|---|---|
| 1. | Sample Data (sort by Laboratory sample #) |
| | 1.1. Sample Results Data Sheets (must include dates and times of analysis and both preparation and analytical batch information for each analyte). |
| 2. | Calibration Summary Forms (sort by Instrument & Sequence) |
| | 2.1. Tuning Report (if applicable) |
| | 2.2. Daily Performance Report from the data system (if applicable) |
| | 2.3. ICB/CCB Summary Form |
| | 2.4. ICV/CCV Summary Form |
| | 2.5. Low-Level and / or High Level CCV Summary Form(s) (if applicable) |
| | 2.6. Interference Check Summary Form (ICS-A/ICS-AB) (if applicable) |
| 3. | Quality Control Summary Forms (sort by Instrument & Date) |
| | 3.1. Method Blank Summary Form |
| | 3.2. LCS/LCSD QC Summary Form |
| | 3.3. MS/MSD QC Summary Form |
| | 3.4. PDS/PDSD QC Summary Form (if applicable) |
| | 3.5. Serial Dilutions |
| | 3.6. Method Detection Limits |
| | 3.7. Linear Ranges |
| | 3.8. Interelement Correction Factors |
| | 3.9. All raw data from the data system (run logs, tune, instrument printouts) |
| 4. | Preparation Logs (if applicable) |
| | 4.1. Sample Preparation Logbook Report |
| 5. | Chemical Receiving Logs and Standards Preparation Logs |
| 6. | Chain-of-Custody Records |
| | 6.1. Receiving Documentation |
| | 6.2. Completed Internal COC (if applicable) |
| 7. | Standard Concentrations and Sample Calculations |
| 8. | Anomalies Report |
| | 8.1. Completed Anomalies Report |

Some data will not be available via the EDDs or will not be found on the laboratory reporting sheets—Examples include the identity of blind QC samples and most of the field-recorded information. Data of this type will be manually checked, potentially by means of direct entry into interim databases and provided separately to the DV Consultant

EDD files will be archived by the Data Manager. Data review, verification, and validation are described below.

### 3.3.3 *Traceability*

Traceability is the extent to which data can be substantiated by hardcopy documentation. Logs and record-keeping specified in this Work Plan will document sample collection, sample shipping and storage, laboratory analyses, and quality assurance and quality control.

### 3.3.4 *Data Reduction*

Data reduction involves generating qualitative and quantitative site information through observations, field procedures, analytical measurements, and calculations. QC measures developed to promote data quality are described throughout this Work Plan.

Field data obtained from direct-read instruments will be used as reported from the instruments, while other data may require data reduction of field measurements.

A legible record of each calculation will be prepared on paper or in a computer spreadsheet. Each calculation will be checked by a qualified person. Both the originator and reviewer will initial the calculation sheet, and the original sheet will be placed in the project files. Data transformation of units will be performed within the spreadsheet database, where practicable, to reduce transcriptional errors. The number of significant figures will remain unchanged regardless of any data transformation; results will not be rounded or truncated.

The laboratory will reduce analytical data in accordance with the method protocols and laboratory SOPs (Appendix B).

### 3.3.5 *Project Records*

Current hardcopy project information will be placed in the Project Manager's project files. Non-current documents will be stored in retrievable boxes at a separate storage facility. During fieldwork, photocopies of project documents will be stored at the Field Staff's offices.

## 4. Data Verification, Data Validation, Usability, and Reporting

Data generated during the Pilot Test will be verified and validated to assess compliance with data quality objectives and to determine usability. Data that fail to substantively meet data quality objectives will be rejected and clearly documented.

### *4.1 Data Verification and Data Validation*

Data verification is a completeness check to confirm that all required activities were conducted, all specified records (both field and laboratory) are present, and the contents of the records are complete. Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods/SOPs (Appendix B), and the measurement performance criteria. The QA/QC checklists included in Appendix A will be used to assist tracking data verification and validation.

Analytical laboratories will provide a copy of all receiving documentation including signed Chain-of-Custody (COC) records, condition of samples upon receipt, any anomalies encountered during receipt of the samples, and a list of analyses to be performed. The Project Manager or designee will review these documents for completeness and correctness in a timely manner.

Verification will be performed at the start of the data review process by evaluating the completeness, correctness, and conformance of a data set against data generation requirements. For example, the chain-of-custody forms will be reviewed to confirm that they were properly filled out, that field quality control blanks and duplicates were collected, and that the requested testing matches the monitoring requirements. Laboratory sample receipt reports will be reviewed to verify that the samples arrived in good condition and that the requested analyses will be or have been completed.

Validation will be performed as data are provided by the laboratory. As noted above, Stage 2B data packages will be requested from the laboratory. 100% of the field sample data for the Pilot Study will undergo formal verification and validation procedures in accordance with the "National Functional Guidelines for Inorganic Data Review" (USEPA, 2014), "National Functional Guidelines for Organic Data Review" (USEPA, 1999), with guidance from the "National Functional Guidelines for Organic Data Review" (USEPA, 2014) because the PCB samples are to be analyzed by SW-846 methodology and not by CLP methodology, and with the processes described for Stage 2B Verification and Validation checks found in "*Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*", EPA 540-R-08-005 (USEPA, 2009). The minimum checks (if applicable) are as follows:

- Documentation is present that identifies the laboratory receiving and conducting analyses that includes all samples submitted by the project or requester for analyses.
- Requested analytical methods were performed and the analysis dates are present.
- 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).
- Requested target analyte result units are reported (along with their associated uncertainty units if required, e.g., for radiochemical analyses).
- 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).

- 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.
- 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.
- Requested methods (handling, preparation, cleanup, and analytical) are performed.
- 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.
- 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).
- Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes, post digestion spikes) have been added, as appropriate.
- Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.
- Frequency of QC samples is checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch).
- 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.
- 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.
- Appropriate number and concentration of initial calibration standards are present.
- 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.
- Reported samples are bracketed by CCV standards and CCBs standards as appropriate.
- 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).
- Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatography-mass spectroscopy (GC-MS) tunes have been run every 12 hours).
- 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.

In order for the DV Consultant or QAM to perform these checks, it is necessary for the laboratory to provide summary forms for all of the calibration and quality control results associated with each method. The requested laboratory deliverables are presented in Section 3.3.2.

After reviewing all of the data in a data set, the DV Consultant or QAM will prepare and submit a Data Validation Report which identifies any problems found with the data quality. Based on a comparison of the reported results to specified measurement performance criteria, the DV Consultant or QAM will assign data qualifiers as appropriate. Potential impacts on project-specific data quality objectives will be discussed in the data validation report.

Data validation will be assessed by comparing QC parameters to the appropriate criteria of limits specified in this Work Plan. Review of laboratory controls will be documented in the QA/QC Checklist A 1 included in Appendix A: Field Forms and Checklists. Laboratory control includes verification of sample preservation and holding times are included in Table A 2.

Laboratory control samples, laboratory duplicates such as MSDs, surrogates will be assessed using criteria provided in Table A 5 through Table A 8. A summary of findings (i.e., results of blank analyses and RPD for each media for each analyte) will be presented in Quality Control Charts. QA/QC Checklist A 2 will be used if samples or data are flagged.

Field quality control data and field records will verified as complete and validated as appropriate, including:

- Compilation of daily field notes
- Compilation of equipment calibration records
- Sampling diagrams/surveys
- Chain-of-Custody records
- Relevant Correspondence

## *4.2 Data Usability*

The outcome of the data review verification and validation process is an assessment of data usability—that is, whether the data can appropriately be relied on for project decision making. The general recommendations for qualifying data presented in this Work Plan are not meant to be rigid, but instead should be modified using professional judgment to best resolve issues discovered during data validation. Data that have passed the verification review and appropriate validation review and are judged to be valid are included in the calculation of the completeness goal. The percentage of usable results and unusable results will be also calculated.

## *4.3 Reporting*

The sampling and laboratory methods and procedures detailed the Work Plan and this Project Plan are intended to provide data of sufficient quantity and quality to meet the objectives of the project. A usability report will be prepared. The report will discuss and compare overall precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level. The report will describe limitations on the use of project data if criteria for data quality indicators are not met. The report will be included as an attachment to the Implementation Report.

The report will also include observations and comments regarding the performance of each removal method to achieve the following:

- Effectiveness in achieving post abatement acceptance criteria
- Waste minimization: Which method produced the least waste?
- Water use: Which method used the least amount of water?
- Ease of use
- Safety of use: PPE efficiency, personal air sampling comparisons
- Equipment performance: Which equipment required least maintenance?

Each removal method will be rated on each of these criteria (best, acceptable, and not acceptable). A removal method will be eliminated if it fails to achieve the concentrations specified in the post abatement criteria. A final decision will be made by weighing the performance of each option against the desirable characteristics.

## 5. References

IDQTF, 2012, Optimized UFP-QAPP Worksheets, Intergovernmental Data Quality Task Force Uniform Federal Policy (IDQTF) for Quality Assurance Project Plans

NIOSH, 1994, Polychlorobiphenyls: Method 5503, Issue 2, *NIOSH Manual of Analytical Methods (NMAM)*, Fourth Edition, The National Institute for Occupational Safety and Health, August 1994.

NIOSH, 1994, Lead by GFAAS: Method 7105, Issue 2*, NIOSH Manual of Analytical Methods (NMAM)*, Fourth Edition, The National Institute for Occupational Safety and Health, August 1994.

NIOSH, 2003, Elements by ICP (Hot Block/HCl/HNO3 Digestion): Method 7303, Issue 1*, NIOSH Manual of Analytical Methods (NMAM)*, Fourth Edition, The National Institute for Occupational Safety and Health, August 1994.

USEPA, 1994, Inductively Coupled Plasma - Mass Spectrometry, Method 6020, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846),* United States Environmental Protection Agency, December 1994.

USEPA, 1996, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Method 8082, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846),* United States Environmental Protection Agency, December 1996.

USEPA, 1999, *National Functional Guidelines for Organic Data Review* United States Environmental Protection Agency, October 1999.

USEPA, 2009, *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*", EPA 540-R-08-005, United States Environmental Protection Agency, January 2009.

USEPA, 2012, *Final Draft Guidance on Quality Assurance Project Plans*, CIO 2016-G-05 QAPP, United States Environmental Protection Agency, January 2012.

USEPA, 2014, *National Functional Guidelines for Organic Data Review*, United States Environmental Protection Agency, August 2014.

# Appendix A

# Field Forms and Checklists

Project:
Sample Date:
COC No.:
Lab ID (SDG) No.:

# QA/QC Checklist A 1: Stage 2B Data Review

Sampling Contractor _____

Chain of Custody #_____

Lab ID #_____

Data Reviewer_____
(Name)

Samples Collected: _____
(Date)

Samples Analyzed: _____
(Date)

Results Received on time_____
(Date)

Laboratory _____

Sample ID: _____

Lab Batch #: _____

When all QA/QC procedures have been completed and verified, please sign and file original with lab report. Alert Database Manager that the QC is complete and specify whether any data require flagging and whether revisions to the original report were made.

- ❑ Are data flagged for quality?
- ❑ Are data flagged for results detected below the RLs?

Signature of Project Manager Date

| | Acceptable | Comments | Date Resolved If Additional Info. Needed | Initials |
|---|---|---|---|---|
| 1. Chain-of-Custody Complete and Correct | ❑ Yes ❑ No | | | |
| 2. Required Analyses Reported | ❑ Yes ❑ No | | | |
| 3. Holding Times | ❑ Yes ❑ No | | | |
| 4. Laboratory Report Complete, Signed, Dated, on Time | ❑ Yes ❑ No | | | |
| 5. Field Blanks | ❑ Yes ❑ No | | | |
| 6. Field Duplicates | ❑ Yes ❑ No | | | |
| 7. Laboratory Method Blanks | ❑ Yes ❑ No | | | |
| 8. Matrix Spikes/Matrix Spike Duplicates | ❑ Yes ❑ No | | | |
| 9. Laboratory Control Samples | ❑ Yes ❑ No | | | |
| 10. Instrument checks | ❑ Yes ❑ No | | | |
| 11. Calibrations | ❑ Yes ❑ No | | | |
| 12. Surrogates / Internal Standards | ❑ Yes ❑ No | | | |
| 14. Reporting Limits Met | ❑ Yes ❑ No | | | |
| 151. Other Issues Identified | ❑ Yes ❑ No | | | |
| 16 Data Reasonable | ❑ Yes ❑ No | | | |
| 17. Were any revised laboratory report sheets submitted? | ❑ Yes ❑ No | | | |
| 18. Has an updated EDD been provided, if needed, based on laboratory report sheets | ❑ Yes ❑ No | | | |
| 19. Is any corrective action needed by the sampling contractor? | ❑ Yes ❑ No | | | |
| 15. Is re-sampling necessary? | ❑ Yes ❑ No | | | |

Project:
Sample Date:
COC No.:
Lab ID (SDG) No.:

# QA/QC Checklist A 1: Stage 2B Data Review (continued)

Continue discussion below, if needed.

Information Request/Corrective Action / Data Quality Discrepancy

Date Action was Initiated: _____ Date Action was Completed: _____

Description of Problem:

Recommended Course of Action:

Describe Resolution:

# QA/QC Checklist A 2: Additional QA/QC Worksheet

Project:
Sample Date:
COC No.:
Lab ID (SDG) No.:

| Sample | Analyte(s) | Method | Lab Flag | Verification Flag | Comments/Reasons for Flagging |
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**Wipe Sampling Log Form**

*Project Name/Address:*

*Project Number:*

*Sampler Signature(s):*

*Sampling Protocol: __ HUD/E1782 (Lead) __ D6661 (PCBs) __ D6966 (Metals)*

| # | Sample ID | Sample Location | Sample Substrate | Date | Time | Clean the template | Don new gloves | Install the template, tape down | Select wipe, ensure moist & free from dust, fungus or material | Place at once corner, make first pass side to side in an "s" motion for template opening | Fold wipe over (dirty side in), Make second pass, top to bottom in an "s" motion | Fold wipe over (dirty side in), Make third pass, around the edge of the template | Fold wipe over and place in centrifuge tube | Label Container, Note Sample location on Drawing & Log on COC | Place in Cooler / Transport Container | Note Sampling Area Dimension or Size | Discard Gloves |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FB | | | Field Blank | | | | | | | | | | | | | | |
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A·C·C ENVIRONMENTAL CONSULTANTS

| Report Results | | | |
|---|---|---|---|
| Report To: | | Phone: | |
| Email Address: | | | |
| Turnaround Time: | | | |

# BULK & WIPE SAMPLE ANALYSIS REQUEST FORM

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Project Name: | | | | | | | |
| Project Address: | | | | Analysis Requested | | | |
| ACC Project Number: | | | | ☐ PLM: Standard | ☐ TEM: Qualative | ☐ PCB's (Arochlors): Bulk | ☐ Bacteria |
| Collected By: | | Date: | | ☐ PLM: Point Count (400) | ☐ Lead: Bulk | ☐ PCB's (Arochlors): Wipe | ☐ Particulate |
| Notes/ Comments: | | | | ☐ PLM: Point Count (1000) | ☐ Lead: Wipe | ☐ Fungi: Direct Exam | ☐ Other |

| Sample ID | Sample Date | Sample Time | # Containers | Method Preserved | Material Description | Sample Location | Size/Area |
|---|---|---|---|---|---|---|---|
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| Released By (Name): | | Released By (Signature): | | Date: | | Time: | |
| Received By (Name): | | Received By (Signature): | | Date: | | Time: | |
| Laboratory Performing Analysis: | | | | | | | |

www.accenv.com
Northern California: 7977 Capwell Drive, Suite 100 • Oakland, CA 94621 • (510) 638-8400 • Fax (510) 638-8404
Southern California: 1055 Wilshire Boulevard, Suite 1450 • Los Angeles, CA 90017 • (213) 353-1240 • Fax (213) 353-1244

| Report Results | | | |
|---|---|---|---|
| Report To: | | Phone: | |
| Email Address: | | | |
| Turnaround Time: | | | |

A·C·C ENVIRONMENTAL CONSULTANTS

# AIR SAMPLE ANALYSIS REQUEST FORM (v2015.12.09)

| | | | | Analysis Requested | | Filter Medium | Filter Pore Size | |
|---|---|---|---|---|---|---|---|---|
| Project Name: | | | | | | | | |
| Project Address: | | | | ☐ PCM: NIOSH 7400 Method | ☐ Flame AA | ☐ MCE | ☐ 0.8 µm (PCM) | |
| ACC Project Number: | | Collected By: | | ☐ TEM: AHERA | ☐ Non-Viable Fungi | ☐ PCV | ☐ 0.45 µm (TEM) | |
| Work Activity: | | | | ☐ TEM: Level II | ☐ Nuisance Dust | ☐ Matched Weight | ☐ Other/ Notes | |
| Notes: | | | | ☐ TEM: 7402 Method | ☐ Other/ Notes | ☐ Absorbent Tubes | Manometer ID: | |

| Sample Number | Lab ID Number | Date Collected | Type* | LPM On / LPM Off | Average Flow Rate (Liters per Minute) | Time On / Time Off | Total Time (Minutes) | Total Volume (Liters of Air) | Sample Information Type/ Location Description/ Notes | Fibers | Fields |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | Results (f/cc) | |
| | | | | | | | | | | | |
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| Sample Type* | FB = Field Blank | OSHA = Personal | Released By: | | Signature: | | Date: | | Time: | |
|---|---|---|---|---|---|---|---|---|---|---|
| B = Background | IWA = Inside Work Area | O = Outside Air | Recieved By: | | Signature: | | Date: | | Time: | |
| C = Clearance | OWA = Outside Work Area | P = Perimeter | Laboratory Performing Analysis: | | | | | | | |

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# CHAIN OF CUSTODY RECORD

PAGE _____ OF _____

CONSULTING ENGINEERS AND SCIENTISTS 1870 Ogden Drive, Burlingame CA 94010 PHONE: 650-292-9100 FAX: 650-552-9012

**Project Name:** Hangar 1 Pilot Scale Abatement Study

**Project No.**

**Location:**

**Sampled By:**

**Reporting:**

Electronic Format: EDF Hard Copy Format: PDF

EPA Data Report Level: II Reporting Basis: Dry

Please report results to the following:
(1) Jill Henes: henes.jill@gmail.com
(2) _____: _____@accenv.com
(3) _____: _____@accenv.com
(4) John Montgomery-Brown: jmontgomery-brown@ekiconsult.com
(5) EKI: labs@ekiconsult.com

**Laboratory:**

Curtis & Tompkins
2323 5th Street Berkeley, CA
94710
(510) 486-0900

**ANALYSES REQUESTED**

**EKI COC No.:** (YYYYMMDD-#)

Revision: _____ (A, B, C, D, etc.)

Date: By:

| Field Sample Identification | Lab Sample No. | Date | Time | Matrix | Number / Type of Container (Preservative) | ISM Sample Preparation (Method No.) | Lead (EPA 6020) | PCBs (EPA 8082) | Percent Moisture (ASTM D2216) | | | | | PLACE ON HOLD | EXPECTED TURNAROUND TIME | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | X | X | X | X | | | | | | Standard | See below for minimum ISM protocols |
| | | | | | | | | | | | | | | | | |
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**Special Instructions:** **Please see attached "Laboratory Handling Instructions for ISM Preparation of Soils Samples" for minimum ISM preparation protocols.**
**Temperature blank included**

| Relinquished by: | (Signature/Affiliation) | Date | Time | Received by: | (Signature/Affiliation or Carrier/Air Bill No.) |
|---|---|---|---|---|---|
| Relinquished by: | (Signature/Affiliation) | Date | Time | Received by: | (Signature/Affiliation) |
| Relinquished by: | (Signature/Affiliation) | Date | Time | Received by: | (Signature/Affiliation) |

# Laboratory Handling Instructions for ISM Preparation of Soil Samples

*General*

1) All samples shall be sieved and mixed upon receipt by the laboratory in accordance with the laboratory's standard procedures.

2) For PCBs analysis, concentrations are expected to be <20 mg/kg, so pursuant to United States Environmental Protection Agency (USEPA) Method 3550C the sample aliquot size for extraction shall be 30 grams.

3) For all samples tested for lead, a 10-gram sample aliquot shall be used for digestion. Digest this sample in five 2-gram digestion vials and combine digestates[1] prior to analysis by USEPA Method 6020.

4) For PCBs analysis, report Aroclor 1262 and 1268 in addition to the standard Aroclors. Positive results must be reported from an instrument with an acceptable 5-point initial calibration curve and a valid MDL study. Non-detect results must be reported from an instrument with a valid Method Detection Limit Study (MDL as defined by 40 CFR Part 136, Appendix B).

5) For lead analysis quality control, run an MS, MSD, and duplicate for precision and accuracy.

*Sample Splitting for Integrated Sampling Methodology (ISM)*

1) For select samples listed on the chain-of-custody (CoC), after completing the laboratory's sieving/mixing procedure (see General Item 1):
    a. collect one aliquot of the sample for each analysis using standard procedures;
    b. collect a second aliquot of the sample for each analysis using the ISM subsampling procedure described below (systematic 30-increment sub-sampling).

*ISM Laboratory Subsampling Procedure (for select samples listed on chain of custody):*

1) Evenly distribute the sieved and mixed sample (from General Item 1) across a stainless steel baking sheet. Collect a minimum of 30 subsamples of the material on the baking sheet. The subsamples shall be collected in a systematic manner (e.g., divide the baking sheet into 30 quadrants and collect a subsample from the center of each quadrant). Each of the subsamples shall be of approximately equal size and collected with a stainless steel rectangular spatula with sides designed for the collection of representative small increments of particulate solids. While it is not necessary to decontaminate the spatula between subsamples, the spatula shall be decontaminated between each sample.

[1] When combining the digestates for analysis ensure that the ratio of the final volume to the weight of the sample is consistent with standard laboratory practices (i.e., if a 2-gram sample is diluted to a final volume of 50 milliliters (mL) prior to analysis, then the 10-gram sample should be diluted to a final volume of 250 mL prior to analysis).

a. For the analysis of PCBs, the total mass of the 30 subsamples[2] shall be approximately 30 grams; the entire 30 gram sample shall be used to prepare an extract for analysis by USEPA Method 8082.

b. For the analysis of lead (Pb), the total mass of the 30 subsamples[3] shall be approximately 10 grams. In accordance with the CoC instructions, prepare two additional aliquots as detailed in (1) above of the specified sample for the MS and Duplicate quality control check.

2) Prepare one sample from the remaining homogenized material for moisture testing and report results on a dry weight basis.

3) Prepare and analyze laboratory blank samples. These laboratory blank samples shall be subject to the same ISM subsampling procedures used to prepare the actual samples for analysis. Laboratory grade sand or another compound such as sodium sulfate, broken glass, or glass beads shall be used for the blank to mimic the subsampling procedure.

   Note: For metals, the laboratory may use sand that is cleaned by repeated digestion with acids for blanks. The laboratory may also consider the use of broken glass or glass beads. For PCBs, the laboratory may use Laboratory grade sand.

4) Include copies of the laboratory preparatory bench sheets with the final report.

[2] Record the total number of subsamples used to create the 30 gram sample for PCB analysis on the Laboratory Sample Split/Preparation Log.
[3] Record the total number of subsamples used to create the 10 gram sample for lead analysis on the laboratory Sample Split/Preparation Log.

# DAILY PROJECT CHECKLIST (V2015.12.09)

A·C·C ENVIRONMENTAL CONSULTANTS

## Project Information

| | | | |
|---|---|---|---|
| | | Date: | |
| Project Name: | | | |
| Project Address: | | | |
| Client Name: | | ACC Project Number: | |
| ACC Field Technician: DOSH Certification No. | | ACC Project Manager: | |

## Shift Activity Summary
Check All That Apply

| Mobilization | Pre-Cleaning | Setup | Containment Inspection | Bulk Removal | Detail Cleaning | Load-Out | Visual Inspection | Tear Down | De-Mobilize |
|---|---|---|---|---|---|---|---|---|---|
| ☐ | ☐ | ☐ | ☐ | ☐ | ☐ | ☐ | ☐ | ☐ | ☐ |

| Work Area Location | General Debris Materials Removed During Shift | Regulated Materials (Asbestos & Lead) Removed During Shift | Quantity SF/ LF | |
|---|---|---|---|---|
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|---|---|---|---|
| Total No. of Work Areas: | | ☐ | See Notes for Additional Work Areas/ Materials Not Listed Above |

| ACM OSHA Work Class Check All That Apply | ☐ Class I | ☐ Class II | ☐ Class III | ☐ Class IV |
|---|---|---|---|---|

## Removal Activity Details
Check All That Apply

| | | | |
|---|---|---|---|
| | ☐ ACM Wall Materials | ☐ Lead-Based Coating (LBC) | ☐ Mold-Impacted Materials |
| ☐ Asbestos Removal ACM and ACCM | ☐ ACM Roofing Materials | ☐ Lead-Containing Coating (LCC) | ☐ Water Damaged Materials |
| ☐ ACM Contractor Assistance | ☐ ACM TSI/Insulation Materials | ☐ Lead Glazed Ceramic Tile | ☐ Lighting Wastes |
| ☐ ACM Spot Abatement | ☐ ACM Surfacing Materials | ☐ Loose & Peeling Lead Paint | ☐ PCB Ballasts |
| ☐ ACM Ceiling Materials | ☐ ACM Miscellaneous Materials | ☐ Lead Sheeting | ☐ Mercury Vapor Light Tubes |
| ☐ ACM Flooring Materials | ☐ Lead Removal LBC and LCC | ☐ Indoor Air Quality (IAQ) | ☐ Mercury Thermostat Switches |

## Contractor Information

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Contractor Name: | | | | Foreman Name: | | | |
| Crew Size: Including Supervisor | | No. of Personnel Samples: | | 8-hour TWA: | | Excursion: 30 Minute | |
| Start Time: 24-Hour Format | | Lunch Time: 24-Hour Format | | Finish Time: 24-Hour Format | | Total Hours: | |

## Personal Protection Equipment (PPE)
Check All That Apply

| | | | | |
|---|---|---|---|---|
| ☐ ½ Face Respirator | ☐ HEPA/ P100 Cartridges | ☐ Piggy-back Cartridges | ☐ Hard Hat | ☐ Gloves |
| ☐ Full Face Respirator | ☐ Organic Vapor Cartridges | ☐ Disposable Suit | ☐ Safety Glasses | ☐ Steel Toe/Shank Boots |
| ☐ PAPR | ☐ Acid Gas Cartridges | ☐ Neon Vest | ☐ Hearing Protection | ☐ Fall Protection |
| ☐ Supplied Air Respirator | ☐ Ammonia Cartridges | ☐ Other: | | |

**Project Number:** **Date:**

## Air Monitoring Information

| Air Sampling Performed by ACC During Shift? | ☐ Yes ☐ No | No. of Samples Collected by ACC During Shift: | |
|---|---|---|---|

| No. of Samples | Type of Sample | | Sample Number(s) | | | | |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

| Onsite PCM Analysis Performed? | ☐ Yes ☐ No | Name of Analyst: | |
|---|---|---|---|
| Laboratory Name, City: | | | |

## Engineering Controls & Work Area Set-Up

| | | | | |
|---|---|---|---|---|
| ☐ Negative Pressure Enclosure | ☐ Splash Guards | ☐ Three-Stage w/Shower | ☐ Building Power | ☐ No Odor Mastic Remover |
| ☐ Mini Containment | ☐ Drop Sheet | ☐ Two-Stage w/Hudson | ☐ Temp Power Box | ☐ Wet Removal Methods |
| ☐ Clean Cube | ☐ View Ports | ☐ One-Stage w/Hudson | ☐ Contractor Supplied Power | ☐ NPU Charcoal Filters |
| ☐ Glove Bags | ☐ English Warning Signs | ☐ "Z" Flap Air-Locks | ☐ GFCI Protection | ☐ Fire Extinguishers |
| ☐ Critical Barriers | ☐ Spanish Warning Signs | ☐ No Decon Required | ☐ Temporary Lighting | ☐ DOP Test Air Filtration Unit |
| ☐ Poly Walls (min 4-mil.) | ☐ Hazard Barrier Tape | ☐ Separate Load-Out | ☐ Contractor Supplied Water | ☐ DOP Test HEPA Vacuum |
| ☐ Poly Floors (min. 6-mil) | ☐ -0.02" Negative Pressure | ☐ Shut Down HVAC | Exhaust Location: | |
| ☐ Poly Ceiling (min 4-mil.) | ☐ -0.04" Negative Pressure | ☐ Protect Existing Floor | Other: | |

## Contractor Work Practice Information

| | Yes | No | NA |
|---|---|---|---|
| Have copies of worker documents been received from the contractor in compliance with the scope of work? | ☐ | ☐ | ☐ |
| Are 'OSHA' personal air monitoring sample results being posted daily? | ☐ | ☐ | ☐ |
| Are workers going through the proper decontamination sequence upon leaving the work areas? | ☐ | ☐ | ☐ |
| Are good safety practices being followed at the job site? | ☐ | ☐ | ☐ |
| Are workers demonstrating good "housekeeping" techniques? | ☐ | ☐ | ☐ |
| Is ACM (greater than >1%) being bagged and labeled as asbestos waste? | ☐ | ☐ | ☐ |
| Is water being used continuously to mist air, wet materials during removal and keep waste bags/ materials saturated? | ☐ | ☐ | ☐ |
| Are waste containers properly lined with poly, labeled, sealed, secured/ locked to prevent public access? | ☐ | ☐ | ☐ |

## Waste Information

| Waste Type | Manifest Type | Manifest Number | Date | Transporter(s) |
|---|---|---|---|---|
| | ☐ Hazardous Waste<br>☐ Non-Hazardous Waste<br>☐ Recycled | | | 1.<br>2. |
| | ☐ Hazardous Waste<br>☐ Non-Hazardous Waste<br>☐ Recycled | | | 1.<br>2. |
| EPA ID Number: | | | | |
| Was Regulated Waste Transported Off-Site During the Work Shift? | | ☐ Yes ☐ No | | |

# DAILY PROJECT SUMMARY (V2015.12.09)

A·C·C ENVIRONMENTAL CONSULTANTS

| Project Information | | Date: | |
|---|---|---|---|
| Project Name | | | |
| Project Address: | | | |
| Client Name: | | ACC Project Number: | |
| ACC Field Technician:<br>DOSH Certification No. | | ACC Project Manager: | |

## Summary of Activities

| Time (24 hr.) | Activity/ Observation/ Notable Occurrence | Manometer Reading |
|---|---|---|
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**ACC Field Technician Signature, DOSH Certification No:**

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# DAILY PROJECT SUMMARY CONTINUED

A·C·C ENVIRONMENTAL CONSULTANTS

| Project Information | | Date: | |
|---|---|---|---|
| Project Name: | | | |
| Project Address: | | | |
| Client Name: | | ACC Project Number: | |
| ACC Field Technician:<br>DOSH Certification No. | | ACC Project Manager: | |

## Summary of Activities

| Time (24 hr.) | Activity/ Observation/ Notable Occurrence | Manometer Reading |
|---|---|---|
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**ACC Field Technician Signature, DOSH Certification No:**

# ACC Field Sketch

| Project Information | | Date: | |
|---|---|---|---|
| Project Name: | | | |
| Project Address: | | | |

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A·C·C ENVIRONMENTAL CONSULTANTS

# CONTRACTOR WORKER CERTIFICATION CHECKLIST (V2015.12.09)

| Project Information | | | | | |
|---|---|---|---|---|---|
| | | | Project Start Date: | | Project End Date: |
| Project Name: | [Subject] | | | | |
| Project Address: | | | | | |
| Client Name: | | | ACC Project Number: | [Title] | |
| ACC Field Technician: DOSH Certification No. | [Manager] | | ACC Project Manager: | | |
| Contractor Name: | [Company] | | Supervisor Name: | [Status] | |
| Type of Work: Check All That Apply | ☐ Asbestos Class I | ☐ Asbestos Class II | ☐ Asbestos Class III | ☐ Asbestos Class IV | ☐ Lead ☐ Mold |

| | | | Expiration Dates | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Name | Asbestos Classification | Lead Classification | Asbestos Supervisor | Asbestos Worker | Lead Supervisor | Lead Worker | Medical Exam | Fit Test (1/2 Face) | Fit Test (Full Face) | Fit Test (PAPR) |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |

# CONTRACTOR WORKER CERTIFICATION CHECKLIST (V2015.12.09)

A·C·C ENVIRONMENTAL CONSULTANTS

## Project Information

| | | | |
|---|---|---|---|
| Project Name: | [Subject] | | |
| ACC Field Technician:<br>DOSH Certification No. | [Manager] | ACC Project Number: | [Title] |
| Contractor Name: | [Company] | Supervisor Name: | [Status] |

| | | | Expiration Dates | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Name | Asbestos Classification | Lead Classification | Asbestos Supervisor | Asbestos Worker | Lead Supervisor | Lead Worker | Medical Exam | Fit Test (1/2 Face) | Fit Test (Full Face) | Fit Test (PAPR) |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |
| | N/A | N/A | | | | | | | | |

# CONTAINMENT INSPECTION CHECKLIST (V2015.12.09)

A·C·C ENVIRONMENTAL CONSULTANTS

Note deficiencies & corrections in comments section.

| Project Information | | | |
|---|---|---|---|
| | | Date: | [Publish Date] |
| Project Name: | | | |
| Project Address: | | | |
| Client Name: | | ACC Project Number: | [Subject] |
| ACC Field Technician:<br>DOSH Certification No. | | ACC Project Manager: | |
| Contractor Name: | | Supervisor Name: | |
| Containment Location: | | | |
| Type of Work<br>Check All that Apply | ☐ Asbestos Class I ☐ Asbestos Class II ☐ Asbestos Class III ☐ Asbestos Class IV ☐ Mold ☐ Other: | | |

| Site Observations | Yes | Deficient* | Corrected* | N/A | Correction Date & (Time) |
|---|---|---|---|---|---|
| Is the work area isolated? | ☐ | ☐ | ☐ | ☐ | |
| Are OSHA Notifications posted? | ☐ | ☐ | ☐ | ☐ | |
| Are EPA/NESHAPS notifications posted? | ☐ | ☐ | ☐ | ☐ | |
| Are warning signs (English & Spanish) posted at all entrances to work area? | ☐ | ☐ | ☐ | ☐ | |
| Is access to work area adequately restricted? | ☐ | ☐ | ☐ | ☐ | |

| Setup and Containment Inspection | Yes | Deficient* | Corrected* | N/A | Correction Date & (Time) |
|---|---|---|---|---|---|
| Is polyethylene sheeting flame retardant? | ☐ | ☐ | ☐ | ☐ | |
| Are critical barriers installed over doors windows and other openings? | ☐ | ☐ | ☐ | ☐ | |
| Has the HVAC system been shut down, locked out and registers covered? | ☐ | ☐ | ☐ | ☐ | |
| Are 2 layers of poly (6 mil.) on the floor and 2 layers (4 mil.) on the walls? | ☐ | ☐ | ☐ | ☐ | |
| Is poly adequately secured to walls and floors? | ☐ | ☐ | ☐ | ☐ | |
| Are drop cloths in place? | ☐ | ☐ | ☐ | ☐ | |
| Are emergency exits identified? | ☐ | ☐ | ☐ | ☐ | |
| Is there adequate lighting (200 watts/1000 square feet)? | ☐ | ☐ | ☐ | ☐ | |
| Have temporary power system equipped with GFCI been installed? | ☐ | ☐ | ☐ | ☐ | |
| Waste load-out path-of-travel protected? | ☐ | ☐ | ☐ | ☐ | |
| Is dumpster lined with OSHA warning signs installed? | ☐ | ☐ | ☐ | ☐ | |
| Are site conditions or pre-existing damage noted and photographed? | ☐ | ☐ | ☐ | ☐ | |
| Are EPA, UN and OSHA waste labels on-site & ready for waste containers? | ☐ | ☐ | ☐ | ☐ | |
| Is local ventilation in-place for the work activities? | ☐ | ☐ | ☐ | ☐ | |

| Negative Pressure Systems | Yes | Deficient* | Corrected* | N/A | Correction Date & (Time) |
|---|---|---|---|---|---|
| If required, is a strip or circle-chart manometer installed and functioning properly? | ☐ | ☐ | ☐ | ☐ | |
| Is negative pressure measuring to project requirements? | ☐ | ☐ | ☐ | ☐ | |
| If DOP testing of equipment is required, have they been tested? | ☐ | ☐ | ☐ | ☐ | |
| Are failed DOP-tested units removed from site or clearly marked to prevent use? | ☐ | ☐ | ☐ | ☐ | |
| Has containment passed smoke test & with no stagnant air present? | ☐ | ☐ | ☐ | ☐ | |

**Project Number:** [Subject] **Date:** [Publish Date]

## Emergency and Safety Equipment

| | Yes | Deficient* | Corrected* | N/A | Correction Date & (Time) |
|---|---|---|---|---|---|
| Are SDS sheets on site and accessible? | ☐ | ☐ | ☐ | ☐ | |
| Is there and adequate first-aid kit on site? | ☐ | ☐ | ☐ | ☐ | |
| Are all fire extinguishers inspected (yearly and monthly) and up-to date? | ☐ | ☐ | ☐ | ☐ | |
| Are emergency numbers posted on site, with routes to the hospital? | ☐ | ☐ | ☐ | ☐ | |
| Is a floor plan indicating all exits and major equipment posted? | ☐ | ☐ | ☐ | ☐ | |
| Is the main power in the work area shutdown and locked out? | ☐ | ☐ | ☐ | ☐ | |
| Are all power cords elevated off the floor to prevent electrical hazards and tripping? | ☐ | ☐ | ☐ | ☐ | |
| Does all scaffolding have safety rails, toe-kicks & fall protection if necessary? | ☐ | ☐ | ☐ | ☐ | |
| Is there a designated area for resting & eating with drinking water available? | ☐ | ☐ | ☐ | ☐ | |

## Decontamination Unit

| | | Yes | Deficient* | Corrected* | N/A | Correction Date & (Time) |
|---|---|---|---|---|---|---|
| Clean Room | Are ceilings and walls covered with poly? | ☐ | ☐ | ☐ | ☐ | |
| | Are linens and/or towels available? | ☐ | ☐ | ☐ | ☐ | |
| | Is there an air lock to the shower? | ☐ | ☐ | ☐ | ☐ | |
| | Are entrance doors properly constructed? | ☐ | ☐ | ☐ | ☐ | |
| Shower Facility | Are soap, shampoo and towels available? | ☐ | ☐ | ☐ | ☐ | |
| | Is HOT water available? | ☐ | ☐ | ☐ | ☐ | |
| | Is the floor beneath the shower pan properly protected? | ☐ | ☐ | ☐ | ☐ | |
| | Does the shower provide a good spray? | ☐ | ☐ | ☐ | ☐ | |
| | Is water being filtered through a 3-stage to 1-micrometer filter? | ☐ | ☐ | ☐ | ☐ | |
| Dirty Room | Is there a bag for disposal of protective clothing? | ☐ | ☐ | ☐ | ☐ | |
| | Is there a drop cloth on the floor? | ☐ | ☐ | ☐ | ☐ | |
| | Is there an air lock to the work area? | ☐ | ☐ | ☐ | ☐ | |
| | Are air locks properly constructed? | ☐ | ☐ | ☐ | ☐ | |

### Equipment Decontamination Unit

| | | | | | |
|---|---|---|---|---|---|
| Is there a separate decontamination chamber designated for equipment only? | ☐ | ☐ | ☐ | ☐ | |
| Are the air-lock flaps properly constructed? | ☐ | ☐ | ☐ | ☐ | |
| Appropriate number of air locks? | | | | | |

## Comments*

Deficiencies and Corrections

| |
|---|
| |
| |
| |
| |

**ACC Field Technician Signature, DOSH Certification #**

# MANOMETER LOG (V2015.12.09)

A·C·C ENVIRONMENTAL CONSULTANTS

| Project Information | | Date: | |
|---|---|---|---|
| Project Name: | | | |
| Project Address: | | | |
| Client Name: | | ACC Project Number: | |
| ACC Field Technician:<br>DOSH Certification No. | | ACC Project Manager: | |
| Contractor Name: | | Supervisor Name: | |
| Containment Location: | | | |
| Type of Work<br>Check All that Apply | ☐ Asbestos Class I ☐ Asbestos Class II ☐ Asbestos Class III ☐ Asbestos Class IV ☐ Mold ☐ Other: | | |

| Date | Time | Pressure Reading | Manometer Zeroed? | Initials | Comments |
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ACC Field Technician Signature, DOSH Certification No:

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Northern California: 7977 Capwell Drive, Suite 100 • Oakland, CA 94621 • (510) 638-8400 • Fax (510) 638-8404
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# Appendix B

# Analytical Laboratory SOPs

Curtis & Tompkins

PCB Sonication Extraction_rv13

Curtis & Tompkins, Ltd.

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 | | 7/27/15 |
| Teresa Morrison<br>QA Director | Teresa K. Morrison | 7/27/15 |
| **Reapproved:** | | |
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Curtis & Tompkins, Ltd.

Sonication (EPA 3550) Extraction of Soil Samples for

# POLYCHLORINATED BIPHENYLS (PCB)

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Curtis & Tompkins, Ltd.

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
C&T Client Services SOP 2.3, Subsampling & Compositing (ASTM D6323-98)

*Cleanup Methods:*
EPA 3665A, Sulfuric Acid/Permanganate Cleanup, Rev 1, Dec. 1996
EPA 3640A, Gel-Permeation Cleanup, SW-846 Update 3, 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:*
C&T SOP SVOC 3.2, Polychlorinated Biphenyls by EPA 8082/8082A/608
C&T SOP QA 1.4, Balance Calibration Procedures
C&T SOP QA 1.6, Pipette and Auto-Dispensor Calibration Verification
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 XLAB 1.6, Calibrating Volumetric Glassware
C&T SOP XLAB 1.11, Screening of Solvents & Reagents
C&T SOP XLAB 1.12, Sonicator Tuning
C&T SOP XLAB 1.3, Spill Control & Cleanup
TNI Standard, EL-V1-2009, September 2009
DoD Quality Systems Manual (QSM), Version 4.2, Oct 2010
DoD/DoE Consolidated QSM, Version 5.0, July 2013

## 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.

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>QSMv5.0: <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. |

| | | | |
|---|---|---|---|
| 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 annually or and 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 | Analyze on each | Spike at 1-4x MDL and | See MDL SOP |

| | | | |
|---|---|---|---|
| (LOD) verification | instrument, quarterly (DoD QSM requirement) | <LOQ; analytes 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 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. If any target compounds are detected in the method blank, the entire batch must be re-extracted.

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

Since for water samples there is usually insufficient sample volume to extract a Matrix Spike and Matrix Spike Duplicate, a pair of blank spikes are extracted and analyzed with every batch. This demonstrates that the extraction and analysis procedures are accurate and precise in the absence of matrix interferences. If the recovery or RPD of any of the spike compounds is outside the acceptance limits, the entire batch must be re-extracted.

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):
Matrix spikes are aliquots of a sample to which a known amount of the target compound is added prior to extraction. These extracts are then analyzed to verify the accuracy (%recovery) and reproducibility (%RPD) in the possible presence of matrix interferences.

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; any sample that demonstrates recovery failure must be re-extracted.

5.2.5 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.

5.2.6 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

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/Manuals link.

## 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.

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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 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. 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 C&T 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 C&T 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.

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 Mix each sample, including the MS and MSD, with enough (20 to 100 grams) granular anhydrous sodium sulfate to make the soil free flowing.

8.1.15 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.

8.1.16 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.

8.1.17 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 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.

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.

8.4.7 Concentrate the extract to approximately 15 mL then exchange solvents

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*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 <u>entire</u> 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:

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
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:

10.1 Sample jars or sleeves must be returned to the coldroom for later disposal by Sample Control.

10.2 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.

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.

10.3 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.

10.1 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.

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10.4 Sample extracts, including the vials containing sulfuric acid, should be stored at 4$^{o}$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.

10.5 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 12) was changed as follows:

- Section 5: Combined previous section 6 (definitions into Section 5. Added Sonicator tuning and annual DOC requirement. Added LOD & LOQ definitions
- Section 6: Added equipment section
- Section 10: Added waste accumulation & disposal details

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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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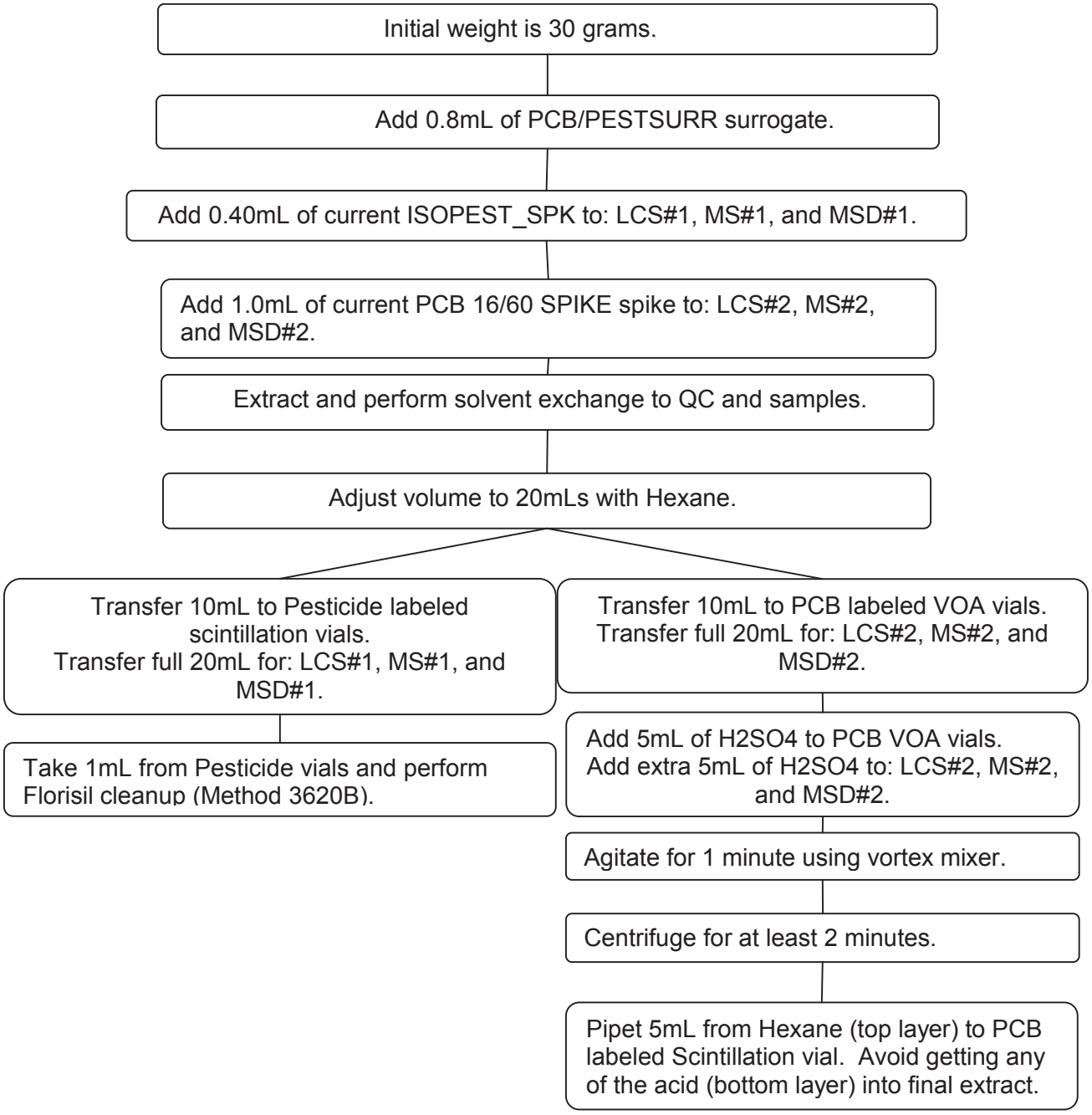
**Flowchart for 8081/8082 combined extraction by method 3550:**

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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 of Refrigerator # 5 in the Extraction Lab at -10 to -20°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.

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 |

**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, C&T 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.

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**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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PCB Liquid-Liquid Extraction_rv15

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Automated Liquid-Liquid Extraction (EPA 3520) of Water Samples for

# POLYCHLORINATED BIPHENYLS (PCBs)

For GC-ECD Methods EPA 608, EPA 8082 and EPA 8082A

| **Approved by:** | Signature: | Date: |
|---|---|---|
| Aaron Grenier<br>Group Leader | | 2/15/16 |
| Teresa Morrison<br>QA Director | Teresa K Morrison | 02/15/16 |
| **Reapproved:** | | |
| | | |
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Curtis & Tompkins, Ltd.

Automated Liquid-Liquid Extraction (EPA 3520) of Water Samples for

# POLYCHLORINATED BIPHENYLS (PCB's)

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Automated Liquid-Liquid Extraction of Water Samples for
**POLYCHLORINATED BIPHENYLS (PCBs)**

## 1.0 SCOPE

This document describes the extraction of Polychlorinated Biphenyls (PCBs) from water samples by continuous liquid-liquid extraction (EPA 3520) for later analysis by GC-ECD method EPA 8082, EPA 8082A, or EPA 608-(PCB fraction only). See Appendix 1 for a summary of this extraction procedure.

Using a 1 liter initial volume and 25mL final extract volume, the reporting limits for this procedure are 0.5 to 1.0 µg/L.

## 2.0 REFERENCES

*Extraction Methods:*
EPA 3500B, Organic Extraction and Sample Preparation, SW-846, Dec.1996
EPA 3500C, Organic Extraction and Sample Preparation, SW-846, Feb. 2007
EPA 3520C, Continuous Liquid-Liquid Extraction, SW-846, Dec.1996
Client Services SOP 2.4, Compositing Liquid Samples

*Cleanup Method:*
EPA 3665A, Sulfuric Acid Cleanup, SW-846, Dec. 1996

*Analysis Method:*
EPA 8082, PCB's by GC/ECD, SW-846, Dec.1996
EPA 8082A, Polychlorinated Biphenyls (PCB's), Feb 2007
EPA 608, Organochlorine Pesticides & PCBs, 40CFR136 Appendix A

*Related QA SOPs and Guidance Documents:*
C&T SOP XLAB 1.6, Calibrating Volumetric Glassware
C&T SOP QA 1.6, Pipette, Dispensor & Burette Calibration Verification
C&T SOP QA 4.1, Establishing Control Limits
C&T SOP QA 4.4, Determining Method Detection Limits (MDL) and Limit of Detection (LOD)
C&T SOP QA 4.6, Limit of Quantitation (LOQ)
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, July 2013

## 3.0 SAMPLE PRESERVATION & HOLDING TIME

Bottles: Samples should be submitted in a 1L amber glass bottle; plastic bottles are not appropriate for organic analyses.

Preservation: No chemical preservation required.
Store at >0.0 to 6°C

Holding Times: **Waste water / NPDES**
(**LIMS prod: 608-PCB**, Method EPA 608)
Holding Time: 1 year from sample collection until extraction
1 year from extraction until analysis
(40CFR136 MUR, Table II, 05/18/12)

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**Ground water & surface water**
(**LIMS prod: PCB,** SW-846 Methods EPA 8082 & 8082A)

California: 7 days from sample collection until extraction
40 days from extraction until analysis
(SW-846 Update III, Chapter 4, 1996)

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 sulfuric acid, a very corrosive substance that will cause 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 carcinogenic (dichloromethane) solvents. 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<br><br>Final volume must match that of the lowest final volume in the batch. | <1/2 RL (LOQ) | Report samples that are ND or >10x contamination; rerun all others |

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| | | | |
|---|---|---|---|
| Blank Spike (BS)/<br>Blank Spike Duplicate (BSD) | 1 pair for every batch of 20 or fewer samples<br><br>(if sufficient sample volume was submitted for an MS/MSD, a single blank spike may be extracted; label as an LCS in LIMS) | %Recovery & %RPD within acceptance 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)/<br>Matrix Spike Duplicate (MSD) | 1 pair for every batch of 20 or fewer samples (if sufficient sample volume was submitted) | %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 |
| Volumetric Glassware | Upon receipt (Class A are exempt from initial verification), then annually or and upon evidence of deterioration | Within tolerances listed in Glassware Calibration SOP | See Glassware Calibration SOP |
| Method Detection Limit (MDL) Study | When new instrument installed, or significant change made to process | <1/3 RL (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 <RL (LOQ); analytes detected | See MDL SOP |
| Limit of Quantitation (LOQ) | Analyze on one instrument, quarterly (DoD QSM requirement) | Spike at or above (up to 2x) the RL (or lowest ICAL standard). Recovery within 50-150% | See LOQ SOP |

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| | | | |
|---|---|---|---|
| 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 Batch QC Definitions

5.2.1 Method Blank (MB):
A method blank 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. If any target compounds are detected in the method blank, the entire batch must be re-extracted.

5.2.2 Blank Spike (BS) / Blank Spike Duplicate (BSD) – or Laboratory Control Sample (LCS):
Since there is usually insufficient sample volume to extract a Matrix Spike and Matrix Spike Duplicate, a pair of blank spikes are extracted and analyzed with every batch. This demonstrates that the extraction and analysis procedures are accurate and precise in the absence of matrix interferences. If the recovery or RPD of any of the spike compounds is outside the acceptance limits, the entire batch must be re-extracted.

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):
If sufficient sample volume was submitted, extract and analyze an MS/MSD, to demonstrate the effectiveness of the procedure in real world samples, which may be subject to matrix interferences. If the recovery or RPD of any spike compound is outside acceptance limits, the batch may need to be re-extracted.

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; any sample that demonstrates recovery failure must be re-extracted.

5.2.5 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.

5.2.6 Limit of Quantitation (LOQ) samples are aliquots of deionized water that are spiked at 1-2x the standard reporting limit (or lowest calibration standard) and used to verify the accuracy of the procedure at the reporting limit. The LOQ must be determined quarterly

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on a single instrument for DoD (or annually for NELAC). See the QA SOP 4.6 'Limit of Quantitation' for details.

## 6.0 INTERFERENCES & TROUBLE-SHOOTING

6.1 If samples contain a significant amount of sediment, the water should be decanted and the sediment left in the bottle, as sediment will clog the extractor.

6.2 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.

6.3 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 is effect has been observed when Acetonitrile was inadvertently used in place of hexane.

## 7.0 EQUIPMENT & MAINTENANCE

Automated Liquid/Liquid extractor bodies
Condensers
Narrow range pH paper
Boiling flasks, 500mL, flat-bottomed
Heating mantles
Recirculating chiller, temperatures set to 10-15°C
Funnels
K-D concentrators and receivers

Broken glassware should be given to the Group Leader for disposal or repair.

Maintenance & trouble-shooting notes for the recirculating chiller can be found in the Operators Manual in the Group Leader's office.

## 8.0 PROCEDURE

### 8.1 Automated Liquid-Liquid Extractor Set up

8.1.1 Rinse through the top of the condensers then around the entire area of the ground glass joint using Dichloromethane.

*Note:* Do not allow solvent to contact the Tygon tubing or contamination of the extract will result. Rinse the joint with solvent again if this happens.

8.1.2 Select enough boiling flasks for the entire batch. Examine each for cracks and chips, discarding any broken flasks.

8.1.3 Rinse each boiling flask with Dichloromethane, discarding the solvent into the waste solvent jar. Turn the boiling flask upside down and place on white wooden rack to drain.

8.1.4 After all flasks are rinsed and drained, set flasks on bench-top under fume hood and add 2 or 3 solvent rinsed boiling chips to each flask.

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8.1.5 Add Dichloromethane to each flask until the solvent level is between “Pyrex” and “500mL” (approximately 300mL).

8.1.6 Rinse the liquid-liquid extractor bodies with dichloromethane, discarding the solvent into the waste solvent jar.

8.1.7 Add approximately 150mL of dichloromethane to each extractor body then place in the liquid-liquid rack.

8.1.8 Attach each flask to an extractor body, setting the apparatus at an angle of about 15° away from you by adjusting the placement of the heating mantle.

8.1.9 Label each boiling flask with the sample ID and analysis. The samples should be put up in order of increasing sample#, beginning with the batch QC.

**8.2 Sample Preparation**

8.2.1 Record the sample number and *letter* for each sample in the benchbook, so that each sample container can be tracked.

8.2.2 When the sample is contained in a 1000mL narrow mouth ESS amber bottle, align it next to the calibrated 1000mL bottle used for measuring and record the sample volume.

If the sample arrives in any other container, mark the liquid level on the side of the sample bottle with a marking pen then see Step 8.2.10 for measuring the volume.

Homogenize each sample by inverting the sample container several times. If no headspace is present in the container, pour a small amount of the sample into the extractor body before homogenizing the sample. Retain the bottle caps for future homogenization steps.

*Note:* If the bottle contains an excessive amount of sediment at or exceeding a ½” thick sediment layer, decant the sample into a Dichloromethane-rinsed 1,200mL graduated cylinder. Mark the water level on the side of the graduated cylinder with a marker then treat the graduated cylinder as the sample container in the following steps. Note the excessive amount of sediment in the benchbook.

8.2.3 Using a borosilicate glass disposable pipette, measure the pH of each sample by placing one drop of sample onto narrow-range pH paper and record the pH in the appropriate benchbook.

*Note:* Never dip the pH strip directly into the sample as it could contaminate the sample.

8.2.4 For the Method Blank, Blank Spike, and Blank Spike Duplicate (or Method Blank and Laboratory Control Sample), add 1 L of deionized water to a Dichloromethane-rinsed 1,200mL graduated cylinder for each of these QC samples.

8.2.5 For the BS and BSD (or LCS, MS, and MSD), add 1.0mL PCB 16/60 SPIKE Solution to the deionized water in the QC Liquid/Liquid extractor bodies or to the MS/MSD sample bottles.

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Document the LIMS ID of the spike and the volume added in the benchbook. See Appendix 2 for instructions on preparing the Matrix Spiking Solution.

8.2.6 To every sample, including the batch QC samples, add 1.0mL of the PEST/PCBSURR Surrogate Solution to the deionized water in the QC graduated cylinders, or directly into the sample bottles. Document the LIMS ID of the surrogate and the volume added in the benchbook. See Appendix 2 for instructions on preparing the Surrogate Solution.

8.2.7 Replace the cap onto each bottle, and homogenize each sample by inverting the capped sample bottle several times. For any samples and QC contained in the graduated cylinders, homogenize by stirring the sample using a disposable pipette.

8.2.8 Pour each homogenized sample into the extractor body labeled with that sample number. Rinse the sample bottle or graduated cylinder with about 10 mL of Dichloromethane and add this solvent to the extractor body, unless a significant amount of sediment is present (see section on interferences).

8.2.9 If necessary, adjust the pH of the sample to between 5-9 by carefully adding either 10N sodium hydroxide (NaOH) or concentrated sulfuric acid ($H_2SO_4$). Stir the sample with a disposable pipette before re-measuring the pH. Record in the benchbook the manufacturer and lot# of the reagent used.

8.2.10 If the sample container required the mark described in Step 8.2.2, measure the sample volume by filling each sample bottle to the mark with water. Pour the water into a Class-1 graduated cylinder, then read the volume to the nearest 10 mL and record it in the appropriate lab notebook.

8.2.11 If any sample volume is less than 900 mL, add sufficient Nanopure water to that extractor body to make the sample volume 1,000 mL.

8.2.12 Adjust the angle of each extractor body until the dichloromethane in the bottom fills the transfer arm and just begins to spill into the boiling flask. *Note:* It may be necessary to add a small volume of Nanopure water if slight adjustments of the angle do not correctly fill the transfer arm.

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**8.3 Extraction**

8.3.1 Attach the condensers to the extractor bodies.

8.3.2 Verify that the recirculator temperature is set $\leq$ 15°C but above 10°C. If the temperature is set too low, the recirculator will freeze. If set too high, solvent will escape out the top of the condensor and the boiling flask will go dry.

8.3.3 Verify that the recirculator pressure is between 15 - 30 psi.

8.3.4 While the pump is on, open the top hatch of the recirculator. Open the white cap to the water reservoir and make sure the reservoir is full. If not, fill with 1:1 DI water:ethylene glycol only.

8.3.5 Turn on the heating mantles. The controllers should be set to 2.5 on the dial.

8.3.6 After 15 to 20 minutes, check to see that the system is cycling properly and that the condensers are cold to the touch. The extractor is operating properly when 3-4 drops per second are dropping into the sample from the bottom of the condenser.

8.3.7 Check the solvent level in the round bottom flask; it should not drop below the top edge of heating mantle. Adjust the solvent level in the flask by adjusting the tilt of the apparatus from the vertical by changing the placement of the heating mantles. To increase the amount of solvent in the flask, carefully tilt the apparatus more to the vertical.

8.3.8 Extract the samples for 18-24 hours. Document the start and stop times in the appropriate laboratory notebook.

8.3.9 Turn off the heating mantles and allow the system to cool to room temperature.

8.3.10 Remove the condensors, then the round bottom flasks.

**8.4 Filter Funnels & K-D Concentrator Assembly**

8.4.1 Using solvent-rinsed, baked glass wool, place a glass wool plug in the bottom of each funnel.

8.4.2 Rinse the walls of the funnel and the wool with dichloromethane, collecting the rinsate in a waste jar.

8.4.3 Add approximately 40g of baked granular or powder sodium sulfate to each funnel(powder is preferred for soil).

8.4.4 Place the funnels in the bottom rack and rinse the funnel with dichloromethane, again collecting the rinsate in the waste jar.

8.4.5 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.

8.4.6 Rinse each receiver with three aliquots of dichloromethane, discarding the rinsate into a waste-solvent jar. Immediately connect each receiver to a K-D.

8.4.7 After receivers have been added to each K-D, use plastic clips to secure the two pieces together.

8.4.8 Label each K-D and receiver with the sample ID and analysis, then replace in the top rack right-side up.

8.4.9 After all concentrators have been assembled and labeled, add a solvent-rinsed boiling chip to each.

8.4.10 Discard the waste dichloromethane in the jars into the waste solvent jug.

8.4.11 Reverse the filters and concentrators so that the filters are on the top rack and the concentrators are on the bottom rack.

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8.4.12 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.

8.5 **Filtration**

8.5.1 Place the flasks in order in front of the appropriate K-D apparatus. Verify that the receivers are securely attached to the K-D concentrators.

*Note:* If water is noticed in the round bottom flask dry the extract by adding about 15 g (about ½ scintillation vial) of solvent rinsed granular anhydrous sodium sulfate to the boiling flask. Agitate the flask; if the sodium sulfate clumps up, add more sodium sulfate until there is free flowing granular sodium sulfate in the flask.

8.5.2 Pour the extract through the funnel into the K-D concentrator.

8.5.3 Rinse each boiling flask with three 10 mL aliquots of Dichloromethane, adding these rinsates to the appropriate funnel.

8.5.4 After the third rinse, turn the boiling flask so the label faces the left (at 9 o'clock) to indicate that rinsing has been completed.

8.5.6 Allow the funnel to drain completely then use dichloromethane to rinse around the top of the funnel (~25mL), washing any remaining extract through the funnel into the concentrator and allow to drain completely.

8.5.7 While the filters are draining, rinse each Snyder column 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.

8.6.8 After the funnels have completely drained, remove the funnels from the rack and dump the sodium sulfate into the drying tray under the hood.

**8.6 CONCENTRATION**

8.6.1 Add water to any hot water bath that is not completely full.

8.6.2 Verify that the dial setting is at '10' and that the water bath is boiling.

8.6.3 Verify that each K-D concentrator contains 1-2 boiling chips. Attach a rinsed 3 ball Snyder column to each K-D concentrator.

8.6.4 Place each K-D apparatus 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.

8.6.5 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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8.6.6 Concentrate the extract to 5 - 10 mL then exchange solvents.

*Note:* Reducing the volume to only 15 mL or so prior to solvent exchange may result in incomplete elimination of the acetone and cause instrument problems or low surrogate recoveries.

8.7 **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.

8.7.1 After the extract volume has been reduced to 5 - 10mL, but while the extract is still boiling, add 10 - 15 mL of Hexane through the top of the Snyder column.

8.7.2 Concentrate to about 5-10 mL and again add 10 - 15 mL Hexane.

8.7.3 Concentrate to between 5 - 10 mL.

8.7.4 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.

8.7.5 Let the extract cool to room temperature.

8.7.6 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.

8.7.7 Remove the Snyder column, remove the plastic clip and dry the joint with a ChemWipe.

8.7.8 Carefully loosen the K-D/ receiver joint, remove the K-D concentrator and place the receiver in the metal rack in numeric order.

8.7.9 Adjust the volume to 25 mL with Hexane.

8.7.10 Record the final volume of extract in the benchbook.

8.7.11 Bring the metal rack over to the vortex mixer and, beginning with the batch QC, use the vortex mixer to homogenize the extracts.

8.7.12 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.

**8.8 CLEAN UP PROCEDURE**

8.8.1 Add 10 mL of concentrated Sulfuric Acid and agitate for 60 seconds with a Vortex mixer.

8.8.2 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.

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8.8.3 Select enough pre-cleaned scintillation vials for the entire batch. Label them (cap and vial) with the sample numbers, batch number, and “PCB”.

8.8.4 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. Make sure to *not* transfer any of the acid layer into the scintillation vial.

8.8.5 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.

8.8.6 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.

8.8.7 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.

8.8.8 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.

8.8.9 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:

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, filters, and pH paper
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.

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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

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 (Flammable Solvent, Toxic Solvent, etc. ), and the date that the first contents are added to the container

After the extraction steps are completed, the spent sample and any waste solvent must be properly discarded by:

10.1 Pour the contents of the extractor body into a separatory funnel and let it settle. Drain the Dichloromethane (bottom layer) and discard in the Dichloromethane waste container. Make sure to pour out all of the dichloromethane, as excess dichloromethane in the waste water disposal drum is unacceptable and will result in increased waste disposal costs and liability.

10.2 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.

10.3 Neutralize the aqueous layer sodium bicarbonate and discard by pouring into the plastic drum, labeled “Toxic Waste”, of dichloromethane-contaminated aqueous waste. This aqueous waste may be accumulated in the extraction lab for up to 9 months, after which it must be transferred to the 275-gallon Aqueous Waste tote in the 90-day accumulation area of the garage.

10.4 Waste Hexane 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.

10.5 Sample extracts (including the hexane/ sulfuric acid filled vials) should be stored at 4$^{o}$C for at least 40 days, after which they may be transferred intact to the “PCB+Pesticides” 55 gallon open-head poly drum, located in the waste room.

10.6 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 production cost.

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## 12.0 REVISION HISTORY

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

- Section 3: Updated holding time information
- Section 5: Added LOD & LOQ definitions. Added annual DOC
- Section 9: Added requirement to document the lot# of the pH paper
- Section 10: Expanded waste accumulation and disposal details
- Appendix_2: Added note regarding miscibility of spike and surrogate standards in water

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**APPENDIX_1:** **PCB WATER EXTRACTION SUMMARY**

| | | |
|---|---|---|
| Sample Volume: | 1,000 mL | |
| Surrogate Solution: | PEST/PCBSURR<br>TCMX & DCB @ 1 µg/mL | Add: 1.0 mL |
| PCB Spike Solution: | PCB 16/60 SPIKE<br>AR1016 & AR1260 @ 5 µg /mL | Add: 1.0 mL |
| Extraction pH: | 5-9 | |
| Extraction Solvent: | Dichloromethane | |
| Final Solvent: | Hexane | |
| Water Bath Temp: | Boiling | |
| Final Volume: | 25 mL | |
| Sample Cleanup: | Sulfuric Acid (required) | |

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## APPENDIX_2: 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#.

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. Label the standard bottle with the contents, the LIMS S#, the expiration date of the standard, and the prep chemist's initials.

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.

Use Class-A volumetric flasks and the volumes and standards listed below. Store in the freezer of Refrigerator # 5, at -10 to -20°C, in the Extraction Lab.

**Procedure:**

1. Prepare all working standards in a **water-soluble solvent** (MeOH or MeOH:Dicloromethane mix) using Class-A volumetric flasks, and the volumes and standards listed below.

   ***Important!*** Each solution (spike & surrogate) added to batch QC must be fully miscible (soluble) in water and should be observed to fully disperse when added to water. *Do not* use straight Dichloromethane, as this will cause the spike/surrogate to drop directly through the water in the liquid-liquid extractor into the solvent layer and will not be a true measure of the effectiveness of the extraction.

2. Bring the source standards to room temperature before using them to make the working standards.

3. Place each standard's ampule 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.

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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.

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#.

Label each container with the LIMS S# and expiration date. 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 of Analysis into LIMS, then file it in the appropriate folder.

Store the source standards in the freezer section of Refrigerator # 5, at -10 to -20°C. Stock standards must be replaced after any manufacturers' expiration date or two years, or sooner if comparison with check standards indicates a problem.

| 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:* 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.

Dichloromethane, EM Science, Omni-Solv grade, VWR Cat# TXDX0837-39CUT
Store at room temperature for up to 1 year

Hexane, EM Science, HR-GC grade, VWR Catalog# EM-HX0297-1
Alternative Hexane, J.T. Baker, 95% n-Hexane, 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
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.

Sodium Hydroxide (NaOH), 50%, J.T. Baker, VWR Catalog# JT3727-3
Store at room temperature for up to 1 year

Sulfuric Acid, Concentrated, 90.5-92.7% by volume,
Fisher Scientific, Catalog# SA174212
Store in a Corrosives cabinet for up to 5 years

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## APPENDIX_3: 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 1µL, 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 µL of the source standard, dilute 1.0mL of the source standard into 9mL of the solvent, then use 3.0 µL 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 (µg/L)
Cws = Concentration (µg/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 (µg/Kg)
Cws = Concentration (µg/mL) of the Working Standard
Pdf = Final Volume of Extract (mL) / Initial Mass of Sample (g)

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PCBs by GC-ECD 8082 + 608 + TO-10_rv11

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:** | | |
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# 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

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

*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

## 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 |

| 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. |

| 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 |

| 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. |

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

EZChrom data acquisition and processing software

## 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.

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.

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.

**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.

## 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.

**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

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.

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).

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

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.

## 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

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

(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.

## 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.

- 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

## 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

### 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

$$\text{Standard Deviation} = SD = \sqrt{\{[\sum (RFi - RFavg)^2] / (n-1)\}}$$

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

$$\text{Slope (m)} = [(\sum wx_iy_i * \sum w) - (\sum wx_i * \sum wy_i)] / [(\sum w * \sum wx_i^2) - (\sum wx_i * \sum wx_i)]$$

$$\text{Intercept (b)} = y_{avg} - (m * x_{avg})$$

Correlation Coefficient (r)

$$r = \frac{[(\sum w * \sum wx_iy_i) - (\sum wx_i * \sum wy_i)]}{\sqrt{\{[(\sum w * \sum wx_i^2) - (\sum wx * \sum wx_i)] * [(\sum w * \sum wy_i^2) - (\sum wy_i * \sum wy_i)]\}}}$$

$$\text{Coefficient of Determination } (r^2) = r * r$$

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:

%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

## 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

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.

**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.

| 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.

**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 |

**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 |

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

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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 | | | | |

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

**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)

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. <u>Warning – baking out a detector will change its response;</u> 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

## 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.

## 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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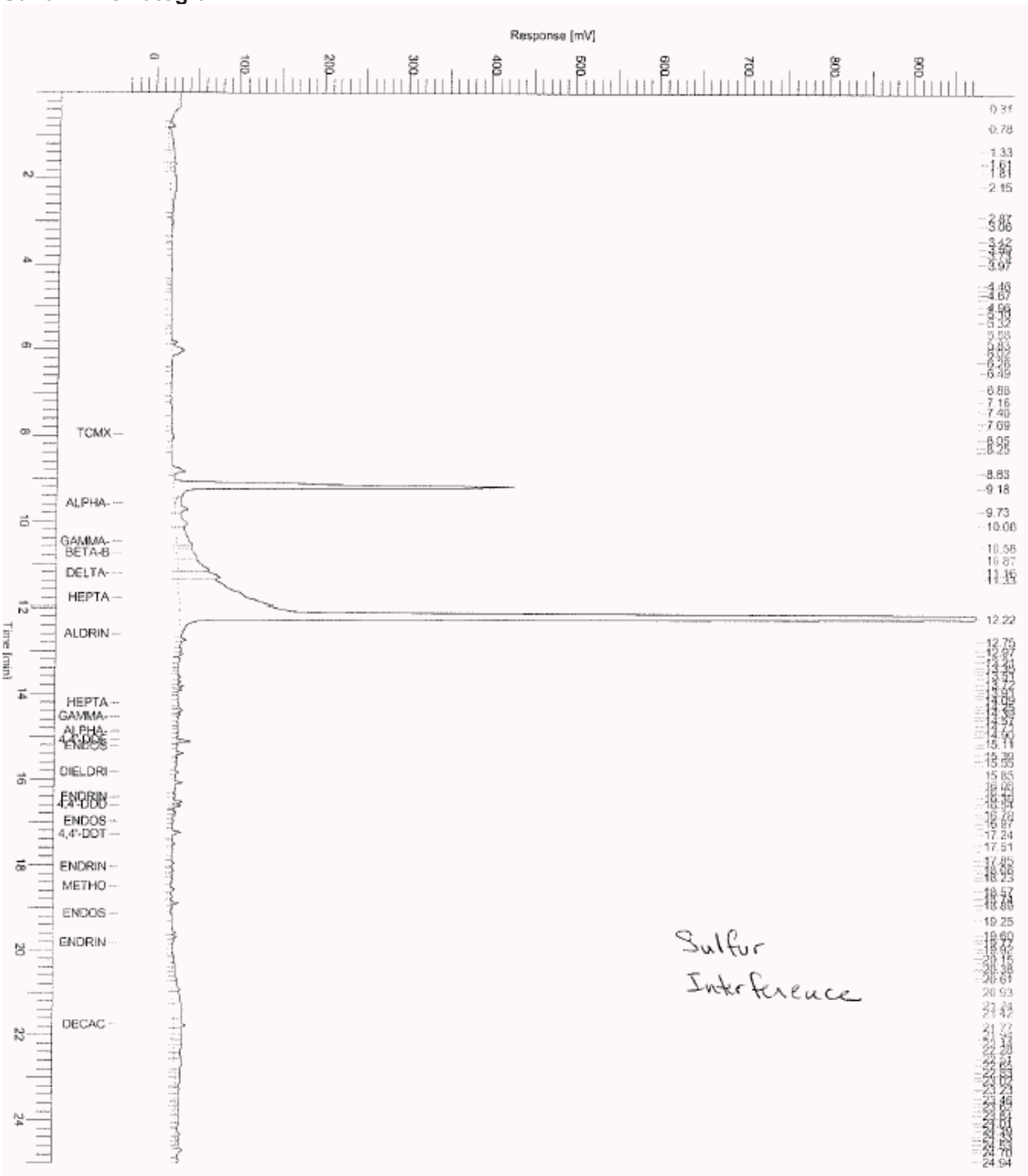
**Sulfur Chromatogram**



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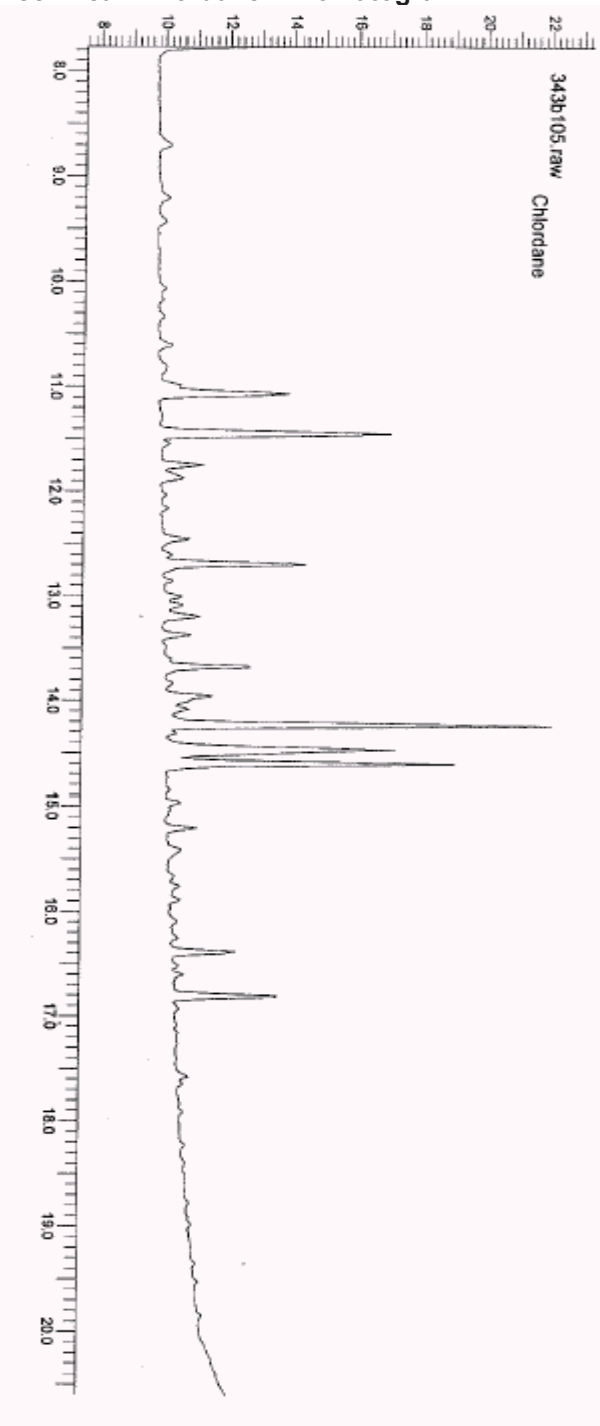
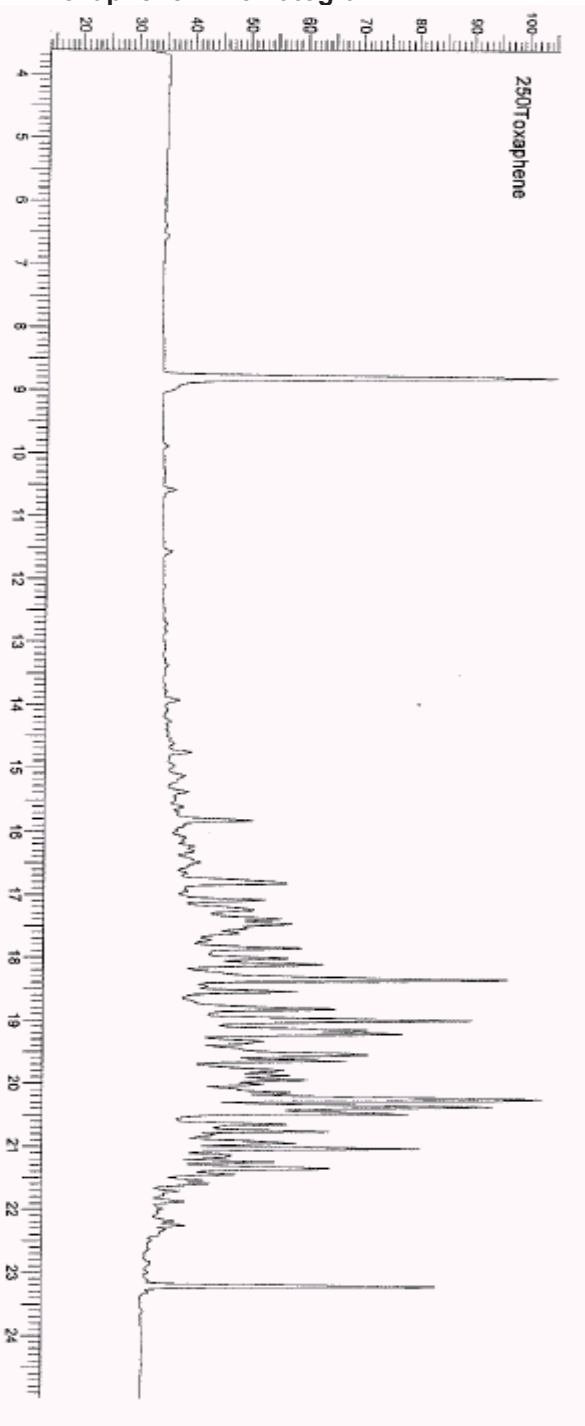
**Technical Chlordane Chromatogram**




**Toxaphene Chromatogram**



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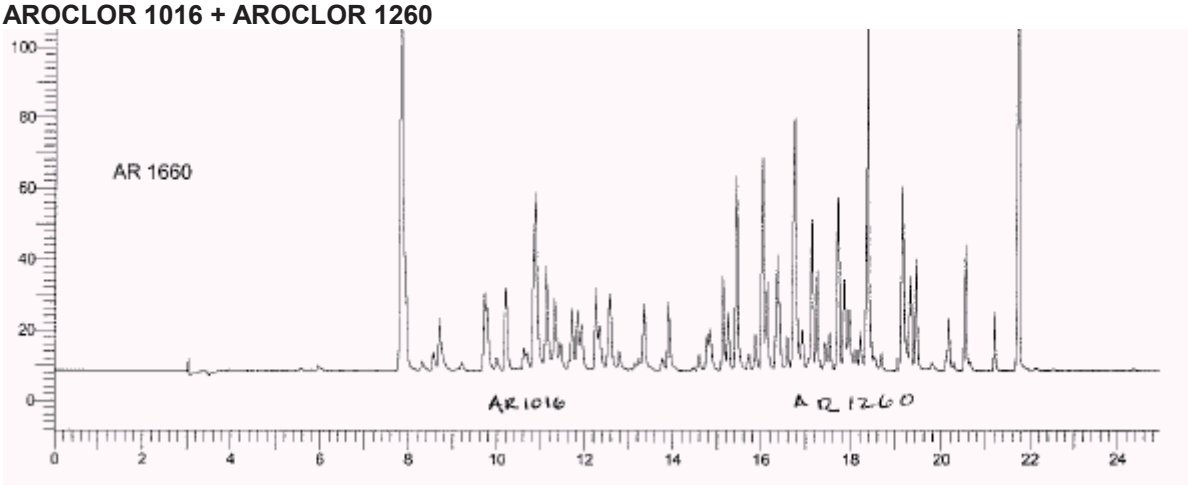
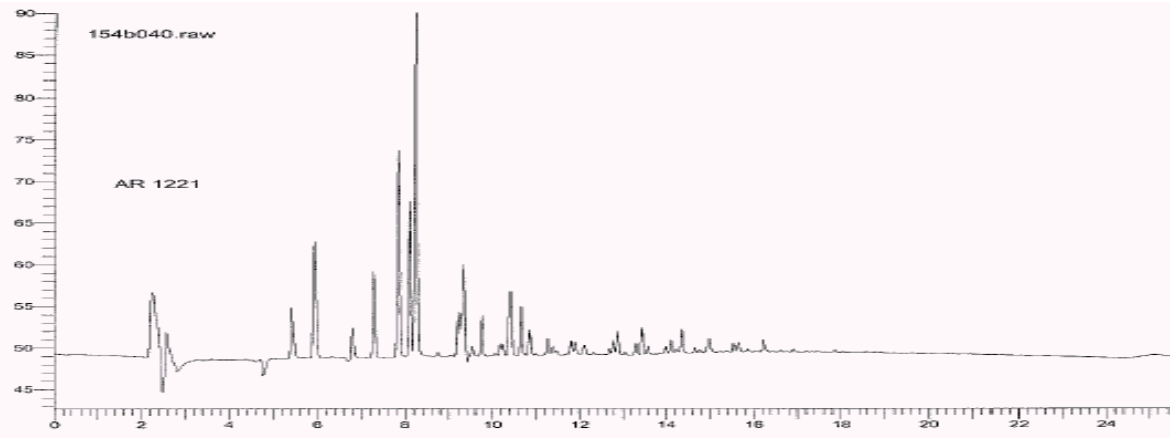
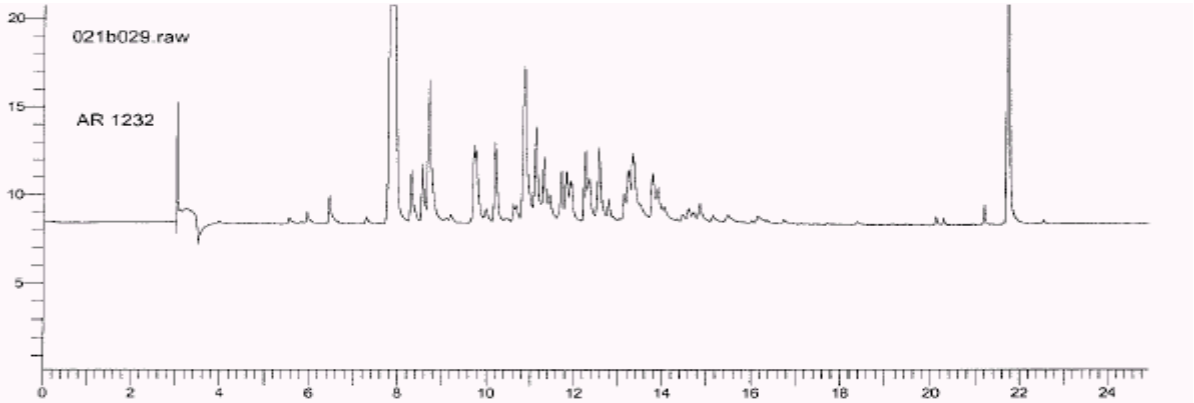
**AROCLOR 1016 + AROCLOR 1260**




**AROCLOR 1221**




**AROCLOR 1232**



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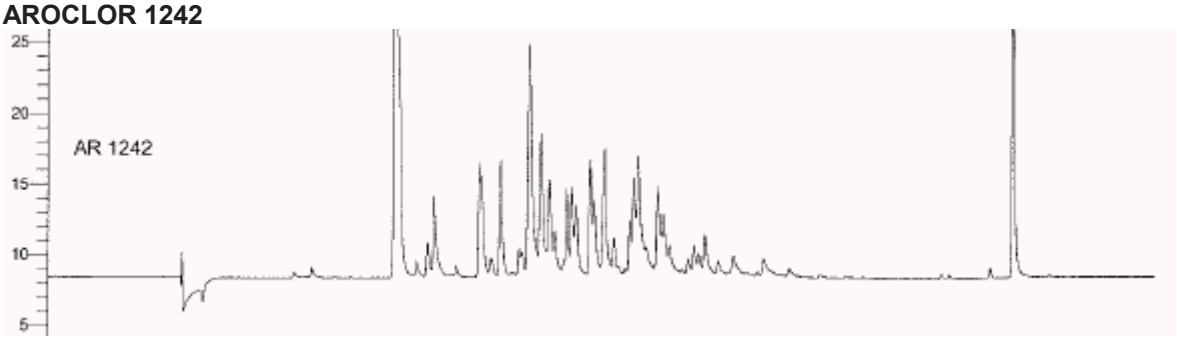
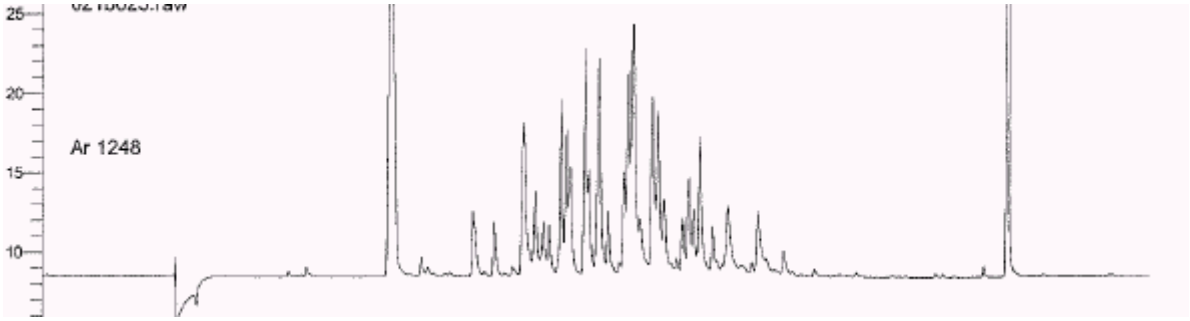
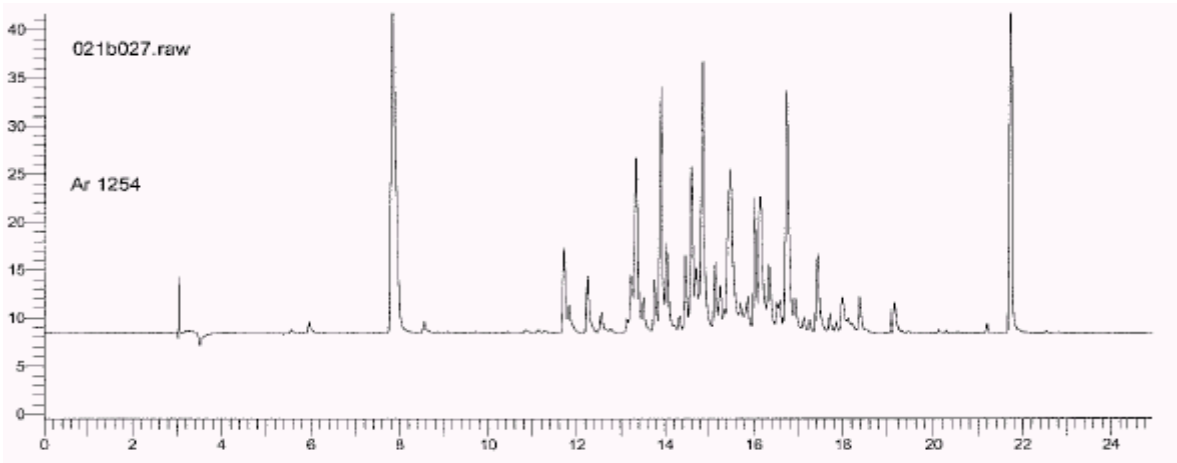
**AROCLOR 1242**




**AROCLOR 1248**




**AROCLOR 1254**



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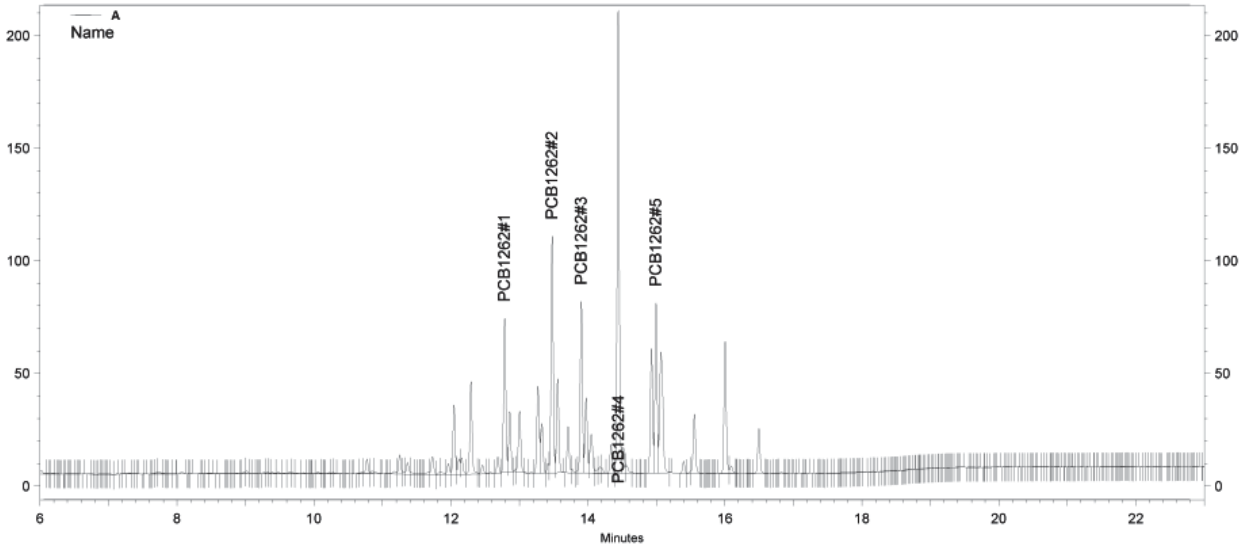
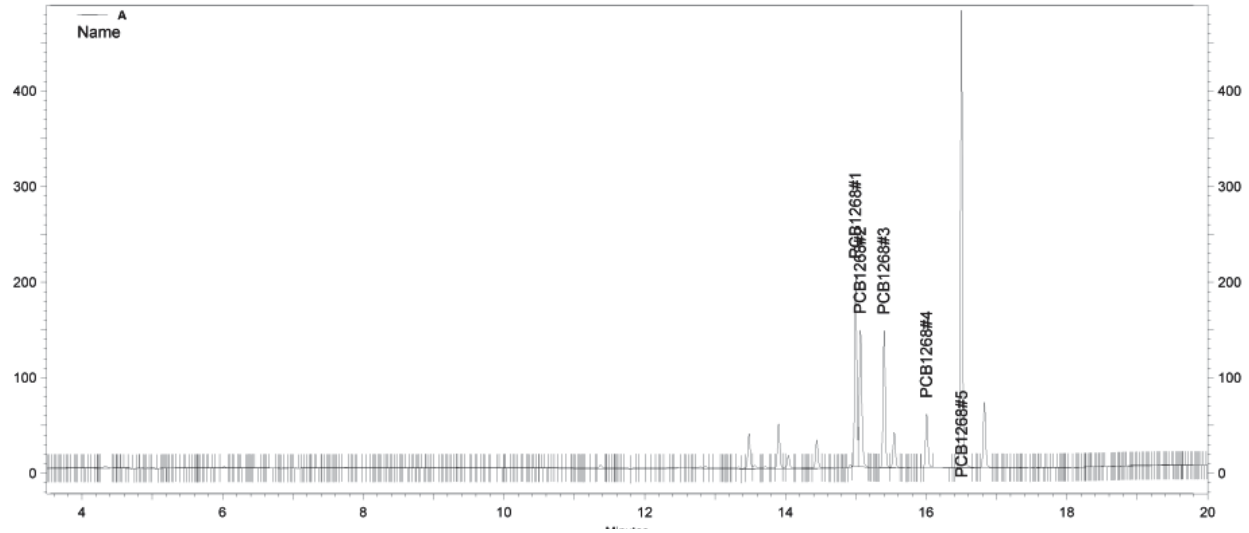
**AROCLOR 1262**




**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 $\leq$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.

**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.

## 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.

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.

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.

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.

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.

## 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:

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 <u>illegal</u> and is grounds for immediate termination.**

## 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.

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).

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.

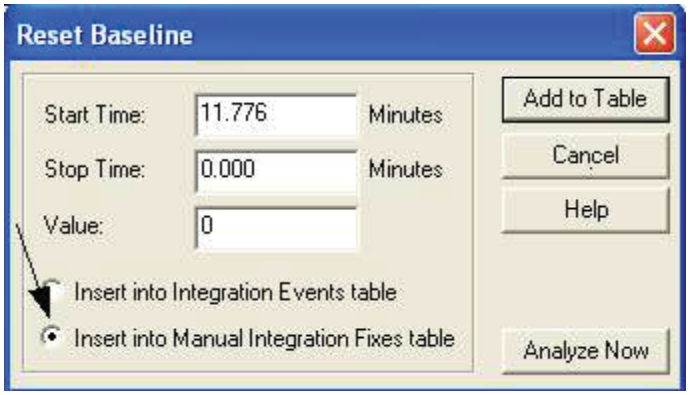
4. Open the data file by right clicking on the row of the run in the sequence. Choose OPEN DATA. The chromatograms for the two channels should appear.
5. Check the integrations for each target analyte.
6. If you need to do any integrations, the following are the common integration events:

| | |
|---|---|
| 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.*** |

| | |
|---|---|
| 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.* |

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.



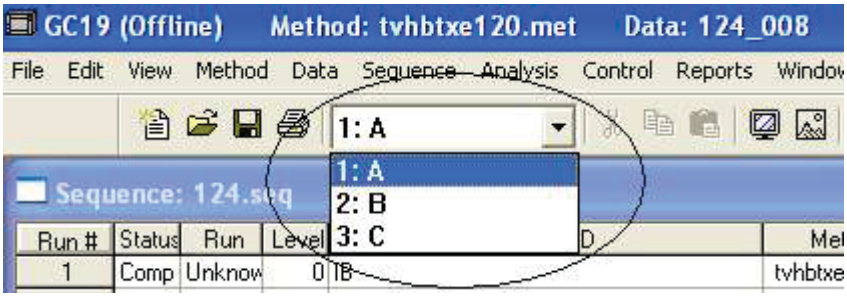

If you want to remove an integration event:

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.

*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.

## 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.

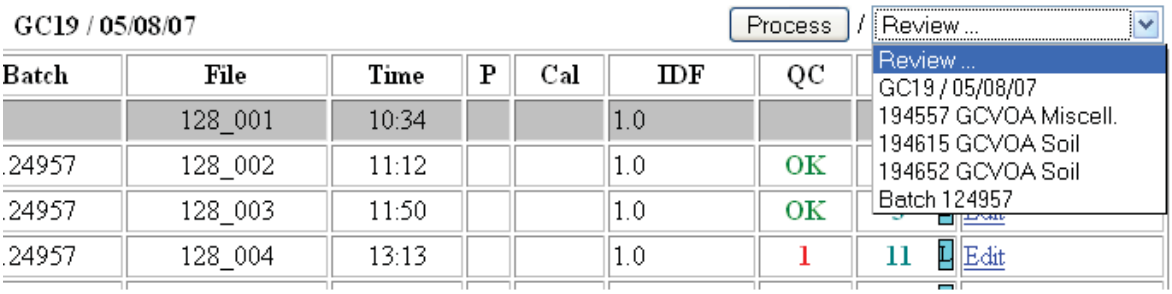
4. Check the sequence for any errors. LIMS takes the run information directly from the EZChrom sequence so any data entry error in the EZChrom sequence will affect the data processed by LIMS.

   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).



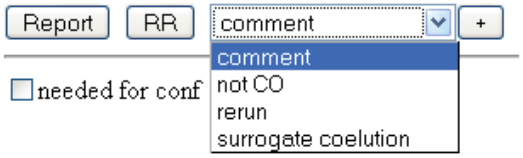

7. Two windows should open. The window on the left shows the main Review App frame (sequence, batch, ICAL) and the other shows the supporting documents (ie: EZChrom raw data reports, scanned prep logs). Clicking on an item in the left-hand frame will bring up the supporting documents for that item.

***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.

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".

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).

**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.

**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.

**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 $^{o}$C

**Initial Calibration**
A multi-point (minimum 5pt) calibration is required for Ar1016/1260, with a %RSD $\leq$ 20.

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.

3050 Soil Digestion for ICP-6010 and ICPMS-6020_rv15

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:** | | |
| | | |
| | | |
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Curtis & Tompkins, Ltd.

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

# EPA 3050B DIGESTION

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Curtis & Tompkins, Ltd.

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*

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

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 |

| 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) |

| 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 |

| 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 |

## 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

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.

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.

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 ($\pm$ 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.

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

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.

## 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.

**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.

**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 |

**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.

ICPMS Metals Analysis by 6020 + 200.8_rv10

Curtis & Tompkins, Ltd.

# 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 |
| | | |
| | | |
| | | |

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# METALS by ICP/MS

EPA 6020, EPA 6020A, EPA 200.8

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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

## 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.

## 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. |

| | | | |
|---|---|---|---|
| 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. |

| | | | |
|---|---|---|---|
| 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 |

| | | | |
|---|---|---|---|
| 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. |

| | | | |
|---|---|---|---|
| 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 |

| | | | |
|---|---|---|---|
| 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. |

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.

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

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.

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)

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.

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.

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**

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.

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

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.

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

- 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.

**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 |

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

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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 |

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

*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).

## 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 | | |

*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 |

## 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).

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).

**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.

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.

## 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".

**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:

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.

**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

## 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<br>(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 |

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

## 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

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.

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.

## 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.

**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

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

## 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:

$$\%RPD = |( (Wms/Wmsd)*Cms - Cmsd )| / (( (Wms/Wmsd) * Cms + Cmsd)/2) * 100$$

## 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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NIOSH 5503 PCB

mixture: $C_{12}H_{10-x}Cl_x$ [where x = 1 to 10] MW: ca. 258 (42% Cl ; $C_{12}H_7Cl_3$); ca. 326 (54% Cl ; $C_{12}H_5Cl_5$) CAS: Table 1 RTECS: Table 1

**METHOD: 5503, Issue 2** **EVALUATION: PARTIAL**

**Issue 1: 15 February 1984**
**Revision #1: 15 August 1987**
**Issue 2: 15 August 1994**

**OSHA :** 1 mg/m$^3$ (42% Cl); 0.5 mg/m$^3$ (54% Cl)
**NIOSH:** 0.001 mg/m$^3$/10 h (carcinogen)
**ACGIH:** 1 mg/m$^3$ (42% Cl) (skin); 0.5 mg/m$^3$ (54% Cl) (skin)

**PROPERTIES:** 42% Cl: BP 325 to 366 °C; MP -19 °C; d 1.38 g/mL @ 25 °C; VP 0.01 Pa (8 x $10^{-5}$ mm Hg; 1 mg/m$^3$) @ 20 °C
54% Cl: BP 365 to 390 °C; MP 10 °C; d 1.54 g/mL @ 25 °C; VP 0.0004 Pa (3 x $10^{-6}$ mm Hg; 0.05 mg/m$^3$) @ 20 °C

**SYNONYMS:** PCB; 1,1'-biphenyl chloro; chlorodiphenyl, 42% Cl (Aroclor 1242); and 54% Cl (Aroclor 1254)

## SAMPLING

**SAMPLER:** FILTER + SOLID SORBENT (13-mm glass fiber + Florisil, 100 mg/50 mg)

**FLOW RATE:** 0.05 to 0.2 L/min or less

**VOL-MIN:** 1 L @ 0.5 mg/m$^3$
**-MAX:** 50 L

**SHIPMENT:** transfer filters to glass vials after sampling

**SAMPLE STABILITY:** unknown for filters; 2 months for Florisil tubes [1]

**BLANKS:** 2 to 10 field blanks per set

## ACCURACY

**RANGE STUDIED:** not studied

**BIAS:** none identified

**OVERALL PRECISION ($\hat{S}_{rT}$):** not evaluated

**ACCURACY:** not determined

## MEASUREMENT

**TECHNIQUE:** GAS CHROMATOGRAPHY, ECD ($^{63}$Ni)

**ANALYTE:** polychlorobiphenyls

**DESORPTION:** filter + front section, 5 mL hexane; back section, 2 mL hexane

**INJECTION VOLUME:** 4-µL with 1-µL backflush

**TEMPERATURE-INJECTION:** 250 to 300 °C
**-DETECTOR:** 300 to 325 °C
**-COLUMN:** 180 °C

**CARRIER GAS:** $N_2$, 40 mL/min

**COLUMN:** glass, 1.8 m x 2-mm ID, 1.5% OV-17/1.95% QF-1 on 80/100 mesh Chromosorb WHP

**CALIBRATION:** standard PCB mixture in hexane

**RANGE:** 0.4 to 4 µg per sample [2]

**ESTIMATED LOD:** 0.03 µg per sample [2]

**PRECISION ($\bar{S}_r$):** 0.044 [1]

**APPLICABILITY:** The working range is 0.01 to 10 mg/m$^3$ for a 40-L air sample [1]. With modifications, surface wipe samples may be analyzed [3,4].

**INTERFERENCES:** Chlorinated pesticides, such as DDT and DDE, may interfere with quantification of PCB. Sulfur-containing compounds in petroleum products also interfere [5].

**OTHER METHODS:** This method revises methods S120 [6] and P&CAM 244 [1]. Methods S121 [7] and P&CAM 253 [8] for PCB have not been revised.

**REAGENTS:**

1. Hexane, pesticide quality.
2. Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
3. Nitrogen, purified.
4. Stock standard solution of the PCB in methanol or isooctane (commercially available).*

* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6-mm OD, 4-mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. (SKC 226-39, Supelco ORBO-60, or equivalent) Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
2. Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
3. Tweezers.
4. Vials, glass, 4- and 7-mL, with aluminum or PTFE-lined caps
5. Gas chromatograph, electron capture detection ($^{63}$Ni), integrator and column (page 5503-1).
6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
7. Syringe, 10-µL.

**SPECIAL PRECAUTIONS:** Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [9-11].

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.

   NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [4]. Under these conditions, the limit of detection was 0.02 µg/m$^3$.
4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

**SAMPLE PREPARATION:**

5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.
6. In a 4-mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane.
7. Allow to stand 20 min with occasional agitation.

## CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 500 ng/mL PCB.
   a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB per sample).
9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
   a. Remove and discard back sorbent section of a media blank Florisil tube.
   b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
   c. Cap the tube. Allow to stand overnight.
   d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. µg PCB recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

## MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
    NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [12].
    NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
12. Sum the areas for five or more selected peaks.

## CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front ($W_f$) and back ($W_b$) sorbent sections, and in the average media blank filter (B) and front ($B_f$) and back ($B_b$) sorbent sections.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b)}{V}, \text{ mg/m}^3.$$

## EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL isooctane [8,13,14]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [1]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 mg/m $^3$ Aroclor 1016 [1].

**REFERENCES:**


[1] NIOSH Manual of Analytical Methods, 2nd ed., V. 1, P&CAM 244, U.S. Department of health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

[2] User check, Southern Research Institute, NIOSH Sequence #4121-U (unpublished, January 25, 1984).

[3] Kominsky, J. Applied Ind. Hyg. 1 (4), R-6 (1986).

[4] NIOSH Health Hazard Evaluation Report, HETA 85-289-1738 (unpublished, 1986).

[5] Hofstader, R. A., C. A. Bache, and D. J. Lisk. Bull, Environ. Contam. Toxicol., 11, 136 (1974).

[6] NIOSH Manual of Analytical Methods, 2nd ed., V. 4, S120, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).

[7] Ibid, V. 2, S121, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

[8] Ibid, Vol. 1, P&CAM 253

[9] Criteria for a Recommended Standard . . . Occupational Exposure to Polychlorinated Biphenyls, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-225 (1977).

[10] Current Intelligence Bulletin 7, Polychlorinated Biphenyls (PCBs), U.S. Department of Health and Human Services, Publ. (NIOSH) 78-127 (1975).

[11] Occupational Diseases, A Guide to Their Recognition, revised ed., 255-256, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-181 (1978).

[12] Dunker, J. C. and M. T. J. Hillebrand. Characterization of PCB Components in Clophen Formulations by Capillary GC-MS and GC-ECD Techniques, Environ. Sci. Technol., 17 (8), 449-456 (1983).

[13] Backup Data Report for S120, prepared under NIOSH Contract 210-76-0123, available as "Ten NIOSH Analytical Methods, Set 2," Order No. Pb 271-464 from NTIS, Springfield, VA 22161.

[14] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

[15] Hutzinger, O., S. Safe, and V. Zitko. The Chemistry of PCBs, CRC Press, Inc., Cleveland, OH (1974).


**METHOD REVISED BY:**

James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

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**Table 1. General Information.**

| Compound | CAS | RTECS |
|---|---|---|
| Polychlorinated Biphenyls | 1336-36-3 | TQ1350000 |
| Chlorobiphenyl | 27323-18-8 | DV2063000 |
| Aroclor 1016 (41% Cl) | 12674-11-2 | TQ1351000 |
| Aroclor 1242 (42% Cl) | 53469-21-9 | TQ1356000 |
| Aroclor 1254 (54% Cl) | 11097-69-1 | TQ1360000 |

**Table 2. Composition of some Aroclors [15].**

| Major Components | Aroclor 1016 | Aroclor 1242 | Aroclor 1254 |
|---|---|---|---|
| Biphenyl | 0.1% | <0.1% | <0.1% |
| Monochlorobiphenyls | 1 | 1 | <0.1 |
| Dichlorobiphenyls | 20 | 16 | 0.5 |
| Trichlorobiphenyls | 57 | 49 | 1 |
| Tetrachlorobiphenyls | 21 | 25 | 21 |
| Pentachlorobiphenyls | 1 | 8 | 48 |
| Hexachlorobiphenyls | <0.1 | 1 | 23 |
| Heptachlorobiphenyls | none detected | <0.1 | 6 |
| Octachlorobiphenyls | none detected | none detected | none detected |

NIOSH 7105 Pb by GFAA

# LEAD by GFAAS 7105

Pb MW: 207.19 (Pb); 223.19 (PbO) CAS: 7439-92-1 (Pb); 1317-36-8 (PbO) RTECS: OF7525000 (Pb) OG1750000 (PbO)

**METHOD: 7105, Issue 2** **EVALUATION: PARTIAL** **Issue 1: 15 August 1990**
**Issue 2: 15 August 1994**

**OSHA :** 0.05 mg/m$^3$
**NIOSH:** <0.1 mg/m$^3$; blood Pb ≤60 µg/100 g
**ACGIH:** 0.05 mg/m$^3$

**PROPERTIES:** soft metal; d 11.3 g/cm$^3$; MP 327.5 °C
valences +2, +4 in salts

**SYNONYMS:** elemental lead and lead compounds except alkyl lead

## SAMPLING

**SAMPLER:** FILTER
(0.8-µm cellulose ester membrane)

**FLOW RATE:** 1 to 4 L/min

**VOL-MIN:** 1 L @ 0.05 mg/m$^3$
**-MAX:** 1500 L

**SHIPMENT:** routine

**SAMPLE STABILITY:** stable

**FIELD BLANKS:** 2 to 10 field blanks per set

## ACCURACY

**RANGE STUDIED:** not studied

**BIAS:** not determined

**OVERALL PRECISION ($\hat{S}_{rT}$):** not determined

**ACCURACY:** not determined

## MEASUREMENT

**TECHNIQUE:** ATOMIC ABSORPTION SPECTROPHOTOMETER, GRAPHITE FURNACE

**ANALYTE:** lead

**ASHING:** conc. $HNO_3$, 3 mL; 30% $H_2O_2$; 1 mL; 140 °C

**FINAL SOLUTION:** 10 mL 5% $HNO_3$

**WAVELENGTH:** 283.3 nm

**GRAPHITE TUBE:** pyrolytic coated

**INJETION:** 20 µL + 10 µL matrix modifier,
DRY: 110 °C, 70 sec;
CHAR: 800 °C, 30 sec;
ATOMIZE: 1800 °C, 5 sec.

**BACKGROUND CORRECTION:** $D_2$, $H_2$, or Zeeman

**CALIBRATION:** $Pb^{2+}$ in 5% $HNO_3$

**RANGE** 0.05 to 100 µg per sample [1]

**ESTIMATED LOD:** 0.02 µg per sample [1]

**PRECISION ($\bar{S}_r$):** 0.049 [1]

**APPLICABILITY:** The working range is 0.002 to >1 mg/m$^3$ for a 200-L air sample. If high concentrations are expected, the samples should be analyzed by flame AAS. The method is applicable to elemental lead, including Pb fume, and all other aerosols containing lead. This is an elemental analysis, not compound specific. Aliquots of the sample may be analyzed separately for additional elements.

**INTERFERENCES:** Use $D_2$ or $H_2$ continuum or Zeeman background correction to control molecular absorption. High concentrations of calcium, sulfate, carbonate, sulfate, carbonate, phosphate, iodide, fluoride, or acetate can be offset by an additional sample treatment step.

**OTHER METHODS:** This revises and replaces P&CAM 214 (2). Method 7300 (ICP-AES) is an alternate analytical method. Method 7505 is specific for lead sulfide by X-ray diffraction. Method 7082 is a flame AAS method, with a higher working range.

**REAGENTS:**

1. Nitric acid, conc.*
2. Nitric acid, 5% (v/v). Add 50 mL conc. $HNO_3$ to 500 mL water; dilute to 1 L.
3. Hydrogen peroxide, 30% $H_2O$ (w/w), reagent grade.*
4. Calibration stock solution, 1000 µg/mL Pb. Commercial standard or dissolve 1.00 g Pb metal in minimum volume of $HNO_3$ and dilute to 1 L with 1% (v/v) $HNO_3$. Store in a polyethylene bottle.
5. Matrix Modifier. Place 0.2 g $NH_4H_2PO_4$ and 0.3 g $Mg(NO_3)_2$ in a 100-mL volumetric flask. Add 2 mL conc. $HNO_3$ and bring to volume with distilled or deionized water.
6. Argon, prepurified.
7. Distilled or deionized water.

* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: Cellulose ester membrane filter, 0.8-µm, 37-mm, in 2-piece cassette.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with graphite furnace atomizer and background correction.
4. Lead hollow cathode lamp or electrode dischargeless lamp.
5. Regulators, two-stage, for Argon.
6. Beakers, Phillips, 125-mL, or Griffin, 50-mL with watchglass covers.**
7. Volumetric flasks, 10- and 100-mL.**
8. Assorted volumetric pipets as needed.**
9. Hotplate, surface temperature 140°C.
10. Bottles, polyethylene, 100-mL.

** Clean all glassware with conc. nitric acid and rinse thoroughly with distilled or deionized water before use.

**SPECIAL PRECAUTIONS:** Concentrated nitric acid is an irritant and may burn skin. Perform all acid digestions in a fume hood. Hydrogen peroxide is a strong oxidizing agent, a strong irritant, and corrosive to the skin. Wear gloves and eye protection.

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for up to 8 h for a total sample size of 1 to 1500 L for TWA measurements. Do not exceed a filter loading of ca. 2 mg total dust.

**SAMPLE PREPARATION:**

NOTE: Some matrices, especially bulk samples containing epoxy-based paint, may require a different digestion procedure for complete recovery of lead. See the Appendix of Method 7082 (Lead by Flame AAS) for a microwave digestion procedure which can be used for this purpose.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 3 mL conc. $HNO_3$, and 1 mL 30% $H_2O_2$ and cover with a watchglass. Start reagent blanks at this step.
5. Heat on 140°C hotplate until volume is reduced to about 0.5 mL.
6. Rinse the watchglass and walls of the beaker with 3 to 5 mL 5% $HNO_3$. Allow the solution to evaporate to 0.5 mL.
7. Cool each beaker.
8. Transfer the solution quantitatively to a 10-mL volumetric flask and dilute to volume with distilled water.

**CALIBRATION AND QUALITY CONTROL:**

9. Prepare a series of six working standards covering the range 0.002 to 0.1 µg/mL Pb (0.02 to 1.0 µg Pb per sample).
   a. Add aliquots of calibration stock solution to 100-mL volumetric flasks. Dilute to volume with 5% $HNO_3$. Store the working standards in polyethylene bottles and prepare fresh weekly.
   b. Analyze the working standards together with the blanks and samples (steps 12 through 14).
   c. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
10. Analyze a standard for every 10 samples to check for instrument drift.

11. Check recoveries with at least one spiked media blank per 10 samples.
    NOTE: Perform a matrix spike of a sample occasionally to check for matrix interferences. If an adequate recovery is not obtained (85 to 115%), an alternate method of analysis should be used, such as flame AAS or ICP.

**MEASUREMENT:**

12. Set spectrophotometer as specified by the manufacturer and to conditions on page 7105-1.
    NOTE: An alternate wavelength is 217.0 nm [3]. Analyses at 217.0 nm have slightly greater sensitivity, but poorer signal-to-noise ratio compared to 283.3 nm. Also, non-atomic absorption is significantly greater at 217.0 nm, making the use of $D_2$ or $H_2$ continuum, or Zeeman background correction mandatory at that wavelength.
13. Add matrix modifier to samples and standards in proper ratio of 2 to 1 (sample or standard to matrix modifier).
14. Analyze standards, samples, and blanks. Record absorbance readings.
    NOTE: If the absorbance value for the sample is above the linear range of the standards, dilute with 5% $HNO_3$, reanalyze, and apply the appropriate dilution factor in the calculations.

**CALCULATIONS:**

15. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of lead in the sample, $C_s$, and average media blank, $C_b$, from the calibration graph.
16. Using the solution volumes (mL) of the sample, $V_s$, and media blanks, $V_b$, calculate the concentration, C (mg/m$^3$), of lead in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

**EVALUATION OF METHOD:**

Method P&CAM 214 [2], issued on 1/29/76, was based on a method for metal pollutants in water [4]. Air sampling and digestion procedures follow those in Method 7082. The analytical procedure was evaluated by DataChem Laboratories in 1990 [1]. The LOD was determined at 0.02 µg per sample, with a LOQ of 0.05 µg per sample. The precision of the measurement procedure was 0.049. The overall precision, bias, and accuracy of the method were not determined. The reagents for digesting various lead species are given below:

| Species | Digestion Method |
|---|---|
| Pb metal | $HNO_3$ only |
| Pb metal | $HNO_3 + H_2O_2$ |
| Pb | $HNO_3$ only |
| PbS | $HNO_3$ only |
| $PbO_2$ | $HNO_3$ only |
| $PbO_2$ | $HNO_3 + H_2O_2$ |
| Pb in paint* | $HNO_3$ only |
| Pb in paint* | $HNO_3 + H_2O_2$ |

* Standard Reference Material #1579, U.S. National Institute of Standards and Technology.

**REFERENCES:**

[1] Backup Data Report for Method 7105 submitted to NIOSH by DataChem Laboratories, NIOSH (Unpublished, September, 1990).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 214, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
[3] Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer Corporation (1976).
[4] Fernandez, F.J. and D.C. Manning. Atomic Absorbtion Analyses of Metal Pollutants in Water Using a Heated Graphite Atomizer Atomic Absorption Newsletter 10, 65 (1971).

**METHOD REVISED BY:**

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NIOSH 7303 Metals (Cu & Pb)

# ELEMENTS by ICP
# (Hot Block/HCl/$HNO_3$ Digestion)

**7303**

MW: Table 1 CAS: Table 2 RTECS: Table 2

**METHOD: 7303, Issue 1** **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

**OSHA:** Table 2
**NIOSH:** Table 2
**ACGIH:** Table 2

**PROPERTIES:** Table 1

**ELEMENTS:**

| | | | | | |
|---|---|---|---|---|---|
| aluminum | cadmium | indium | nickel | strontium | zinc |
| antimony* | calcium | iron | palladium | tellurium | |
| arsenic | chromium | lead* | phosphorus | thallium | |
| barium | cobalt | magnesium | platinum | tin* | |
| beryllium | copper | manganese | potassium | titanium | |
| bismuth* | gallium | molybdenum | selenium | vanadium | |
| boron | gold | neodymium | sodium | yttrium | |

* With certain restrictions (see Table 3)

## SAMPLING

**SAMPLER:** FILTER (0.8-µm, cellulose ester membrane)

**FLOW RATE:** 1 to 4 L/min

**VOL-MIN:** Table 1
-MAX: Table 1

**SHIPMENT:** Routine

**SAMPLE STABILITY:** Stable

**BLANKS:** 2 to 10 field blanks per set

## ACCURACY

**RANGE STUDIED:** 5,000 to 50,000 µg/sample

**BIAS:** Not determined

**OVERALL PRECISION:** Not determined

**ACCURACY:** Not determined

## MEASUREMENT

**TECHNIQUE:** INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY

**ANALYTE:** See element list above

**REAGENTS:** Conc. HCl, 1.25 mL; and conc. $HNO_3$, 1.25 mL

**FINAL SOLUTION:** 5% HCl and 5% $HNO_3$, 25 mL

**WAVELENGTH:** Element and instrument specific

**BACKGROUND CORRECTION:** Spectral wavelength shift

**CALIBRATION:** Elements in 5% HCl, 5% $HNO_3$

**RANGE:** LOQ to 50,000 µg/sample [1]

**ESTIMATED LOD:** Varies with element; Table 1

**PRECISION (Š):** Not evaluated

**APPLICABILITY**: The working range of this method is up to 100 mg/m$^3$ for each element in a 500-L sample (the minimum range depends on the LOD for each sample; see Table 1). The analysis is not compound specific. Certain elemental compounds are known to be acceptable or unacceptable by this method (see Table 3). For unverified compounds, a test run should be conducted using a known amount of the compound in question to determine acceptability.

**INTERFERENCES:** Interferences are spectral in nature and are accounted for by choosing appropriate wavelengths, applying interelement correction factors, and background correction.

**OTHER METHODS:** Alternative, more sensitive methods exist for some elements by graphite furnace atomic absorption spectroscopy. This method is similar to NIOSH Method 7301, differing only in the use of the hot block for digestion of the sampler.

**REAGENTS:**

1. Hydrochloric acid,* conc., ultra pure.
2. Nitric acid,* conc., ultra pure.
3. Calibration stock solutions, 50-1000 µg/mL. Commercially available single element solutions or multielement solutions prepared as instructed by the instrument manufacturer.
4. Argon, prepurified.
5. Distilled, deionized, Type II water.
6. Diluting solution: 5% HCl : 5% $HNO_3$. To about 600 mL of deionized water in a 1-L volumetric flask, slowly add 50 mL conc. HCl and 50 mL conc. HNO3. Dilute to the mark with deionized water.

* See SPECIAL PRECAUTIONS

**EQUIPMENT:**

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled argon plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Hot block apparatus at 95 °C.
5. Digestion vessels and caps, 50-mL.
6. Watchglasses.
7. Pipettes, electronic and mechanical.
8. Regulator, two-stage, for argon.
9. Forceps.

**SPECIAL PRECAUTIONS:** Concentrated acids are powerful oxidizers, toxic, and corrosive liquids. Wear protective clothing and work in a fume hood.

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

**SAMPLE PREPARATION:**

3. Open the cassette filter holder and with forceps remove the sample filter. Fold the filter into quarters taking care not to lose any sample, and transfer to a clean, 50-mL hot block digestion tube.
4. Add 1.25 mL HCl. Cover with a plastic watchglass. Place in the hot block and heat at an internal temperature of 95 °C for 15 minutes.
   NOTE: The internal temperature may vary from the digital readout. Calibrate the hot block prior to digestion.
5. Remove the sample from the hot block and cool for 5 minutes. Remove watchglass and add 1.25 mL $HNO_3$. Replace watchglass and return to hot block at 95 °C for 15 minutes.
6. Remove the sample from the hot block and cool for at least 5 minutes. Rinse watchglass into the sample container and discard watchglass.
7. Dilute to 25-mL final volume with distilled, deionized Type II water.

**CALIBRATION AND QUALITY CONTROL:**

8. Calibrate the spectrometer according to the manufacturer's recommendations. Use standards consisting of the same 5% HCl : 5% $HNO_3$ matrix as the samples.
9. Analyze a standard every 10 samples.
10. Analyze a media blank every 20 samples, and a reagent blank every 10 samples.
11. Analyze a set of two laboratory control samples every 40 samples of a given matrix for a given analyte.
12. Check recoveries with at least two spiked media blanks per ten samples.
    NOTE: In the determination of lead, there may be a measurement interference (for example, samples with high aluminum levels). More recent instruments have a correction for this.

**MEASUREMENT:**

13. Set spectrometer to conditions specified by manufacturer.
14. Analyze standards, samples and quality control checks.
    NOTE: If the elemental value for a sample is above the linear range of the element(s) in question, dilute the sample solution with 5% HCl : 5% $HNO_3$ diluting solution, reanalyze and apply the appropriate dilution factor in the calculations.

**CALCULATIONS:**

15. Obtain the solution concentrations for the sample, $C_s$ (µg/mL), and the average media blank, $C_b$ (µg/mL), from the instrument.
16. Using the solution volumes of sample, $V_s$ (mL), and media blank, $V_b$ (mL), calculate the concentration, C ($mg/m^3$), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, mg/m^3$$

NOTE: µg/L ≡ $mg/m^3$

**EVALUATION OF METHOD:**

The method was evaluated for all elements and compounds listed in Table 1 and Table 2 between 1999 and 2001 using known amounts of bulk material [4]. Evaluation is ongoing for additional elements and compounds. The limits of detection and quantitation were also determined for each element. Two ICP instruments were used in the evaluation, a Thermal Jarrell Ash Model 61E [5] and a TJA IRIS [6], operated according to the manufacturer's instructions.

**REFERENCES:**

[1] WOHL [2001]. Metals validation using hot block digestion, Unpublished data. Wisconsin Occupational Health Laboratory, Madison, WI.
[2] NIOSH [1994]. Method 7300: Elements by ICP, NIOSH Manual of Analytical Methods, Fourth Edition, Issue 2, Aug. 15, 1994.
[3] WOHL [2001]. Metals Manual 2001, WOHL Internal Document, Updated Apr. 1, 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
[4] WOHL [2001]. WOHL General Operations Procedures Manual, WOHL Internal Document, Updated 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
[5] Thermal Jarrell Ash [1991]. ICAP 61E Plasma Spectrometer Operator's Manual, Thermal Jarrell Ash Corp., Part No. 128832-01, Feb., 1991.
[6] Thermal Jarrell Ash [1997]. IRIS Plasma Spectrometer User's Guide, Thermal Jarrell Ash Corp., Part No. 135811-0, Feb. 4, 1997.

**METHOD WRITTEN BY:**

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**TABLE 1: ANALYTE INFORMATION FOR VALID ELEMENTS AND COMPOUNDS**

| Analyte | Properties MW | Properties MP (°C) | LOD (µg/mL) | LOQ (µg/mL) | Estimated LOQ (µg/sample)* | Minimum** air vol. (L) | Maximum*** air vol. (L) |
|---|---|---|---|---|---|---|---|
| Al | 26.98 | 660 | 0.111 | 0.37 | 9.25 | 2 | 10,000 |
| As | 74.92 | 817 | 0.009 | 0.03 | 0.075 | 8 | 5,000,000 |
| Au | 196.97 | 10.63 | 0.015 | 0.05 | 1.25 | 1 | 3,300 |
| B | 10.81 | 2177 | 0.0094 | 0.0283 | 0.71 | 1 | 3,300 |
| Ba | 137.34 | 3.51 | 0.0018 | 0.006 | 0.15 | 1 | 100,000 |
| Be | 9.01 | 2178 | 0.00075 | 0.0025 | 0.062 | 35 | 25,000,00 |
| Bi | 208.98 | 271 | 0.025 | 0.085 | 2.12 | 1 | 10,000 |
| Ca | 40.08 | 842 | 0.099 | 0.33 | 8.25 | 2 | 10,000 |
| CaO | 56.08 | 2927 | 0.139 | 0.462 | 11.6 | 3 | 10,000 |
| Cd | 112.4 | 321 | 0.0037 | 0.012 | 0.30 | 3 | 500,000 |
| Co | 58.93 | 1495 | 0.003 | 0.011 | 0.27 | 3 | 500,000 |
| Cr | 52.00 | 1890 | 0.009 | 0.03 | 0.75 | 8 | 500,000 |
| Cu | 63.54 | 1083 | 0.020 | 0.060 | 1.50 | 15 | 500,000 |
| Fe | 55.85 | 1535 | 0.070 | 0.20 | 5.00 | 1 | 5,000 |
| $Fe_2O_3$ (as Fe) | 159.69 | 1462 | 0.070 | 0.20 | 5.00 | 1 | 5,000 |
| Ga | 69.72 | 29.75 | 0.03 | 0.09 | 2.25 | 1 | 3,300 |
| In | 114.82 | 156.3 | 0.015 | 0.05 | 1.25 | 15 | 500,000 |
| Mg | 24.31 | 651 | 0.047 | 0.14 | 3.50 | 1 | 10,000 |
| MgO | 40.32 | 2825 | 0.078 | 0.23 | 5.75 | 5 | 33,000 |
| Mn | 54.94 | 1244 | 0.0012 | 0.004 | 0.10 | 0.05 | 10,000 |
| Mo | 95.94 | 651 | 0.0072 | 0.024 | 0.60 | 0.5 | 10,000 |
| Nd | 92.906 | 2477 | 0.01 | 0.03 | 0.75 | 0.1 | 3,300 |
| Ni | 58.71 | 1453 | 0.012 | 0.039 | 0.98 | 1 | 50,000 |
| P | 30.97 | 44 | 0.3 | 1.0 | 25 | 250 | 500,000 |
| Pb | 207.19 | 328 | 0.023 | 0.07 | 1.75 | 35 | 100,000 |
| Pd | 106.4 | 1550 | 0.009 | 0.03 | 0.75 | 0.1 | 3,300 |
| Pt | 195.09 | 1769 | 0.0045 | 0.015 | 0.38 | 200 | 25,000,000 |
| Sb | 121.75 | 630.5 | 0.018 | 0.06 | 1.50 | 3 | 100,000 |
| Se | 78.96 | 217 | 0.021 | 0.064 | 1.60 | 8 | 250,000 |
| Sn | 118.69 | 232 | 0.015 | 0.05 | 1.25 | 1 | 25,000 |
| Sr | 87.62 | 769 | 0.002 | 0.006 | 0.15 | 300 | 100,000,000 |
| Te | 127.60 | 450 | 0.15 | 0.5 | 12.5 | 125 | 500,000 |
| Ti | 47.90 | 1675 | 0.005 | 0.016 | 0.40 | 0.1 | 10,000 |
| Tl | 204.37 | 304 | 0.044 | 0.133 | 3.32 | 35 | 500,000 |
| V | 50.94 | 1890 | 0.003 | 0.01 | 0.25 | 2.5 | 500,000 |
| Y | 88.91 | 1495 | 0.001 | 0.003 | 0.075 | 0.1 | 50,000 |
| Zn | 65.37 | 419 | 0.022 | 0.066 | 1.65 | 0.5 | 10,000 |
| ZnO | 81.37 | 1970 | 0.027 | 0.082 | 2.05 | 0.5 | 10,000 |

* Value based on a 25-mL sample volume.

** The minimum sampling volume needed to obtain the OSHA PEL at the LOQ for the element/compound at a sample digestion volume of 25 mL.

*** The maximum sampling volume for a given sample, calculated by taking 50,000 µg as the limit for the element/compound per sample.

NOTE: The LOD and LOQ values are dependent on the particular analytical instrument used. Also, LOD and LOQ values may vary for a particular element due to certain interelement interferences.

**TABLE 2. EXPOSURE LIMITS, CAS #, RTECS**

| Element (Symbol) | CAS # | RTECS | Exposure Limits, $mg/m^3$ (Ca = carcinogen) OSHA | NIOSH | ACGIH |
|---|---|---|---|---|---|
| Silver (Ag) | 7440-22-4 | VW3500000 | 0.01 (dust, fume, metal) | 0.01 (metal, soluble) | 0.1 (metal)<br>0.01 (soluble) |
| Aluminum (Al) | 7429-90-5 | BD0330000 | 15 (total dust)<br>5 (respirable) | 10 (total dust)<br>5 (respirable fume)<br>2 (salts, alkyls) | 10 (dust)<br>5 (powders, fume)<br>2 (salts, alkyls) |
| Arsenic (As) | 7440-38-2 | CG0525000 | varies | C 0.002, Ca | 0.01, Ca |
| Barium (Ba) | 7440-39-3 | CQ8370000 | 0.5 | 0.5 | 0.5 |
| Beryllium (Be) | 7440-41-7 | DS1750000 | 0.002, C 0.005 | 0.0005, Ca | 0.002, Ca |
| Calcium (Ca) | 7440-70-2 | -- | varies | varies | varies |
| Cadmium (Cd) | 7440-43-9 | EU9800000 | 0.005 | lowest feasible, Ca | 0.01 (total), Ca<br>0.002 (respir.), Ca |
| Cobalt (Co) | 7440-48-4 | GF8750000 | 0.1 | 0.05 (dust, fume) | 0.02 (dust, fume) |
| Chromium (Cr) | 7440-47-3 | GB4200000 | 0.5 | 0.5 | 0.5 |
| Copper (Cu) | 7440-50-8 | GL5325000 | 1 (dust, mists)<br>0.1 (fume) | 1 (dust)<br>0.1 (fume) | 1 (dust, mists)<br>0.2 (fume) |
| Iron (Fe) | 7439-89-6 | NO4565500 | 10 (dust, fume) | 5 (dust, fume) | 5 (fume) |
| Potassium (K) | 7440-09-7 | TS6460000 | -- | -- | -- |
| Lanthanum | 7439-91-0 | -- | – | – | -- |
| Lithium (Li) | 7439-93-2 | -- | -- | -- | -- |
| Magnesium (Mg) | 7439-95-4 | OM2100000 | 15 (dust) as oxide<br>5 (respirable) | 10 (fume) as oxide | 10 (fume) as oxide |
| Manganese (Mn) | 7439-96-5 | OO9275000 | C 5 | 1; STEL 3 | 5 (dust)<br>1; STEL 3 (fume) |
| Molybdenum (Mo) | 7439-98-7 | QA4680000 | 5 (soluble)<br>15 (total insoluble) | 5 (soluble)<br>10 (insoluble) | 5 (soluble)<br>10 (insoluble) |
| Nickel (Ni) | 7440-02-0 | QR5950000 | 1 | 0.015, Ca | 0.1 (soluble)<br>1 (insoluble, metal) |
| Phosphorus (P) | 7723-14-0 | TH3500000 | 0.1 | 0.1 | 0.1 |
| Lead (Pb) | 7439-92-1 | OF7525000 | 0.05 | 0.05 | 0.05 |
| Antimony (Sb) | 7440-36-0 | CC4025000 | 0.5 | 0.5 | 0.5 |
| Selenium (Se) | 7782-49-2 | VS7700000 | 0.2 | 0.2 | 0.2 |
| Tin (Sn) | 7440-31-5 | XP7320000 | 2 | 2 | 2 |
| Strontium (Sr) | 7440-24-6 | – | – | – | -- |
| Tellurium (Te) | 13494-80-9 | WY2625000 | 0.1 | 0.1 | 0.1 |
| Titanium (Ti) | 7440-32-6 | XR1700000 | -- | -- | -- |
| Thallium (Tl) | 7440-28-0 | XG3425000 | 0.1 (skin) (soluble) | 0.1 (skin) (soluble) | 0.1 (skin) |
| Vanadium (V) | 7440-62-2 | YW240000 | -- | C 0.05 | -- |
| Tungsten | 7440-33-7 | – | 5 | 5<br>10 (STEL) | 5<br>10 (STEL) |
| Yttrium (Y) | 7440-65-5 | ZG2980000 | 1 | N/A | 1 |
| Zinc (Zn) | 7440-66-6 | ZG8600000 | – | -- | -- |
| Zirconium (Zr) | 7440-67-7 | ZH7070000 | 5 | 5, STEL 10 | 5, STEL 10 |

**TABLE 3: VALIDATION SUMMARY**

| Analyte | Status[1] | Analyte | Status | Analyte | Status |
|---|---|---|---|---|---|
| Ag | Not Valid | CuO | Valid | S | Not Valid |
| Al | Valid | Fe | Valid | Sb | Partially Valid[4] |
| $Al_2O_3$ | Not Valid | $Fe_2O_3$ | Valid | $Sb_2O_3$ | Partially Valid[5] |
| As | Valid | Ga | Valid | Se | Valid |
| Au | Valid | In | Valid | Si | Not Valid |
| B | Valid | KCl | Pending | Sn | Partially Valid[6] |
| Ba | Pending | Mg | Valid | SnO | Pending |
| BaO | Pending | MgO | Valid | $SnO_2$ | Pending |
| $BaO_2$ | Pending | Mn | Valid | Sr | Valid |
| $BaCl_2$ | Valid | MnO | Valid | $SrCrO_4$ | Valid (by Cr) |
| $BaSO_4$ | Pending | Mo | Valid | Te | Valid |
| Be | Valid | NaCl | Pending | Ti | Valid |
| Bi | Partially Valid[2] | Nd | Valid | Tl | Valid |
| Ca | Valid | Ni | Valid | V | Valid |
| $CaCO_3$ | Valid | P | Valid | $V_2O_5$ | Valid |
| CaO | Valid | Pb | Partially Valid[3] | Y | Valid |
| Cd | Valid | $PbCrO_4$ | Valid (by Cr) | Zn | Valid |
| Co | Valid | PbO | Valid | ZnO | Valid |
| Cr | Valid | Pd | Valid | Zr | Not Valid |
| Cu | Valid | Pt | Valid | ZrO | Not Valid |

[1] Status definitions

Valid: The method is suitable for samples up to at least 0.0500 g bulk material with recoveries of between 90 and 110 percent. This weight exceeds most expected levels encountered in work environments.

Partially Valid: The method is suitable with bulk-material recoveries of between 90 and 110 percent under certain conditions (as footnoted above).

Not Valid: The method procedure is not suitable for samples at any weight with recoveries of between 90 and 110 percent. An alternative method should be used.

[2] Valid up to 10,000 µg/sample and within 7 days of sample digestion.
[3] Valid up to 50,000 µg/sample and at least 24 hours after sample digestion; Valid up to 15,000 µg/sample within 24 hours of sample digestion.
[4] Valid up to 25,000 µg/sample and within 7 days of sample digestion.
[5] Valid up to 25,000 µg/sample and within 7 days of sample digestion.
[6] Valid up to 30,000 µg/sample and within 7 days of sample digestion.

NOTE: The upper limits of the method can be extended by serial dilution of the samples at the time of analyses.

Test America

Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series and 3600 Series Methods for Analysis by Methods 8081A and 8082 [Methods 3500 Series and 3600 Series]

# Title: Extraction and Cleanup of Organic Compounds From Waters and Soils, Based on SW--846 3500 Series and 3600 Series Methods for Analysis by Methods 8081A and 8082 [Methods 3500 Series and 3600 Series]

**Approvals (Signature/Date):**

| | |
|---|---|
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| Lisa Stafford — Date 4/24/2014<br>Interim Quality Assurance Manager | Robert Hrabak — Date 4/25/14<br>Laboratory Manager |

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## 1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, soil and waste matrices for analysis by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA).

1.1. Extraction procedures for the determinative methods 8081A and 8082 are covered.

1.2. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “Federal Program Requirements” must be checked and incorporated.

1.4. Specific handling procedures for tissue samples are found in WS-WI-0018.

## 2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction: A measured volume of sample is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.1.1. This SOP has procedures for extracting both the traditional sample volume (1000mL) as well as reduced sample volumes (250 or 125 mL). The traditional volume is referred to as a “1 L separatory funnel extraction”, and the smaller volumes are referred to as “250 mL Limited Volume Initiative/Reduced Volume Extraction” (250 mL LVI/RVE) and “125 mL Limited Volume Initiative/Reduced Volume Extraction (125 mL LVI/RVE) in this SOP.

2.2. Sonication Extraction: A measured weight of sample, typically 30 g (10 g for biologics), is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted with 1:1 methylene chloride: acetone three times using an ultrasonic horn. In pre-approved situations, other solvents or solvent combinations may be used to control co-extracted matrix interferences.

2.3. Soxhlet Extraction: A 15 g sample is mixed with anhydrous sodium sulfate to form a fee flowing powder. This is extracted with refluxing 1:1 methylene chloride:acetone for 16-24 hours.

2.4. Waste Dilution: A measured weight of sample (typically 1 g), is diluted to 10 mL with hexane which is now ready for cleanup.

2.5. Microwave Extraction: A measured weight of sample, typically 15 g (10 g for biologics), is extracted with 1:1 methylene chloride: acetone.

2.6. Cleanup and Concentration: Procedures are presented for removing interferents from sample extracts, and for drying and concentrating the extract to final volume for analysis.

## 3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

## 4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3. To prevent analyte loss, extracts which are not immediately concentrated by KD must be protected from light during storage. Extracts may be either refrigerated or stored in an opaque container.

## 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The PCB solutions used to prepare standards for this method may arrive at concentrations as high as 1,000 ppm. Exercise extreme caution when handling these high concentration standards.

5.1.2. If any PCB standards are spilled inside or outside of a fume hood at concentrations equal to or greater than 5 PPM, all materials in contact must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions before cleaning up the spill.

5.1.3. If any PCB standards are spilled outside the fume hood at concentrations greater than 200 ppm, the Emergency Response Team must be activated for special clean-up procedures, and possibly additional testing of the spill location and additional regulatory notifications.

5.1.4. All glassware and solid lab trash in contact with any PCB's at concentrations equal to or greater than 5 PPM must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions before disposing of this waste.

5.1.5. Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Microwaves used for these processes must be equipped with an automated vapor sensor and shutoff system that automatically shuts down the digestion/extraction process when vapor pressure reaches pre-specific levels. Users must follow procedures in the microwave operator's manual to ensure that the vapor sensors are functional and working properly prior to starting each extraction/digestion batch.

5.1.6. Hearing protection is required when sonicating samples.

5.1.7. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.8. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.9. Laboratory procedures such as repetitive use of pipettes, repetitive transferring

of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.10. Sulfuric acid cleanup must not be performed on any matrix that may have water or acetone present as a violent reaction between the acid and water or acetone may result in acid exploding out of the vessel.

5.1.11. Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and cleanup techniques before handling this material. Spilled mercury requires that special cleanup tools and procedures be used. .

5.1.12. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar® or similar cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware. This includes when seating Teflon stoppers into glass separatory funnels.

5.1.13. The use of vacuum systems during Florisil cartridge cleanup presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.14. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.1.15. Ensure that the vacuum exhaust hose used during the Florisil cartridge cleanup is securely anchored inside of a fume hood so that solvent vapors are not pumped into the working environment.

5.1.16. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the process may be performed behind a closed fume hood sash. After each of the three extraction cycles, ensure that the

stoppers are removed from the top of the separatory funnels.

5.1.17. When Soxhlet extractions are performed overnight or unattended, special precautions must be taken. Open chiller values to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.1.18. The kiln used to clean glassware creates an extreme risk of severe burns. Allow sufficient time for the kiln to cool before opening the doors. Ensure that appropriate heat protective gloves are worn when working with freshly kilned glassware. Other surfaces in the kiln may remain hot, so exercise caution when reaching into the kiln.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table**. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

| **Material (1)** | **Hazards** | **Exposure Limit (2)** | **Signs and symptoms of exposure** |
|---|---|---|---|
| Acetone | Flammable | 1000 ppm-TWA | Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache. |
| Hexane | Flammable Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| Mercury | Poison | 0.1 Mg/M3 Ceiling (Mercury Compounds) | Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. **May affect the central nervous system.** Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage. |

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Methylene Chloride | Carcinogen Irritant | 25 ppm-TWA 125 ppm-STEL | Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin. |
| Sodium Hydroxide | Corrosive Poison | 2 ppm, 5 mg/m3 | This material will cause burns if it comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system. |
| Sulfuric Acid (1) | Corrosive Oxidizer Dehydradator | 1 mg/m3 | This material will cause burns if it comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

## 6. EQUIPMENT AND SUPPLIES

6.1. Glassware is kilned at 400°C for at least 2 hours. Extremely dirty glassware can be cleaned with soap and water prior to using the kiln. Refer to SOP WS-OP-0011 for details of glassware cleaning.

***Heat resistant gloves must be used when handling kilned glassware.***

6.2. Equipment and supplies for extraction procedures

6.2.1. Separatory funnel, 2L (Teflon or glass)

6.2.2. Separatory funnel, 250mL (Teflon or glass)

6.2.3. Separatory funnel, 500mL (Teflon or glass)

6.2.4. Separatory funnel rotator with timer

6.2.5. Balance: >1400 g capacity, accurate to ±0.1 g.

6.2.6. pH indicator paper, wide-range: covers extraction pH.

6.2.7. Graduated cylinder: 1 liter, other sizes may be used.

6.2.8. Erlenmeyer flask, beaker, or french square jar: 125 & 300 mL (other sizes optional).

6.2.9. Solvent dispenser pump or 100 mL graduated cylinder.

6.2.10. Round or flat bottom flask: 250, 500 mL or 1 L.

6.2.11. Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).

6.2.12. Cooling condensers.

6.2.13. Heating mantle: rheostat controlled, with timer.

6.2.14. Beakers: 250 & 400 mL, graduated

6.2.15. Soxhlet extractor.

6.2.16. Balance: >100g capacity, accurate to ±0.01g

6.2.17. Sonicator (at least 300 watts), with sonicator horn, 3/4 inch. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2.18. Kuderna-Danish (KD) apparatus: 500mL, with 10mL concentrator tube, and 3-ball macro Snyder column.

6.2.19. Locally assembled solvent vapor condensing/recovery system for use with 6.2.17 above.

6.2.20. Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.2.21. Nitrogen blowdown apparatus.

6.2.22. Culture tubes: 16 mL, 16 mm x 125 mm

6.2.23. Syringe: 1 mL.

6.2.24. Glass wool.

To ensure glass wool is free of contaminants, it is necessary to soxhlet rinse glass wool in methylene chloride for 18 hours.

6.2.25. Glass funnel: 75 X 75 mm.

6.2.26. Disposable pipettes.

6.2.27. Aluminum foil.

6.2.28. Paper towels.

6.2.29. Pressure rotor and pressure reactor

6.2.30. Extraction vessel and extraction vessel cover

6.2.31. Glass inserts

6.2.32. Microwave – Milestone Ethos EX

6.3. Equipment and Supplies for Cleanup Procedures

6.3.1. Vacuum system for eluting multiple cleanup cartridges. Vac elute manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.

6.3.2. Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.

6.3.3. Vacuum pressure gauge.

6.3.4. Rack for holding collection vessels in the manifold.

6.3.5. Mechanical shaker or mixer: vortex genie or equivalent.

6.3.6. Disposable pipettes

6.3.7. Culture tubes: 8 mL, 13 mm x 100 mm

## 7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

7.1.1. All reagents must be ACS reagent grade or better unless otherwise specified.

7.1.2. Sodium hydroxide (NaOH) solution: 10N reagent, grade.

7.1.3. Sulfuric acid ($H_2SO_4$), concentrated; reagent grade.

7.1.4. Sulfuric acid (1:1): carefully add 500 mL of $H_2SO_4$ to 500 mL of reagent water. Mix well.

**WARNING: Always add concentrated acids or bases to water, never add water to the concentrated acid or base solution. Due to the reaction between acid and water, add the acid slowly to water to prevent breaking of the mixing vessel**

7.1.5. Organic free reagent water (deionized water). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks, in accordance with SOP WS-QA-0014, "Monitoring of Reagent-Grade Laboratory Water.

7.1.6. Sodium sulfate ($Na_2SO_4$), granular, anhydrous, reagent grade: purify by heating at 400°C for a minimum of two hours.

7.1.7. Extraction/exchange solvents: methylene chloride, hexane, acetone, pesticide quality or equivalent

7.1.8. Acetone: used for cleaning

7.1.9. Nitrogen, reagent grade

7.2. Reagents for Cleanup Procedures

7.2.1. Florisil: 500 mg or 1 g cartridges with stainless steel or PTFE frits (JT Baker cat # 7420-07 or equivalent)

7.2.2. Styrene Divinylbenzene polymer sorbent: 500 mg pre-packed in 6mL polypropylene cartridges with polyethylene frits (Phenomenex, cat # 8B-S014-HCH, or equivalent).

7.2.3. Graphitized Carbon sorbant cleanup: 500 mg pre-packed in 6mL polypropylene cartridges with polyethylene frits (Restek "CarboPrep-90", cat # 26092 or equivalent).

7.2.4. Mercury: triple distilled.

7.2.5. Sulfuric acid, concentrated.

7.3. Standards

7.3.1. Source Standards

7.3.1.1. Source standards are purchased as certified solutions or prepared from neats. Semivolatile source standards are stored at room temperature. All stock standards must be protected from light.

7.3.1.2. Source standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Standards must be allowed to come to room temperature before use.

7.3.1.3. Expired standards must be rotated out of the lab to the Hazardous Waste storage area.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 3. Surrogate spiking standards are prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the Matrix Spike (MS), Matrix Spike (MSD), and the Laboratory Control Sample (LCS). Prepare MS/LCS spiking standards at the concentrations listed in Table 3. Spiking standards are purchased or prepared as dilutions of the source standards. Spiking solutions must be refrigerated and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner. Endrin aldehyde and BHC standards are given a 2 week expiration date from the time of preparation. Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated.

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 0 - 6°C in glass containers with Teflon®-lined caps

8.3. Holding Times

8.3.1. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2. For TCLP leachates, extraction is initiated within seven days from when the leaching procedure is completed.

8.3.3. Analysis of the extracts must be completed within forty days of extraction.

## 9. QUALITY CONTROL

Quality Control Batch: The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, an LCS and a MS/MSD. (In some cases, at client request, it may be appropriate to process a

matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

9.1. Definition of Matrix
The possible matrix types are aqueous, solid, waste, TCLP leachate, and biologics. Refer to WS-WI-0018 for tissue sample handling.

9.2. Insufficient Sample
If insufficient sample is available to process a MS/MSD, a second LCS may be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented using the non-conformance memo (NCM) program in TALS.

9.3. Sample Count
Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.4. Method blanks for biologic samples (fish, plants, worms, etc.) normally use 10 g of sodium sulfite spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5. Method Blank
A MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous MB use 1000 mL for 1L separatory funnel extraction, 125 mL for 125 mL LVI/RVE, and 250 mL for 250 mL LVI/RVE of organic free reagent water (deionized water) spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 30 g of kilned sodium sulfate for 3550B and 15 g Ottawa sand for 3546 spiked with the surrogates. The MB goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP and STLC MB use 200 mL of leachate fluid spiked with the surrogates. Dilute to 1000mL with organic free reagent water (deionized water). The MB goes through the entire analytical procedure, including any cleanup steps.

9.5.4. MB for waste dilution procedures consist of 1 g of vacuum oil spiked with the surrogates. The MB goes through the entire analytical procedure, including

any cleanup steps.

9.5.5. MB for tissue consists of 200 uL fish oil (Sigma F8020-500mL) mixed with either 10 g sodium sulfate (3550B/C) or 10 g Ottawa sand (3546).

9.6. Laboratory Control Sample (LCS)

LCS is a well-characterized, laboratory generated sample used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the MB (See Sections 9.5.1 - 9.5.5), but is spiked with the LCS standard and the surrogate.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS is an environmental sample to which known concentrations of target analytes have been added. A MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. The MS and MSD are spiked with the LCS standard and the surrogate.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.11. Sacramento QC Program Further details of QC and corrective action guidelines are presented in the Sacramento QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

## 10. CALIBRATION

10.1. On the day of use, measure 2.0 mL of solvent into a 16mL culture tube using a 1.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Seal the culture tube – this is the 'check' or 'reference' vial for cleanup with Florisil. Using the same procedure, create a 10.00mL 'check' vial using a 5.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Record the container lot, solvent, gastight syringe or serological pipet ID, date, and initials of preparer for all check vials in a controlled logbook. The culture tubes containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently within the criteria of $10 \pm 0.3$ or $2 \pm 0.06$ mL as required.

10.2. On a daily basis, calibrate any auto-pipettors to be used in accordance with SOP WS-QA-0004.

10.3. On the day of use, calibrate any balances to be used in accordance with SOP WS-QA-0041.

10.4. Prior to use, ensure any containers used for quantititative steps (e.g final volume and Florisil) are verified as described in SOP WS-QA-0004.

10.5. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

10.5.1. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

## 11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (Section 11.2), sonication extraction (Section 11.3), waste dilution (Section 11.4), extract concentration (Section 11.5), and extract cleanup (Section 11.6) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Method 3510C, Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and LCS spiking standards from refrigerator and allow standards to warm to room temperature.

11.2.1.1. If the sample bottle contains a layer of sediment greater than approximately ½" in depth, contact the department manager and the project manager to determine how to proceed with the sample extraction. Options may include the following:

a) Decanting and extracting only the liquid fraction in a separatory funnel before adding the surrogate and/or spike solutions.
b) Adding the surrogate and/or spike solutions to the sample with the sediment still in the original sample container.
c) Processing the aqueous portion as described in a) along with creating a new sample id for the soil matrix. The sample bottle would then be rinsed with 60 mL of DCM, spiked with an appropriate volume of surrogate solution and shaken for 3 minutes. The addition of 60 mL of DCM will be repeated 2 more times and all solvents combined to create a second extract for the sample that is to be added to the extraction batch of water samples

NOTE: In the case of option "b", after adding the appropriate surrogates/spikes, the sample would be gently swirled for mixing purposes and then gently poured into the separatory funnel, thus minimizing the amount of sediment transferred to the extraction v vessel during the process. The original sample container would then be rinsed with 60 mL of DCM. This DCM rinseate would then be poured into the extraction vessel.

11.2.2. Measure the initial sample pH with wide-range pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log and note on the benchsheet if there is any discrepancy.

11.2.3. The normal sample volume is 1 liter for 1L separatory funnel extraction, 125 mL for 125 mL LVI/RVE, and 250 mL for 250 mL LVI/RVE. Other sample volumes may be used to obtain specific reporting limits, dirty samples, and reduced sample volumes, diluted to 1 liter, 125mL, or 250 mL with organic free reagent water (deionized water).

11.2.4. Obtain sample weight(s) to ± 0.1 g. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter. This may be done in two ways depending if a dilution is necessary.

11.2.4.1. For samples not requiring a dilution weigh the gross container on a tared balance and after extraction has been initiated weigh the tared container on a tared balance and use the difference as the sample weight.

11.2.4.2. For samples requiring a dilution, subsample the desired aliquot amount in accordance with SOP-QA-0018 into a tared container. Dilute to 1L nominal for 1L separatory funnel extraction and 250mL nominal for 250 mL LVI/RVE and 125 mL nominal for 125 mL LVI/RVE with organic free reagent water (deionized water). This must be documented in an NCM, as there may be a low bias to the analysis when the entire container is not use, and the container is therefore not rinsed and the rinsate combined with the extraction solvent.

11.2.4.3. For TCLP samples, use 200mL of leachate and dilute to 1L in organic free reagent water.

**Note:** Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

11.2.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for spiking volumes and concentrations.

11.2.6. For a TCLP batch prepare a MB as specified in section 9 of this SOP. In addition, prepare a TCLP method blank by measuring 200mLs of buffer solution used in the leaching procedure and dilute to 1L nominal using the method described in 11.2.4. No leachate LCS is required by this method.

11.2.7. After samples have warmed to room temperature, add the surrogate standard to all samples and the LCS solution to the necessary samples as described in Section 9 of this SOP. See Tables 3 and 4 for spiking volumes and concentrations. Return spiking solutions to the refrigerator as soon as possible after spiking.

11.2.8. Adjust sample pH to between 5 and 9. Use the minimum amount of 1:1 $H_2SO_4$ or 10N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper.

11.2.9. Record adjusted pH, spiking volumes standard numbers, and reagent lots on the benchsheet.

**Note:** If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer part of the sample to the separatory funnel and then add the spike to the bottle.

11.2.10. Mix well, or if the sample contains sediment, decant before transfer. Transfer the sample to the separatory funnel. Rinse the sample bottle with 60 mL methylene chloride for 1L separatory funnel extraction. 30 mL for 250 mL LVI/RVE and 15 mL for 125 mL LVI/RVE. Transfer the rinsate into the 2L glass or Teflon separatory funnel for 1L separatory funnel extraction, 500 mL glass or Teflon separatory funnel for 250 mL LVI/RVE, or 250 mL glass or Teflon separatory funnel for 125 mL LVI/RVE.

11.2.11. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. As described in 5.1.11, Venting should be done at a minimum of four rotations followed by eight rotations on the first extraction cycle and eight rotations on the second extraction cycle.

***WARNING:* Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.**

11.2.12. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (at least 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.2.13. One method for reducing the effects of emulsions is to swirl the funnel after allowing it to rest for the required amount of time, wait approximately a minute, and then drain the extract without collecting the emulsion. After the third (final) extraction and rest period, add a squirt of DCM to identify where the layer of DCM and water is separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water.

***Note:** 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable for 1L separatory funnel extraction. Subsequent shakes should recover at least 50 mL of solvent for 1L separatory funnel extraction. Similarly, it is anticipated that only 20 of the 30 mL used (or 10 of 15 mL used) may be recovered on the first shake when extracting smaller water volumes.

11.2.14. Fill a funnel with anhydrous sodium sulfate (10-15g for LVI/RVE, nearly a

full funnel for 1L separatory funnel extraction). The funnel can be plugged with glass wool to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just ***before*** the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

***Note:*** Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate prior to the concentration step. Ensure to minimize the amount of water collected.

11.2.15. Repeat the extraction process two more times using fresh portions of solvent (60 mL for 1L separatory funnel extraction, 30 mL for 250 mL LVI/RVE, and 15 mL for LVI/RVE) combining the three solvent extracts in the collection container.

11.2.16. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.17. Dispose of solvent and water remaining in the extractor into the LLE waste drum. Waste methylene chloride goes to the waste solvent drum for disposal.

11.2.18. Cap the collection container and refrigerate the container if the extract is not concentrated immediately. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.3. Method 3546, Solid Extraction by Microwave

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow standards to warm to room temperature.

11.3.2. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.3.3. Weigh 15 g of sample (10 g for biologics) ± 1.0 g into an extraction vessel. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.3.3.1. For visually dirty samples, it may be necessary to weigh the sample into a glass insert to be placed into an extraction vessel. Contact your supervisor to determine if this is the case.

11.3.4. Mix weighed sample with a spatula. If clay clumps or other sample

aggregation is evident, add Ottawa sand (not to exceed 5 g) as needed to facilitation disaggregation and mix as necessary.

11.3.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.3.6. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.3.7. Immediately add 25 mL of 1:1 hexane:acetone to extraction vessels.

**Note:** Steps 11.3.6 - 11.3.7 should be performed rapidly to avoid loss of the more volatile extractables.

11.3.8. Add flared extraction vessel covers to each extraction vessel containing sample and QC aliquots.

11.3.8.1. It is important that each cover fits snugly to ensure a proper seal. The cover should not slide easily or loosely inside the extraction vessel, but should require some finger pressure to insert firmly. A cover flaring tool should be used.

11.3.8.2. For the visually wettest sample, add the thermowell liner into the extraction vessel cover to create the representative sample that the ATC temperature sensor can be inserted into.

11.3.9. Place each extraction vessel into a pressure reactor. Screw on the pressure cap/safety lid. The pressure cap should be hand tightened until the sealing valve is flush with the top of the cap.

11.3.9.1. For the representative sample created in 11.3.8.2, add the protection foil and appropriate safety lid.

11.3.10. Place all the extraction vessels into the rotor so that the pressure-release valves are facing outside of the rotor on the outside ring and inside toward the center on the inside ring.

**WARNING: Microwave ovens used for extraction or digestion of samples can create elevated pressure in the extraction/digestion containers. Follow procedures in the operator's manual to ensure that the vapor sensor(s) are functional and working properly prior to starting each extraction/digestion batch.**

11.3.10.1. Place the rotor in the microwave oven and insert the ATC temperature sensor into the representative sample (11.3.8.2 and 11.3.9.1).

11.3.11. Close the microwave oven and start the appropriate extraction profile.

11.3.11.1. A 10 minute ramp from 25C to 100 C, hold for 25 minutes at 100C, followed by a 15 minute cool down to 25C

11.3.12. After the extraction period and cool down, pressure reactors should be at ambient temperature prior to removal from the rotor and opening.

11.3.13. Proceed to Section 11.8 for concentration and Section 11.9 for cleanup.

11.3.13.1. Discard any glass inserts. To clean extraction vessels and pressure cap/safety lid, use soap and water followed by a solvent rinse for. Use only solvent to rinse the probe. Allow to air dry prior to use.

11.4. Method 3550B, Solid Extraction by Sonication

11.4.1. Sonicator Tuning

11.4.1.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

11.4.1.2. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11.4.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.4.3. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.4. Weigh 30 g of sample (10 g for biologics) ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate (10 g for biologics) for the method blank and LCS.

11.4.5. Mix weighed sample with a spatula or blending rotor, adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.6. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.4.7. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.4.8. Immediately add a minimum of 100 mL of 1:1 DCM/acetone to the beaker. In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

**Note:** Steps 11.4.7 - 11.4.8 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.9. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.4.10. Place the bottom surface of the sonicator disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

**WARNING: Hearing protection is required when sonicating samples.**

***Note:*** Do ***not*** use ***Microtip*** probe.

11.4.12. Decant extracts into a clean beaker.

11.4.13. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication.

11.4.14. Cap the collection container. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.5. Method 3550C, Solid Extraction by Sonication

Method 3550C differs from 3550B in the order in which samples are dried and spiked. Be certain that the method performed reflects the method requested in the LIMS (TALS).

11.5.1. Sonicator Tuning

11.5.1.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed, or annually, whichever is sooner.

11.5.1.2. Newer models of sonicators may be self-tuning. Read the manufacturer's directions prior to operating this equipment.

11.5.1.3. ***Note:*** Do ***not*** use ***Microtip*** probe.

11.5.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.5.3. Homogenize the sample by mixing thoroughly. (See SOP WS-QA-0018). Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.5.4. Weigh 30 g of sample (10 g for biologics) ± 1.0 g into a 250 mL beaker, 400 mL beaker or glass jar of sufficient size that the sodium sulfate and extraction solvents can be added. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate (10 g for biologics) for the method blank and LCS.

11.5.5. Prepare a MB, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

11.5.6. Add the surrogate spiking solution to each sample, MB, LCS, and MS/MSD. Add the appropriate matrix spiking solution to each MS/MSD and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.5.7. Mix weighed sample with a spatula or blending rotor, adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.5.8. Immediately add a minimum of 100 mL of 1:1 DCM/acetone to the beaker. In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

**Note:** Steps 11.5.6 - 11.5.8 should be performed rapidly to avoid loss of the more volatile extractables.

11.5.9. Make sure that all sonicator horns have been cleaned prior to sonicating. To achieve a clean sonicator horn, immerse each sonicator horn in a tri-pour

beaker of de-ionized (DI) water for 2-3 pulses. Immediately rinse the horns with acetone to remove any excess water. Finish by rinsing each horn with dichloromethane (DCM). Sonicators are ready for use.

11.5.10. Place the bottom surface of the sonicator disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.5.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

**WARNING: Hearing protection is required when sonicating samples.**

11.5.12. Decant extracts into a clean beaker.

11.5.13. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication.

11.5.14. Cap the collection container. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.6. Method 3540C, Extraction of Solids with Soxhlet Apparatus

**WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before you turn the heating element on. Check all of the condensers above 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this extraction cycle is to be left unattended (e.g., overnight), the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.**

11.6.1. Decant and discard any waste layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless the client requires extraction of this material. If the sample consists primarily of foreign materials, consult with the client. Document on the benchsheet if a water layer was discarded.

11.6.2. Remove surrogate and matrix spiking solutions from refrigerator and allow solutions to warm to room temperature.

11.6.3. Weigh 15 g of sample ± 1.0 g into a beaker or glass jar, recording the weight to the nearest 0.1 g on the benchsheet. Use 15 g of sodium sulfate for the method blank and LCS. Add 15 g of anhydrous sodium sulfate to the samples

and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period.

11.6.3.1. Sample weights less than 15 g but over 5 g may be used if the appropriate reporting limits can be met.

11.6.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.6.5. Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Dilution (MS/MSD) and LCS. Refer to Tables 3 – 6 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.6.6. Place approximately 300 mL of 1:1 methylene chloride/acetone into a 500 mL round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16–24 hours at 4–6 cycles per hour. The flask should be heated by setting the controller to 3.5 on the dial. Check the system for leaks at the ground glass joints after it has warmed up. In pre-approved situations, other solvents (such as Hexane or DCM) may be used to control co-extracted matrix interference).

**WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensers are cold before you turn the heating element on. Check all of the condensers about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If the cycle is to be left unattended (e.g. overnight) the first analyst to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.**

11.6.7. Allow the extract to cool after the extraction is complete. Collect all solvent in the round bottom flask. Disassemble by gently twisting the soxhlet from the flask. Do not force the glassware apart.

**WARNING: Use of cut-resistant gloves (Kevlar®) or similar material) is required when assembling or disassembling glassware.**

11.6.8. Collect the dried extract in a KD or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and

add it to the funnel to complete the quantitative transfer.

11.6.9. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration and Section 11.9 for cleanup.

11.7. Method 3580A, Waste Dilution

11.7.1. This method is used for materials that are soluble in an organic solvent.

11.7.2. Remove surrogate and matrix spiking solutions from refrigerator and allow the standards to warm to room temperature.

11.7.3. Tare the vial, then transfer approximately 1 g of sample to the vial. Record the weight to the nearest 0.01 g.

11.7.4. Add the surrogate solution (Table 3).

11.7.5. Dilute to 10 mL with hexane.

11.7.6. Prepare the necessary QC (MB/LCS/LCSD/TOX) samples by adding 1 g of clean oil matrix (vacuum oil or equivalent) to clean 16 mL culture tubes. Add surrogate and matrix spike solution (Table 3) to the appropriate QC samples. Dilute the contents of each tube to 10 mL with hexane.

11.8. Concentration: According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.8.1. Kuderna-Danish (KD) Method

11.8.1.1. For aqueous samples, loosely plug the stem of a 75mm x 75mm glass funnel with glass wool and rinse well with DCM. For solid samples place a folded piece of Whatman #1 filter paper into the glass funnel.

11.8.1.2. For LVI/RVE samples, add approximately 10-15 g of anhydrous sodium sulfate to the funnel cup. For 1L separatory funnel extraction, add anhydrous sodium to nearly fill up the funnel cup. For solid samples, add approximately 5-10 g of anhydrous sodium sulfate to the funnel cup.

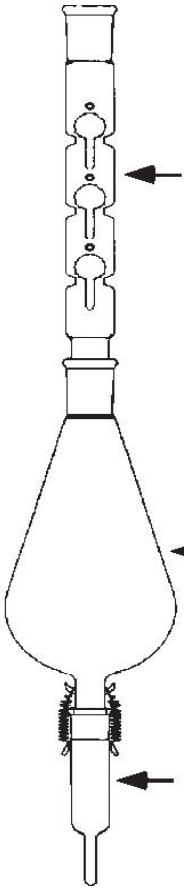
11.8.1.3. Pour the sample through the prepared glass funnel into the KD flask. Add one or two clean boiling chips to the KD flask.

11.8.1.4. Rinse the top of the funnel as well as the sample container with DCM.

11.8.1.5. Prepare the three ball Snyder columns by rinsing them with DCM to ensure that the balls are not stuck and that the column will work properly.

**WARNING: Use of cut-resistant gloves (Kevlar® or similar material) is required when assembling or disassembling glassware.**

**Figure 2, KD Setup (See Section 11.6.1 for description)**



11.8.1.6. Place the KD apparatus on a water bath (85-90°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood. Attach the solvent vapor recovery system to the top of the Snyder column.

**WARNING: Do not concentrate to dryness as this may cause flammable vapor in the KD flask to superheat and explode.**

11.8.1.7. For GPC cleanup, when the apparent volume of the extract reaches 10-15 mL, remove the KD apparatus from the water bath and allow it to drain and cool

11.8.1.8. For samples to be loaded o the Gel Permeation Chromatograph (GPC), adjust the final DCM volume to 10 mL and split this into two, 5mL portions. Archive one of the 5 mL fractions and GPC the other.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.8.1.9. To solvent exchange to hexane, momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the Snyder column. Alternatively, pour the exchange solvent into the top of the Snyder column. For LVI/RVE, add only 25 mL of hexane. No solvent exchange is necessary for MAE.

11.8.1.10. Put the KD assembly back on the water bath and concentrate the extract to ~5mLs, as described in the previous sections, raising the temperature of the water bath, if necessary, to maintain proper distillation

11.8.1.11. Remove the KD apparatus from the water bath and allow it to cool and drain. If the level of the extract is above the level of the concentrator tube joint, add a new boiling chip and continue to distill the solvent as necessary. If redistilling, remove the KD apparatus from the water bath and allow it to cool and drain before proceeding to the next step.

11.8.1.12. Remove the Snyder column and rinse the inside of the top joint of the K-D flask with a minimum amount of DCM, rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask.

Note: When performing KD on the GPC portion of the extract, bring to a final volume of 5 mL (as the 10 mL extract was split in the previous Section).

11.8.1.13. If further concentration is indicated in Table 1, either the micro Snyder column technique or nitrogen evaporation (Section 11.6.2) technique is used to adjust the extract to the final volume required.

11.8.2. Nitrogen Evaporation (N-evap) to Final Concentration

11.8.2.1. Quantitatively transfer the extract into a calibrated container. Alternatively, place the concentrator tube in a warm water bath that is at least 5° C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the

surface of the solvent, but should not create splattering of the extract. Hexane boils at 69°C.

11.8.2.2. During the course of the evaporation rinse the sides of the evaporation tube or container twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 10 mL.

11.8.2.3. When the volume is slightly below 10 mL, remove the container/tube from the n-evap bath. Quantitatively transfer the extract into a calibrated container if the extract is still in the concentrator tube. Compare the volume to that of the calibrated reference, and carefully add solvent to make the final volume of 10 mL. Record the lot of the container that extracts are brought to final volume in.

Note: It is very important not to concentrate to dryness as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.8.3. Proceed to the cleanup steps.

11.9. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/or loss of detector sensitivity.

11.9.1. *Gel Permeation Chromatography* (SOP WS-OP-0012, Method 3640A) is a generally applicable technique which can be used to prepare extracts for semivolatiles (8270), and pesticides/PCB analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.

Note: Gel Permeation Chromatography is generally the first cleanup step.

11.9.2. *Florisil column cleanup* (Section 11.10, Method 3620B) is particularly useful for cleanup of pesticides for analysis by Methods 8081A/8081B and PCBs by Method 8082/8082A and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.

11.9.3. *Styrene Divinylbenzene polymer (SDB-L) sorbant cleanup* (Section 11.11). The SDB-L Styrene-Divinylbenzene polymer SPE tubes (Phenomenex) or DVB SPE tubes (Restek) may be helpful for cleanup of pesticides for analysis by Methods 8081A/8081B and PCBs by Method 8082/8082A. This cleanup

should be applied routinely to all extracted soil samples. Use on other matrices is also appropriate if co-extracted contamination is problematic at instrument analysis. This synthetic polymer is used to remove a variety of unwanted matrix-related compounds.

11.9.4. *Graphitized Carbon sorbent cleanup* (Section 11.12). The CarboPrep-90 (Restek) or EnviCarb (Supelco) pre-packed cartridges may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This clean-up is not applied routinely as there is approximately 50% recovery of TCX surrogate. Its use should be pre-approved by the department manager. This clean-up removes oils and humic substances.

11.9.5. *Sulfuric acid cleanup* (Section 11.13, Method 3665A) is applied to samples requiring analysis for 8082/8082A Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid. In pre-approved situations, this acid cleanup can be used for 8081A/8081B samples, however, there will be significant loss (or increase) of some compounds, namely: Aldrin, Dieldrin, Endrin, Endosulfan I, Endosulfan II, Endrin Aldehyde, Methoxychlor, Endrin Ketone, and Endosulfan Sulfate

**WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.**

11.9.6. *Sulfur cleanup* (Section 11.14, Method 3660A) is generally applied to samples for analysis by methods 8081A/8081B and 8082/8082A, since the Electron Capture Detector responds strongly to sulfur. It is performed as the last cleanup.

11.10. Method 3620B, Florisil Cartridge Cleanup

***Note:*** Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, Zymark Benchmate, or equivalent.

11.10.1. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.10.1.1. Place a valve liner (Supelco, P/N# 57059, disposable flow control valve liner, or equivalent) into each port of the vacuum manifold.

**WARNING: Use of vacuum systems creates a significant risk of implosion. Thoroughly inspect all glassware and do not use any that has been chipped, rubbed, cracked, or marred in any fashion.**

**WARNING: Ensure that the exhaust line from the vacuum pump is secured well inside of a fume hood so that it cannot fall out of the hood.**

11.10.2. Place one Florisil cartridge into the vacuum manifold for each sample extract.

11.10.3. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (1:1). Rinse with 5mL of hexane. Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.10.4. Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.10.5. Place a rack of clean labeled 16 mL test tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.10.6. After the clean tubes are in place, vacuum to the manifold is restored, add 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) of the extract to the appropriate Florisil cartridge. Archive the remaining extract.

11.10.7. The pesticides/Aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 16 mL test tube or held in the rack inside the vacuum manifold. Allow the solvent to drip through the sample without vacuum until drip stops. Then apply vacuum to extract remaining eluent.

11.10.8. Use nitrogen evaporation (Section 11.6.2) to concentrate the extract to the same amount that was used in 11.8.6. Afterward the sample extract can proceed to the subsequent clean-up steps in the following sections.

11.10.8.1. Florisil lot verification procedure – see Section 11.15.

11.11. Divinylbenzene Styrene Polymer Cartridge Cleanup

11.11.1. This clean-up should be conducted simultaneously with Florisil cleanup. Follow the same elution procedure as the Florisil cleanup from the previous section. The SDB (dimethylbenzene styrene) cartridge should be stacked on top of the Florisil cartridge referenced in Section 11.10.3.

11.11.2. Each lot should be verified for recovery of target analytes using the same procedure as that for Florisil ((Section 11.15). The evaluation of 2,4,5-TCP recovery is omitted for this clean up material.

11.12. Graphitized Carbon Cartridge Cleanup

11.12.1. This clean-up should be conducted after Florisil (if approved). Follow the same elution procedure as the Florisil cleanup from the previous section.

11.12.2. Each lot should be verified for recovery of target analytes using the same procedure as that for Florisil (Section 11.15). The evaluation of 2,4,5-TCP recovery is omitted for this clean up material.

11.13. Method 3665A, Sulfuric Acid Cleanup

11.13.1. Add approximately 2 mL of concentrated sulfuric acid to 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) sample extract in a Teflon capped vial.

**WARNING: There must be no water present in the extract or the reaction may shatter the sample container.**

11.13.2. Shake or vortex for about thirty seconds and allow it to settle. Centrifuge if necessary.

11.13.3. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial.

**CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.**

11.13.4. If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.13.5. Dispose of the sulfuric acid waste to the waste sulfuric acid drum for recycling.

11.13.6. Proceed with Section 11.13, sulfur removal.

11.14. Method 3660A, Sulfur Removal with Elemental Mercury.

If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

**Note:** Use mercury sparingly in order to minimize exposure and disposal costs.

11.14.1. Transfer 2.0 mL (1L separatory funnel extraction and 3550B/3550C) or 1.5 mL (LVI/RVE and MAE) of the sample extract into a clean concentrator tube or Teflon sealed vial.

11.14.2. Add one to three drops (about 0.25mL) of mercury to the extract vial and seal.

**WARNING: Do not use latex or vinyl gloves when working with mercury and organic solvents at the same time.**

11.14.3. Vortex for 15-30 seconds.

11.14.4. If black precipitate forms, sulfur was present. Centrifuge, then transfer the supernate to a clean test tube and repeat steps 11.14.2 and 11.14.3. Do this until relatively little precipitate remains. Document if samples require more than five sulfur cleanups. If precipitate is still present after 10 cleanups, re-start with a minimum 5X dilution.

**WARNING: Do NOT centrifuge glass tubes containing mercury. Due to the density of the mercury, the test tube could break in the centrifuge.**

11.14.5. If no precipitate is present, remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

11.14.6. Rotate the mercury waste out of the laboratory to the Hazardous Waste storage area for labpack disposal.

11.15. Clean-up Cartridge lot verification

11.15.1. Florisil lot verification procedure.

Each lot of Florisil cartridges must be evaluated before use, following this performance check procedure:

11.15.1.1. Combine the following in a clean test tube:

- 1.0mL of a 0.1µg/mL solution of 2,4,5-trichlorophenol solution (in hexane)
- 1.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.1.2. Add this mixture to a pre-washed Florisil cartridge. Per the instructions in Section 11.10, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 1.0mL and transfer to glass vials for instrument analysis.

11.15.1.3. Analyze the Florisil check solution and blank, along with an aliquot of the 0.1µg/mL TCP solution per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of peaks interfering with target compounds, and ≤5% TCP recovery (when peak area is compared between the Florisil check and the native TCP solution).

11.15.2. Styrene Divinylbenzene (SDB) polymer lot verification procedure.

Each lot of SDB cartridges must be evaluated before use, following this performance check procedure:

11.15.2.1. To a pre-washed SDB cartridge, add 2.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.2.2. Per the instructions in Section 11.11, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 2.0mL and transfer to glass vials for instrument analysis.

11.15.2.3. Analyze the SDB check and blank, per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of peaks interfering with target compounds.

11.15.3. Graphitized Carbon lot verification procedure.

Each lot of Graphitized Carbon cartridges must be evaluated before use, following this performance check procedure:

11.15.3.1. To a pre-washed Graphitized Carbon cartridge, add 2.0mL of the 8081 INDA/B Level-5 (Typical OC Pesticide mix @ 20-200µg/mL)

11.15.3.2. Per the instructions in 11.12, elute with 8mL hexane /acetone (90:10). For a blank sample, repeat this step on another cartridge loaded with 2mL of clean hexane. Concentrate the eluates to final volumes of 2.0mL and transfer to glass vials for instrument analysis.

11.15.3.3. Analyze the SDB check and blank, per method 8081. Expected recoveries are 80-120% of all pesticide analytes and surrogates with

the exception of Endrin Aldehyde, which cannot typically meet this criterion. At a minimum, the recoveries will meet the current LCS limits for waters. Additionally, there should be no indication of peaks interfering with target compounds.

## 12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

## 13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

## 14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability.) Employees must

abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracts, extract dilutions, all disposable materials (filters, disposable glassware, etc.) with concentrations of Aroclors greater than 50 ppm. These must receive special handling, shipping and disposal as PCB waste. Contact the Hazardous Waste Technician for specific instructions, as these cannot be disposed in normal waste streams.

15.2. Extracted aqueous/TCLP leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.3. Extracted soil samples, sodium sulfate, glass wool/filter paper, used florisil or other cleanup cartridges, and thimbles contaminated with methylene chloride and acetone from sonication and soxhlet extraction and from sample drying. Pour any excess liquid from the extracted soil samples as outlined below (Section 15.5). Dump these types of wastes into an orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the appropriate lab trash collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Used mercury contaminated with sulfur compounds from the sulfur cleanup. Pour the contaminated mercury into a 250-ml plastic bottle labeled for contaminated mercury. When full or after one year, whichever comes first, transfer this jar to the waste collection area for shipment.

15.5. Assorted flammable solvent and methylene chloride waste from soil extraction, various rinses or pre-elutions. Collect the waste solvents in tripours during use. Empty the tripours into a 1liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.6. Used sulfuric acid from the cleanup during 8082/PCB extraction. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

## 16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III

- Method 3500B, Organic Extraction and Sample Preparation, Revision 2, December 1996
- Method 3510C, Separatory Funnel Liquid Liquid Extraction, Revision 3, December 1996
- Method 3550B, Ultrasonic Extraction, Revision 2, December 1996
- Method 3580A, Waste Dilution, Revision 1, July 1992
- Method 3600C, Cleanup, Revision 3, December 1996
- Method 3620B, Florisil Cleanup, Revision 2, December 1996
- Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II

- Method 3660A, Sulfur Cleanup, Revision 1, July 1992

16.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV

- Method 3500C, Organic Extraction and Sample Preparation, Revision 3, December 2007
- Method 3546, Microwave Extraction, Revision 0, February 2007
- Method 3550C, Ultrasonic Extraction, Revision 3, February 2007

16.4. SOP WS-QA-0025, Glassware Handling Safety

## 17. METHOD MODIFICATIONS

17.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Aqueous sample volumes are determined by weight.

## 18. ATTACHMENTS

18.1. Table 1 – Exchange Solvents, Final Volumes, and Aliquot Volumes

18.2. Table 2 – Spiking Volumes

18.3. Table 3 – Spike Solution Components

18.4. Table 4 – Sacramento Method 8081/8082 Extraction Summary

## 19. REVISION HISTORY

19.1. WS-OP-0002, Revision 4.2, Effective 05/27/2016

19.1.1. Inserted Section 11.2.9 before 11.2.7 bo ensure samples are warmed and spiked prior to adjusting sample pH.

19.1.2. Table 2 – changed concentration of spiking volumes for 8081A / 8082 surrogates from 0.2 µg/mL to 0.8 µg/mL.

19.1.3. Section 11.8/.1.7 – inserted "For GPC cleanup…" in front of "when the apparent volume of the extract reaches…".

19.1.4. Table 3 – Changed concentration of surrogate spiking amount from 0.2 µg/mL to 0.8 µg/mL.

19.1.5. Editorial changes.

19.2. WS-OP-0002, Revision 4.1, Effective 04/02/2014

19.2.1. Section 2.1.1, revised to read: "This SOP has procedures for extracting both the traditional sample volume (1000mL) as well as reduced sample volumes (250 or 125 mL). The traditional volume is referred to as a "1 L separatory funnel extraction", and the smaller volumes are referred to as "250 mL Limited Volume Initiative/Reduced Volume Extraction" (250 mL LVI/RVE) and "125 mL Limited Volume Initiative/Reduced Volume Extraction (125 mL LVI/RVE) in this SOP."

19.2.2. Section 10.1, changed to read, "On the day of use, measure 2.0 mL of solvent into a 16mL culture tube using a 1.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Seal the culture tube – this is the 'check' or 'reference' vial for cleanup with Florisil. Using the same procedure, create a 10.00mL 'check' vial using a 5.00 mL gastight syringe or a serological pipet that is verified in accordance with SOP WS-QA-0004. Record the container lot, solvent, gastight syringe or serological pipet

ID, date, and initials of preparer for all check vials in a controlled logbook. The culture tubes containing the sample extracts are then compared against the "check" vial to ensure that the final volume is consistently within the criteria of 10 ± 0.3 or 2 ± 0.06 mL as required."

19.2.3. Section 10.2, changed the frequency of autopipetter verification to daily to reflect changes in WS-PQA-003.

19.2.4. Inserted Section 10.4, "Prior to use, ensure any containers used for quantititative steps (e.g final volume and Florisil) are verified as described in SOP WS-QA-0004."

19.2.5. Section 11.2.4.2, appended to the end of the section, "This must be documented in an NCM, as there may be a low bias to the analysis when the entire container is not use, and the container is therefore not rinsed and the rinsate combined with the extraction solvent."

19.2.6. Inserted Section 11.2.3, "One method for reducing the effects of emulsions is to swirl the funnel after allowing it to rest for the required amount of time, wait approximately a minute, and then drain the extract without collecting the emulsion. After the third (final) extraction and rest period, add a squirt of DCM to identify where the layer of DCM and water is separated and collect only the DCM. Alternatively, on the final pour collect the emulsion and use a second filtration step at KD with glass wool and pipet off excess water."

19.2.7. Appended to the end of the note following Section 11.2.1.4, "Similarly, it is anticipated that only 20 of the 30 mL used (or 10 of 15 mL used) may be recovered on the first shake when extracting smaller water volumes."

19.2.8. Appended to the end of the note following Section 11.2.1.4, "Ensure to minimize the amount of water collected."

19.2.9. Section 11.8.1.11 changed to read, "Remove the KD apparatus from the water bath and allow it to cool and drain. If the level of the extract is above the level of the concentrator tube joint, add a new boiling chip and continue to distill the solvent as necessary. If redistilling, remove the KD apparatus from the water bath and allow it to cool and drain before proceeding to the next step.

19.2.10. Inserted Section 11.8.1.12 to read, "Remove the Snyder column and rinse the inside of the top joint of the K-D flask with a minimum amount of DCM, rotating the flask as necessary to ensure the rinsate covers the sides of the KD flask.

19.2.11. Section 11.8.2.1, inserted at the beginning, "Quantitatively transfer the extract into a calibrated container."

19.2.12. Section 11.8.2.1, changed the second sentence to start, “Alternatively, place the concentrator tube in a warm water bath that…”

19.2.13. Section 11.8.2.3 changed to read, “When the volume is slightly below 10 mL, remove the container/tube from the n-evap bath. Quantitatively transfer the extract into a calibrated container if the extract is still in the concentrator tube. Compare the volume to that of the calibrated reference, and carefully add solvent to make the final volume of 10 mL. Record the lot of the container that extracts are brought to final volume in.”

19.2.14. Added references for both 125 mL and 250 mL extraction options under Method 3510C (Separatory Funnel Extraction. Sections updated include: 9.5.1, 11.2.3, 11.2.4.2, 11.2.10, 11.2.1.5, Table 2 (Spiking Volumes), and Table 4.

19.2.15. Tables 2 and 4, the concentration of the technical chlordane standard was changed from 10 ug/mL to 1.0 ug/mL to permit spiking at a volume consistent with SW846 recommendations.

19.3. WS-OP-0002, Revision 4.0, Effective 7/3/2013

19.3.1. Added warning regarding TSCA requirements for PCB standards at 1000 ppm (Sections 5 and 15)

19.3.2. Removed references to and procedures for continuous liquid-liquid extraction – Section 2 (scope) as well as Section 11 (procedure).

19.3.3. Added references to and procedures for microwave assisted extraction (Method 3546) – Section 2 (Scope) as well as Section 11 (procedure)

19.3.4. Added references to and procedures for Method 3550C – Section 2 (Scope) as well as Section 11 (procedure)

19.3.5. Removed figure 1 (CLLE set-up) as CLLE has been removed.

19.3.6. Added references to SW-846 Update IV methods.

19.3.7. Editorial revisions

19.4. WS-OP-0002, Revision 3.9, Effective 05/10/2013

19.4.1. Updated SOP to conform to current laboratory practices.

19.4.2. Editorial changes.

19.5. WS-OP-0002, Revision 3.8, Effective 04/06/2012

19.5.1. Editorial changes

19.6. WS-OP-0002, Revision 3.7, Effective 07/27/2011

19.6.1. Inserted into Section 11.3.9 and 11.4.6, In pre-approved situations, other solvents (such as Hexane or DCM only) may be used to control co-extracted matrix interference).

19.6.2. Inserted Section 11.7.3, *Styrene Divinylbenzene polymer (SDB-L) sorbant cleanup* (Section 11.10). The SDB-L Styrene-Divinylbenzene polymer SPE tubes (Phenomenex) or DVB SPE tubes (Restek) may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This cleanup should be applied routinely to all extracted soil samples. Use on other matrices is also appropriate if co-extracted contamination is problematic at instrument analysis. This synthetic polymer is used to remove a variety of unwanted matrix-related compounds.

19.6.3. Inserted Section 11.7.4, *Graphitized Carbon sorbant cleanup* (Section 11.11). The CarboPrep-90 (Restek) or EnviCarb (Supelco) pre-packed cartridges may be helpful for cleanup of pesticides for analysis by Method 8081A and PCBs by Method 8082. This clean-up is not applied routinely as there is approximately 50% recovery of TCX surrogate. Its use should be pre-approved by the department manager. This clean-up removes oils and humic substances.

19.6.4. Added to Section 11.7.5, In pre-approved situations, this acid cleanup can be used for 8081 samples, however, there will be significant loss (or increase) of some compounds, namely: Aldrin, Dieldrin, Endrin, Endosulfan I, Endosulfan II, Endrin Aldehyde, Methoxychlor, Endrin Ketone, and Endosulfan Sulfate

19.6.5. Inserted Section 11.9, Divinylbenzene Styrene Polymer Cartridge Cleanup

19.6.6. Inserted Section 11.10, Graphatized Carbon Cartridge Cleanup

19.6.7. Updated Table entries.

19.6.8. Inserted Appendix 1.

19.6.9. Editorial revisions.

19.7. WS-OP-0002, Revision 3.6 Effective 06/10/2011

19.7.1. Inserted Section 11.4, Method 3540C Extraction of Solids with Soxhlet Apparatus.

and concentrate the extract to ~5mLs, as described in the previous sections, raising the temperature of the water bath, if necessary, to maintain proper distillation

19.6.6. Inserted new Section 11.6.1.10: Remove the KD apparatus from the water bath and allow it to drain and cool. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If the level of the extract is above the level of the concentrator joint, continue to distill the solvent as necessary. Adjust the extract to 10.0 mL with the solvent last used.

Note: When performing KD on the GPC portion of the extract, bring to a final volume of 5 mL (as the 10 mL extract was split in the previous Section).

19.6.7. Inserted new Section 11.6.1.11. If further concentration is indicated in Table 1, either the micro Snyder column technique or nitrogen evaporation (Section 11.6.2) technique is used to adjust the extract to the final volume required

19.6.8. Editorial revisions.

| Table 1 | | | | | |
|---|---|---|---|---|---|
| **Exchange Solvents, Final Volumes, and Aliquot amounts** | | | | | |
| Type | Exchange Solvent for GPC | Final Volume for GPC | Exchange Solvent for all other clean-ups | Final Volume for LVI/RVE and MAE | Final Volume for 1L separatory funnel and 3550B |
| No Cleanup | -- | -- | Hexane | 5mL | 10mL |
| GPC Only | DCM | 5 mL[1] | -- | 5mL | 5mL |
| Cartridge clean-up (e.g. Florisil) | -- | -- | Hexane | 5mL - 1.5mL aliquot used | 10mL - 2mL aliquot used |

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

***Note:*** Different final volumes may be necessary to meet special client reporting limit requirements.

| Table 2 | | |
|---|---|---|
| **Spiking Volumes** | | |
| **Solution** | **Solution ID** | **Volume (mL)**<br>**1L Sep / LVI / 3550 /3546 / Waste** |
| Surrogate (8081A, 8082) | 0.2 ug/mL DCB/TCX 3/90 P-Surr | 1.0 / 0.5/ 2.0 / 1.0/ 2.0 |
| LCS (8081A) | 1.0ug/mL Pest Full Spike | 0.5 / 0.2 / 0.5 / 0.25 / 0.5 |
| | 5ug/mLToxaphene (LCS only) | 1.0 / 0.5 / 1.0 / 0.5 / 1.0 |
| | 1 ug/mL TChlordane (LCS only) | 0.5 / 0.5 / 1.0 / 0.50 / 1 |
| LCS (8082) | 2ug/mL PCB Spike Aroclor 1660 | 1.0 / 0.5 / 1.0 / 0.5 / 1.0 |

| Table 3 | | | |
|---|---|---|---|
| **Spike Solution Components** | | | |
| **Solution** | **Solvent** | **Compounds** | **Conc. (µg/mL)** |
| Surrogate | Acetone | Decachlorobiphenyl<br>Tetrachloro-m-xylene | 0.2<br>0.2 |
| Pest Full (8081A) | Acetone | alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Aldrin, Heptachlor, Heptachlor Epoxide, Endosulfan I, Endosulfan II, Endosulfan sulfate, alpha-Chlordane, gamma-Chlordane, Dieldrin, 4,4'-DDE, 4,4'-DDD, 4,4'-DDT, Endrin, Endrin ketone, Endrin aldehyde, Methoxychlor | 1ug/mL (for all analytes) |
| Toxaphene | Acetone | Toxaphene | 5 |
| TChlor | Acetone | Technical Chlordane | 1 |
| PCB 1660 (8082) | Acetone | Aroclor 1016<br>Aroclor 1260 | 2.0<br>2.0 |

| Table 4 | | | | | | | |
|---|---|---|---|---|---|---|---|
| **West Sacramento Method 8081/8082 Extraction Summary** | | | | | | | |
| | Sample Amount | Surrogate | LCS1 | Extract Solvent | Extract Solvent Volume | Extraction Time | Comments |
| 1 L separatory funnel extraction | 1 L | 1.0 mL | See table 3 | DCM | 3 x 60 mL | 2 min shake, 10 min rest | |
| 250 mL LVI/RVE | 250 mL | 0.5 mL | See table 3 | | 3 x 30 mL | | |
| 125 mL LVI/RVE | 125 mL | 0.5 mL | See table 3 | | 3 x 15 mL | | |
| Leachate (SEPF) | 200 mL | 1.0 mL | See table 3 | | 3 x 60 mL | | Make the 200mL to 1L with DI water before extracting |
| Solid (Sonication) | 30 g | 2.0 mL | See table 3 | 1:1 DCM /Ace | 3 x 100mL | 3min | |
| Waste Dilution | 1 g | 2.0 mL | See table 3 | Hex | 10mL | NA | Don't concentrate. |
| Biologics (Sonication) | 10 g | 20 mL | See table 3 | 1:1 DCM /Ace | 3 x 100mL | 3 min | |
| Solid (Microwave) | 15g | 1.0mL | See table 3 | 1:1 Hex /Ace | 25mL | 25min | |

Acid Digestion of Soils, SW-846 Method 3050B [Method EPA 3050B]

# Title: Acid Digestion of Soils, SW-846 Method 3050B [Method EPA 3050B]

Reviewed 03/07/2013 Reviewed 04/14/2014
Reviewed 07/27/2015

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## 1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soils, sediments, wastes, and other miscellaneous solid samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma Atomic Emission Mass Spectroscopy (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS for the elements listed in Tables I and II. Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve most all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment

## 2. SUMMARY OF METHOD

A representative 1 g to 2 g (wet weight) portion of sample is digested in nitric acid, hydrogen peroxide, and hydrochloric acid. The extractions are then filtered and diluted to 100 mL.

## 3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

## 4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3. Boron from borosilicate glassware will leach into the sample solution during and following sample processing. For critical low level determinations of boron only quartz and/or plastic lab ware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

4.8. Available silica is lost during the preparation and therefore silica is not analyzed in solid matrices.

## 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Opening, closing, filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic stretch break, or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustments when there are numerous samples to be processed. There are numerous wrenches and tools available to reduce the impact of opening large numbers of sample containers or digestion tubes.

5.1.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.3. The acidification of samples containing reactive materials may result in the release of toxic gasses, such as cyanides or sulfides. Acidification of samples

must be done in a fume hood.

5.1.4. When digesting samples on a hot plate or digestion block, heat protective gloves and/or hot tongs must be used when handling containers. Ensure that digestion containers are arranged safely and are not overcrowded.

5.1.5. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or over safety goggles during this process.

5.1.6. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Vinyl and nitrile gloves all provide satisfactory protection against the reagents used in this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.7. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred, prepared, digested and cooled in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.8. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Hydrochloric Acid | Corrosive Poison | 5 ppm-Ceiling | Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| Hydrogen Peroxide | Oxidizer Corrosive | 1 ppm-TWA | Vapors are corrosive and very irritating to the respiratory tract, eyes and skin. |
| Nitric Acid | Corrosive Oxidizer Poison | 2 ppm-TWA 4 ppm-STEL | Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

## 6. EQUIPMENT AND SUPPLIES

6.1. Hot block digestion unit capable of maintaining a temperature of 90-95°C.

6.2. Digestion tubes: graduated volume relative to type of hot block.

6.3. Thermometer that covers a temperature range of 0-200°C.

6.4. Whatman No. 41 filter paper.

6.5. Wooden tongue depressors or stainless steel spatulas.

6.6. Top-loading balance capable of accurately weighing to the nearest 0.01 g.

6.7. Bottle-top dispensers.

6.8. Calibrated adjustable air displacement pipettes.

6.9. 4 oz graduated snap cap containers for final transfer of digestions.

6.10. Teflon® boiling chips.

## 7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as certified custom TestAmerica solutions (Tables I, II). All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. All stock standards must be labeled with the date of receipt and the date the standard is opened.

7.4. The LCS and MS samples must contain all the elements designated for analysis. If a non-routine element is required that is not contained in the custom TestAmerica solution, the lab must purchase a solution from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid ($HNO_3$), concentrated, trace metal grade or better.

7.6. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.7. 1:1 Hydrocloric acid: add 500 mL of concentrated HCl to 500 mL of reagent water.

7.8. 30% Hydrogen peroxide ($H_2O_2$), reagent grade.

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil samples do not require preservation but must be stored at 4 °C ± 2 °C until the time of analysis.

## 9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A

batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples. A method blank consists of boiling chips processed through all of the steps, and at the same time as the associated samples. If a method blank exceeds +/- the reporting limit for a given analyte than the samples associated with that batch must be re-prepared. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. For DOD projects, the MB criteria is less than ½ the reporting limit. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. A LCS consists of boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared except when a LCS exhibits high recovery. For such a case, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair as required by client or regulatory requirements. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details

9.6. Quality Assurance Summaries: certain clients may require specific project or program QA information that supersedes the SOP requirements. Quality Assurance Summaries (QAS) should be developed by the PM's to address these requirements. Sample preparation analysts are required to read and print each QAS for every project they

prepare. These are transferred to the instrument analysts with the sample digestions.

## 10. CALIBRATION

10.1. A digestion vessel filled with reagent water is monitored for temperature for each digestion block. The analyst is to monitor this temperature for accuracy throughout the digestion and record the initial and final temperatures on the sample digestion log.

10.2. All air displacement pipettes must be calibrated over their range of use at least monthly. If the analyst suspects a spiking volume problem, calibration may be required more often. See SOP WS-QA-0004 for pipettor calibration verification procedures.

10.3. All volumetric digestion vessels must be calibrated according to the frequency and procedures outlined in SOP WS-QA-0004. If the analyst suspects a vessel's reliability, calibration may be required more often.

## 11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to aliquotting them into the digestion vessels and before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludge and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the

matrix designation. If the sample is logged in as solid but it appears more like a liquid, contact the department manager or project manager for further instructions.

11.6. Always read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. Preparation of Soils, Sediments and Sludges for Analysis by ICP and ICP/MS.

11.8.1. SOP WS-QA-0018 must be followed to properly homogenize and aliquot solid samples.

11.8.2. Weigh out a 1.00 – 2.00 g sample aliquot into a graduated hot block tube using a wooden tongue depressor.

11.8.2.1. A 1.00 g +/- 5% aliquot is normal for samples that show no apparent free liquid phase or glistening from an aqueous phase.

11.8.2.2. A 1.4 g – 1.5 g aliquot should be used for samples with a muddy, glistening texture or small amount of free liquid.

11.8.2.3. A 2.0 g +/- 5% aliquot should be used for samples with a notable free liquid phase that does not mix into a structured muddy texture.

11.8.3. When sub-sampling for the MS/MSD sample aliquots, first transfer the sample per section 11.9.1 into a 50 mL polystyrene beaker and mix thoroughly before weighing the three replicates into the graduated hot block tubes.

**WARNING: Opening, closing, filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic stretch break, or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustments when there are numerous samples to be processed. There are numerous wrenches and tools available to reduce the impact of opening large numbers of sample containers or digestion tubes.**

11.8.4. Weigh out approximately 1.00g of Teflon® boiling chips into digestion tubes for MB and LCS/LCSD. For ICPMS, spike 0.2 mL of spiking solution into

the LCS/MS/MSD. For ICP, use 1.0 mL of spiking solution. Tables I and II show the appropriate solutions to use and their concentrations. The final spiking levels are also listed.

11.8.5. Add 5.0 mL of reagent water to all of the digestion vessels.

11.8.6. Add 5.0 mL of concentrated $HNO_3$ and heat the samples to 90-92ºC and reflux for 10 minutes without boiling. For ICPMS only, add 5.0 mL of 1:1 HCl prior to heating.

11.8.7. Heat the samples to 90-92ºC and reflux for 10 minutes without boiling. Allow the samples to cool.

*Note: Do not allow samples to boil or go dry during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.*

11.8.8. Add 5.0 mL of concentrated $HNO_3$ for both ICP and ICPMS.

11.8.9. Reflux at 90-92°C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.8.10. If brown fumes are observed, add concentrated $HNO_3$ in 5.0 mL aliquot with 30 minute refluxing cycles at 90-92º until no more fumes are observed.

11.8.11. Allow the sample to evaporate to approximately 5 mL while ensuring that no portion of the bottom of the tube is allowed to go dry. Do not heat the samples for more than 2 hrs. Allow the samples to cool.

11.8.12. Add 2 mL of reagent water and 3 mL of 30 % $H_2O_2$. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

*Note: If the samples are very dark or look organic in nature, add the 30% $H_2O_2$ in 1.0 mL aliquots with short heating cycles to assure sample is not lost due to excessive effervescence.*

11.8.13. Heat the samples for 15 minutes. Allow the samples to cool.

11.8.14. Add 3.0 mL $H_2O_2$ and continue heating at 90-92ºC until the volume is reduced to approximately 5 mL. Do not exceed 2.0 hrs.

11.8.15. If the samples are being prepared for ICP analyses, add 5.0 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

11.8.16. If the samples are being prepared for ICPMS, add 5.0 mL of 1:1 HCl and reflux for an additional 15 minutes without boiling.

11.8.17. Allow the samples to cool and filter through Whitman #41 filter paper directly

into 4 oz graduated snap cap containers. Dilute to a final volume of 100 ml with reagent water.

11.9. Filtration procedure:

11.9.1. Carefully pour the contents of each of the hot block digestion tubes into folded filter paper on top of the 4 oz containers. Be careful not to lose any of the contents through splashing.

11.9.2. Allow the contents to flow through the filter paper. Rinse each of the hot block tubes twice through the filter paper with reagent water.

11.9.3. Using reagent water, rinse each of the filter papers three times allowing the contents to filter thoroughly between rinses.

11.9.4. Adjust the final volume of the digestates to 100 mL with reagent water.

## 12. CALCULATIONS/DATA REDUCTION

Not Applicable.

## 13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each

analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

## 14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic waste generated by the digestion and unused acidic digestate containing nitric and hydrochloric acid. This acidic liquid is consolidated into a plastic acidic waste drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

## 16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

16.2. WS-PQA-003: Quality Control Program

16.3. WS-QA-0004: Calibration of Autopippetors, Autodispensers, and Volumetric Containers

16.4. WS-QA-0018: Subsampling

## 17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. Watch glasses are not used with the digestion blocks.

17.3. Samples are not centrifuged, but always filtered with Whatman #41 filter paper.

17.4. Samples are not sieved prior to weighing unless special instructions are given.

17.5. 5.0 mL of 1:1 HCl is added to the ICPMS digestions during the initial addition of reagents as studies have shown this increases the performance of silver analysis.

17.6. Temperature conditions of 95ºC ± 5ºC have been modified to 90-92ºC due to loss of silver at higher temperatures.

17.7. During the second step of $H_2O_2$ addition, 3.0 mL is added to all standards and samples instead of the steady additions of 1.0 mL aliquots.

17.8. For the final addition of HCl to the ICP aliquots, 5.0 mL of concentrated HCl is added instead of 10 mL.

17.9. 5.0 mL of 1:1 HCl is added to the final preparation phase for ICPMS to increase the performance of silver.

## 18. ATTACHMENTS

18.1. Figure 1 — Method 3050B (Modified) Flowchart

18.2. Table 1 — Analyte List Certified Under NELAC Criteria: Method 6010B

18.3. Table 2 — Analyte List Certified Under NELAC Criteria: Method 6020

18.4. Appendix 1 — Contamination Control Guidelines

## 19. REVISION HISTORY

19.1. WS-IP-0002, Revision 5.3, Effective 01/19/2012

19.1.1. Section 6.10: “Teflon boiling chips”.

19.1.2. Section 11.8.4: added “Weigh out approximately 1.00g of Teflon® boiling chips into digestion tubes for MB and LCS/LCSD.”

19.2. WS-IP-0002, Revision 5.2, Effective 12/17/2010

19.2.1. Section 11.8.4: Changed ICPMS spiking level from 2.0 mL to 0.2 mL.

19.2.2. Updated Table II Source and Initial Conc. Levels for all analytes.

19.3. WS-IP-0002, Revision 5.1, Effective 10/19/2009

19.3.1. Revise Section 11.8.4 to read, "For ICPMS, spike 2.0 mL of spiking solution into the LCS/MS/MSD. For ICP, use 1.0 mL of spiking solution. Tables I and II show the appropriate solutions to use and their concentrations. The final spiking levels are also listed."

19.3.2. Revise Section 11.8.5 to read, "Add 5.0 mL of reagent water to all of the digestion vessels."

19.3.3. Add Section 11.9.4 to read, "Adjust the final volume of the digestates to 100 mL with reagent water."

19.3.4. Revised the flow chart (Figure 1) to reflect the changes above.

19.4. WS-IP-0002, Revision 5, Effective 09/30/2008

19.4.1. Updated to current TestAmerica format.

19.4.2. Added HCl to last acid addition for ICPMS analysis.

19.4.3. Change volumes to approximately 5 mL from 5-10 mL for reduction steps.

19.5. WS-IP-0002, Rev. 4, Effective 9/11/07

19.5.1. The SOP format was updated to TestAmerica format.

19.5.2. Reference to Silica was removed.

**Figure 1: Method 3050B (modified) Flowchart**

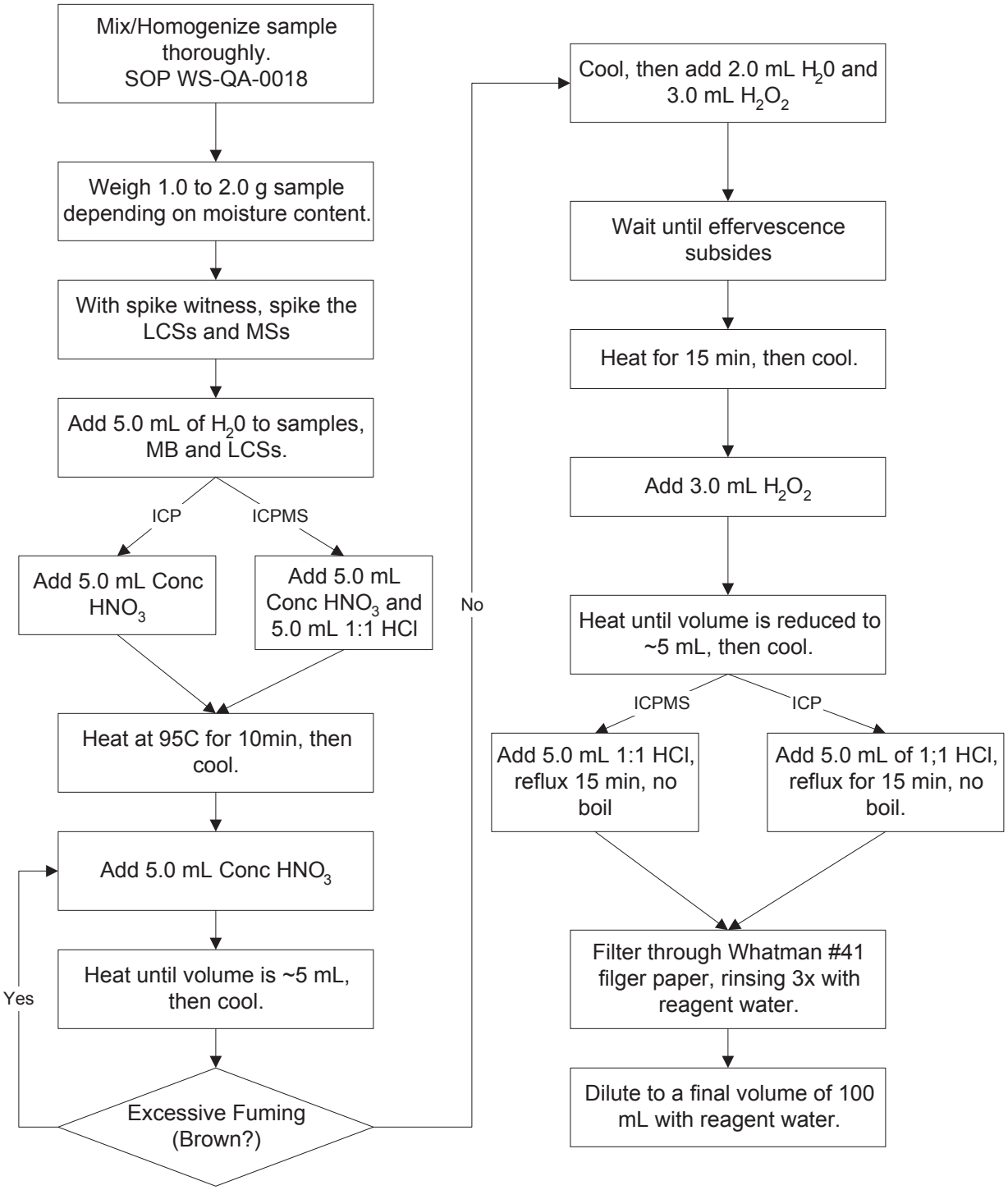

**TABLE I**
**Analyte List Certified Under NELAC Criteria: Method 6010B**

| Element | | LCS/MS/SD Spiking | | Source |
|---|---|---|---|---|
| | | Initial Conc. mg/L | Final Conc. mg/Kg | |
| Aluminum | Al | 200 | 200 | Spex XCAL-36 |
| Antimony | Sb | 50 | 50 | Spex XCAL-36 |
| Arsenic | As | 200 | 200 | Spex XCAL-36 |
| Barium | Ba | 200 | 200 | Spex XCAL-36 |
| Beryllium | Be | 5.0 | 5.0 | Spex XCAL-36 |
| Boron | B | 100 | 100 | Spex XCAL-38 |
| Cadmium | Cd | 5.0 | 5.0 | Spex XCAL-36 |
| Calcium | Ca | 5000 | 5000 | Spex XCAL-36 |
| Chromium | Cr | 20 | 20 | Spex XCAL-36 |
| Cobalt | Co | 50 | 50 | Spex XCAL-36 |
| Copper | Cu | 25 | 25 | Spex XCAL-36 |
| Iron | Fe | 100 | 100 | Spex XCAL-36 |
| Lead | Pb | 50 | 50 | Spex XCAL-36 |
| Lithium | Li | 100 | 100 | Spex XCAL-38 |
| Magnesium | Mg | 5000 | 5000 | Spex XCAL-36 |
| Manganese | Mn | 50 | 50 | Spex XCAL-36 |
| Molybdenum | Mo | 100 | 100 | Spex XCAL-36 |
| Nickel | Ni | 50 | 50 | Spex XCAL-36 |
| Phosphorous | P | 1000 | 1000 | Spex XCAL-38 |
| Potassium | K | 5000 | 5000 | Spex XCAL-38 |
| Selenium | Se | 200 | 200 | Spex XCAL-36 |
| Silver | Ag | 5 | 5 | Spex XCAL-36 |
| Sodium | Na | 5000 | 5000 | Spex XCAL-38 |
| Strontium | Sr | 100 | 100 | Spex XCAL-38 |
| Sulfur | S | 1000 | 1000 | Spex XCAL-38 |
| Thallium | Tl | 200 | 200 | Spex XCAL-36 |
| Tin | Sn | 200 | 200 | Spex XCAL-36 |
| Titanium | Ti | 100 | 100 | Spex XCAL-36 |
| Vanadium | V | 50 | 50 | Spex XCAL-36 |
| Zinc | Zn | 50 | 50 | Spex XCAL-36 |

**Note:** Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

**TABLE II**
**Analyte List Certified Under NELAC Criteria: Method 6020**

| Element | | Symbol | | Source |
|---|---|---|---|---|
| | | Initial Conc. mg/L | Final Conc. mg/L | |
| Aluminum | Al | 500 | 1.0 | TACA-1 |
| Antimony | Sb | 100 | 0.05 | TACA-2 |
| Arsenic | As | 100 | 0.2 | TACA-1 |
| Barium | Ba | 100 | 0.2 | TACA-1 |
| Beryllium | Be | 100 | 0.2 | TACA-1 |
| Boron | B | 100 | 1.0 | TACA-1 |
| Cadmium | Cd | 100 | 0.2 | TACA-1 |
| Calcium | Ca | 500 | 1.0 | TACA-1 |
| Chromium | Cr | 100 | 0.2 | TACA-1 |
| Cobalt | Co | 100 | 0.2 | TACA-1 |
| Copper | Cu | 100 | 0.2 | TACA-1 |
| Iron | Fe | 500 | 1.0 | TACA-1 |
| Lead | Pb | 100 | 0.2 | TACA-1 |
| Lithium | Li | 100 | 0.2 | TACA-1 |
| Magnesium | Mg | 500 | 1.0 | TACA-1 |
| Manganese | Mn | 100 | 0.2 | TACA-1 |
| Molybdenum | Mo | 100 | 0.2 | TACA-2 |
| Nickel | Ni | 100 | 0.2 | TACA-1 |
| Phosphorous | P | 500 | 1.0 | TACA-1 |
| Potassium | K | 500 | 1.0 | TACA-1 |
| Selenium | Se | 100 | 0.2 | TACA-1 |
| Silver | Ag | 25 | 0 .05 | TACA-1 |
| Sodium | Na | 500 | 1.0 | TACA-1 |
| Strontium | Sr | 100 | 0.2 | TACA-1 |
| Thallium | Tl | 25 | 0.05 | TACA-1 |
| Tin | Sn | 100 | 0.2 | TACA-2 |
| Titanium | Ti | 100 | 0.2 | TACA-2 |
| Uranium | U | 100 | 0.2 | TACA-1 |
| Vanadium | V | 100 | 0.2 | TACA-1 |
| Zinc | Zn | 100 | 0.2 | TACA-1 |

**Note:** Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

# Appendix I

## CONTAMINATION CONTROL GUIDELINES

**The following procedures are strongly recommended to prevent contamination**:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Auto sampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

**The following are helpful hints in the identification of the source of contaminants**:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

# Chromatographic Analysis Based on SW-846 Methods 8000B/8000C, 8082/8082A, Method 608, and Compendium Methods TO-4A, and TO-10A

[Methods 8000B, 8000C, 8082, 8082A, 608, TO-4A, TO-10A]

# Title: Chromatographic Analysis Based on SW-846 Methods 8000B/8000C, 8082/8082A, Method 608, and Compendium Methods TO-4A, and TO-10A [Methods 8000B, 8000C, 8082, 8082A, 608, TO-4A, TO-10A]

**Approvals (Signature/Date):**

| | |
|---|---|
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| 3/23/16 | 3.24.16 |
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## 1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used when SW-846 Method 8000B or Method 8000C is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This SOP is to be applied when SW-8/46 Methods 8082 or 8082A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOP, WS-OP-0002. The PCBs are determined and quantitated as Aroclor mixtures.

This procedure is also applicable to the determination of PCBs in municipal and industrial discharges under 40 CFR 136.1.

1.2. Table 1 lists Aroclor mixtures which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. Matrix interferences may result in higher RLs than those listed.

**Note:** This SOP does not include directions for congener specific analysis

1.3. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

## 2. SUMMARY OF METHOD

2.1. In general, aqueous samples are prepared for analysis using separatory funnel liquid-liquid extraction, and semivolatile analytes in solid samples are prepared using sonication or microwave assisted extraction.

2.2. This SOP presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the internal and external standard methods .

## 3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

## 4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Sulfur will interfere and can be removed using procedures described in SOP WS-OP0002.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, as outlined in the preparation SOP WS-OP-0002.

## 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The PCB solutions used to prepare standards for this method may arrive at concentrations as high as 1,000 ppm. Exercise extreme caution when handling these high concentration standards.

5.1.2. If any PCB standards are spilled inside or outside of a fume hood at concentrations equal to or greater than 5 PPM, all materials in contact must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS Coordinator for specific disposal instructions <u>before</u> cleaning up the spill.

5.1.3. If any PCB standards are spilled outside the fume hood at concentrations greater than 200 ppm, the Emergency Response Team must be activated for special clean-up procedures, and possibly additional testing of the spill location and additional regulatory notifications.

5.1.4. All glassware and lab trash in contact with any PCB's at concentrations equal to or greater than 5 PPM must be treated as PCB waste in California. Contact the Hazardous Materials Specialist or EHS

Coordinator for specific disposal instructions before disposing of this waste.

5.1.5. The autovials used on the instrument contain a small amount of solvent after the analysis is completed, which can evaporate into the air through the puncture in the septa. These autovials may be collected in an open tripour or similar cup at the instrument while the analyst is removing them from the tray, but once that is complete the vials must be transferred to a waste container in a fume hood. They cannot be left out on the bench by the instrument.

5.1.6. Ensure that the outlet lines from the instrument are secured and lead into either a fume hood or inlet point to the fume hood exhaust system.

5.1.7. All $^{63}$Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.1.8. All $^{63}$Ni sources shall be inventoried every six months. If a detector is missing the EH&S coordinator shall be immediately notified and a letter sent to the NRC or local state agency.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with open sample extracts, spikes, surrogates or solvents. Latex or vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should only be used when working with closed extract vials.

5.1.10. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Hexane | Flammable<br>Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

## 6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM, Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2. Gas Chromatograph - An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. A data system capable of measuring peak area and/or height is required. The GC injection port must be designed for capillary columns.

6.3. This laboratory utilizes an Agilent 6890 gas chromatograph with ChemStation D.02.00.275 operating system or equivalent and Chrom Peak Review version 2.1 data acquisition system or equivalent.

6.4. A $^{63}$Ni electron capture detector is required.

6.5. Refer to Table 2 for analytical columns.

6.6. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

## 7. REAGENTS AND STANDARDS

All reagents must be ACS reagent grade or better unless otherwise specified.

7.1. Standards

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is supplied in a sealed ampule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.1.2. Internal Standard: Internal standard is added to all samples, standards, and blank analyses. Internal standard is prepared at a concentration of 20 mg/L in hexane.

7.1.3. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standard solutions are stored at ≤ 6ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using. Stock standard solutions must be replaced after one year.

7.1.4. Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at ≤ 6ºC and protected from light. The standards must be replaced at least every six months or sooner if comparisons with check standards indicates a problem.

7.1.5. Refer to Table 3 for details of calibration standards.

7.2. Gases for carrier and make-up: Hydrogen, Helium, and Nitrogen.

7.3. Solvent for dilutions (if needed) and syringe cleaning: Hexane, pesticide grade.

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Extracts must be refrigerated at ≤ 6ºC and analyzed within 40 days of the end of the extraction.

## 9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser

requirements are previously agreed to with the client. The minimum initial demonstration required is analysis of a single point calibration.

9.2. Quality Control Batch

9.2.1. The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. Method 608 requires an LCS frequency equivalent to 10% of all samples analyzed.

9.2.2. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible. QC samples should be analyzed with the field samples, however, field samples may be split between instruments or sequences as needed.

9.2.3. If a QC batch is analyzed on more than one instrument, at the minimum an instrument blank must be analyzed on the second instrument to demonstrate that the system is free of interferences.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions. Surrogate and matrix spike recoveries will be reported in dilutions greater than 5x will be qualified.

9.3.2. All surrogate, LCS, and MS recoveries must be entered into TALS or other database so that accurate historical control limits can be generated.

9.4. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if batch precision data is required by the client or program. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.5. Surrogates

Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference or other evidence of matrix impacts.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect

9.5.1. While samples may be spiked with both TCX and DCB as surrogates, only DCB is used for control purposes. If DCB does not meet the recovery criteria, corrective action must occur.

9.5.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12) unless one column is out of control, in which case the in-control result is reported. For DOD projects, report the surrogate value from the primary column, rather than the higher value.

9.5.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.5.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.6. Method Blanks

One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples, and sodium sulfate for solid samples (refer to the appropriate preparation SOP for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit or at or above 10% of the

measured concentration of that analyte in the associated samples, whichever is higher.

Certain programs may require a more stringent evaluation of the method blank. For instance, DOD requires that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.
- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.6.1. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.7. Laboratory Control Samples (LCS)

9.7.1. For each batch of samples, analyze a LCS. At a minimum, the LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes if so required by the client or program governing the samples, such as DOD. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur.

9.7.2. If dual column analysis is used the choice of which result to report is described in Section 12. For DOD projects, report the value from the primary column, rather than the higher value.

9.7.3. Generally, Aroclors 1016 and 1260 are spiked for the LCS and MS/MSD. Other Aroclors may be spiked as requires by regulatory programs or client requirements. Spiking levels are described in Table 4.

9.7.4. Each Aroclor must be spiked and its recovery measured in at least 2 LCS over a 2 year time period.

9.7.5. LCS recovery for Method 8082/8082A limits and RPD's are set by in-house studies (see WS-PQA-003). The recovery limits for methods TO-4A and TO10A are given in Table 6.

9.7.6. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations
- Check instrument performance
- Reanalyze the LCS, and if still outside of control limits
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.7.7. The analyst should evaluate the anomalous analyte recovery for possible trends, as described in the QC Program document (WS-PQA-003). Control charts will be used to track trends and sporadic marginal exceedances.

9.7.8. Data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples.
- All target requested analytes are within control, but other LCS compounds are out of control.

9.7.9. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.7.10. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.7.11. If the batch is re-extracted past sample holding time, both sets of results should be reported, flagged and narrated.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits, as described in the QC Program document (WS-PQA-003). See Table 6 for TO-4A and TO-10A limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.

- If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed.
- The MS/MSD must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is described in Section 12. For QSM projects, report the value from the primary column, rather than the higher value.

9.8.2. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied. This NCM must be included in the final report.

9.8.3. For Method 608, the spike concentration for each parameter is found in Table 7. Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria. If the spike concentration was lower than the concentration in Table 7, the acceptance criteria must still be met or use the optional acceptance criteria in Table 8. .

9.8.3.1. If the recovery (P) is outside the acceptance criteria in Table 7 a QC check standard (QCCS / LCS) is analyzed and the percent recovery (Ps) is compared to Table 7. Analytes that fail in the matrix spikes and QCCS / LCS) are out of control and can not be reported for regulatory compliance purposes.

9.9. Internal Standards

Internal Standards (IS) are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard that is analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard response is monitored to verify that instrument performance is acceptable Criteria for standards are delineated in Section 10 and must be met before sample analysis can start. Internal standards in samples are monitored for instrument drift. If the internal standard data indicate instrument failure, the samples may require reanalysis. If internal standard response is affected by matrix effects, the impact on sample data is evaluated and the data if lagged appropriately.

9.9.1. Any samples that do not meet internal standard criteria set forth to the continuing calibration (Section 10.6) as compared the Internal Calibration (ICAL) must be evaluated for validity. If multiple calibration mixtures are used, all internal standards are compared back to the primary calibration mixture (Aroclor 1016/1260). Alternatively, the internal standards in the samples may be compared to the response of the daily CCV, on a per batch basis, to meet client or program, requirements. Evaluation may include reanalysis, unless there is obvious matrix interference. If the change in internal standard response is a matrix effect confined in an individual sample (and not due to instrument drift), reanalysis may not be necessary, but should be verified by the department manager or client. Any internal standard responses outside the control criteria should be noted in the case narrative. If the change in the internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.9.2. The internal standard 1-bromo-2-nitrobenzene is added to all standards and samples to result in 200 µg/mL on column. The internal standard is added directly to each individual ICAL point and an internal standard intermediate is used to spike batch QC and samples. For a 1000 µL aliquot of sample, 10 aliquot of sample, 10 µL of a 20 µg/mL internal standard intermediate would be added prior to sample analysis.

9.10. Nonconformance and Corrective Action

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Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager Quality Assurance Summaries

9.11. Quality Assurance Summaries

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Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.12. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

## 10. CALIBRATION

10.1. Initial Calibration

10.1.1. Refer to Table 5 for the initial calibration analytical sequence.

10.1.2. At this time both internal and external calibrations are used. A multi point calibration (6 or 7 points are typical) of the Aroclor 1016/1260 mix is generated, and only mid level single points for the other Aroclor mixes are analyzed.

10.1.2.1. For Methods 8082, 8082A, TO-4A and TO-10A, the average response factor is used to quantitate Aroclors 1260 and 1016; other Aroclors are quantitated from the single point.

10.1.2.2. In the case of Method 608, quantitation of Aroclors must be performed using initial calibrations consisting of at least 3 levels.

10.1.2.3. In the case of work which falls under QSM 5, quantitation of Aroclors must be performed using initial calibrations consisting of at least 5 levels.

10.1.2.4. The analyst may include a full calibration (including ICV) for any of the Aroclors with the initial calibration, if requested by the client or program.

10.1.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the multi-point calibration in addition to the 1016/1260 mix.

10.1.3. The high and low standards for the initial multi-point calibration of 1016/1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

10.1.4. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.1.5. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new (uncalibrated) columns, or replacing the ECD detector. A new calibration is not required after performing column maintenance, replacing the septum or syringe, or performing other minor maintenance.

10.1.6. With the exception of instances detailed in policy CA-T-P-002, Selection of Calibration Points, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5

points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Curve fitting other than average response factor requires a multipoint calibration for each Aroclor mixture.

10.1.7. Internal Standard Calculations

10.1.7.1. Relative response factor (RRF)

***Equation 1***

$$RRF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

Where:

$A_X$ = Area of the compound to be measured
$A_{IS}$ = Area of the internal standard
$C_{IS}$ = Concentration of the internal standard, ng/mL
$C_X$ = Concentration of the compound being measured, ng/mL

10.1.7.2. Standard deviation (SD):

***Equation 2***

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(X_i - X)^2}{n-1}}$$

Where:

$X_i$ = Value of X at i
$N$ = Number of points
$X$ = Average value of $X_i$

10.1.7.3. Percent relative standard deviation (%RSD):

***Equation 3***

$$\%RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where:

SD = Standard Deviation (Eq. 2)

$\overline{RRF}$ = Average of the relative response factors

10.1.7.4. Percent drift – Percent drift is used when average response factor calibration is not used (i.e., for calculating acceptance of continuing calibration of analytes calibrated using a linear or quadratic curve fit). For acceptance of analytes calculated using average response factor, see Section 10.1.7.5, Percent Difference.

***Equation 4***

$$\%Drift = \frac{C_{Found} - C_{Expected}}{C_{Expected}} \times 100$$

Where:

C = Concentration

10.1.7.5. Percent Difference – Percent difference is used when average response factor calibration is used.

***Equation 5***

$$\%Difference = \frac{RRF_{Std} - \overline{RRF}}{\overline{RRF}} \times 100$$

Where:

$\overline{RRF}$ = Average of the relative response factors

10.1.7.6. $RRF_{std}$ = Relative Response factor of the continuing calibration verification

10.1.7.7. Calculating the "on-column" amount in an unknown sample using Average RRF:

***Equation 6***

$$C_X = \frac{A_X \times C_{IS}}{A_{IS} \times \overline{RRF}}$$

Where:

$A_X$ = Area of the compound to be measured
$A_{IS}$ = Area of the internal standard
$C_{IS}$ = Concentration of the internal standard, ng/mL
$C_X$ = Concentration of the compound being measured, ng/mL
$\overline{RRF}$ = Average response factor of the compound.

10.1.8. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak response to the mass or concentration injected may be used to prepare a calibration curve. Peak response is generally measured as the area of the peak. The choice of height or area is made at the time of initial calibration, and may not be altered for a given compound until the next initial calibration.

***Equation 7***

$$\text{Response Factor (RF)} = \frac{\text{Peak Area}}{\text{Concentration of Solution (ng/ml)}}$$

10.1.9. To calibrate Aroclor mixtures, select a minimum of 5 major peaks in the analyte pattern. At least 5 peaks are required (3 peaks for Aroclor 1221). Each selected peak should be at least 25% of the largest Aroclor peak in the pattern. The set of peaks chosen should include at least one peak unique to that Aroclor. Calculate a response factor (Equation 1) for each of the chosen peaks.

10.1.10. The center of the retention time window for each peak is updated with the initial calibration. If more than one calibration level is used for an Aroclor (such as with the 1016/1260), use the mid-level (level 4 from Table 3) standard.

10.2. Calibration Curve Fits

Curve fits other than average response factor require a multi-point calibration for each Aroclor mixture. Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD of the response factor or calibration factor of each analyte is ≤ 20%. Method 608 requires the %RSD to be <10% for the average response factor. If an analyte exceeds the criteria, then a regression line (linear or curved) should be attempted. If the regression lines do not meet criteria (coefficient of determination, r2 ≥ 0.990, or correlation coefficient, r ≥ 0.995), evaluate the calibration, then perform any required instrument maintenance prior to repeating the initial calibration process.

Note: If a regression line is used, multi-level calibrations must be performed for each Aroclor, as the assumption of linearity through the origin for each Aroclor is no longer valid.

10.2.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.2.2. Average response factor - The average response factor may be used for an analyte if the percent relative standard deviation (%RSD) of the response factors for a given analyte is ≤ 20% ( < 10% for Method 608). The equation for average response factor is:

***Equation 8***

$$\overleftarrow{\mathrm{RF}} = \frac{\sum_{i=1}^{n} RF_i}{\mathrm{n}}$$

Where n = Number of calibration levels, and

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.2.3. Linear regression

The linear fit uses the following function

***Equation 9*** $y = ax + b$ ***or*** $x = \frac{(y-b)}{a}$

Where: $y$ = Instrument response
$x$ = Concentration
$a$ = Slope
$b$ = Intercept

10.2.4. Quadratic curve

The quadratic curve uses the following function:

***Equation 10*** $y = ax^2 + bx + c$

Where: $y$ = Instrument response
$x$ = Concentration
$a$ = Curvature
$b$ = Slope
$c$ = Intercept

10.3. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve fit (linear or otherwise) is used, the intercept of the curve at zero response must be less than ± ½ the reporting limit for the analyte.
- For the linear case, the correlation coefficient ($r$) must be greater than or equal to 0.995. For the quadratic case the coefficient of determination ($r^2$) may be used, and must be greater or equal to 0.990.

10.4. Initial Calibration Verification (ICV): A second source standard must be analyzed with the initial calibration curve. The average of the sum of the maximum required peaks for each Aroclor of the second source calibration must be within ± 20% (Method 8082A) or within ± 15% (Methods 608, 8082, TO-4A, and TO-10A) of its expected value when calculated against the initial calibration. Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards

- Recalibrate as needed

10.5. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If calibrations are required, calibration data quality objectives may need to be established with the client.

10.6. Calibration Verification

10.6.1. At a minimum, the working calibration curve or RF must be verified by the analysis of a mid-level calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The working calibration curve or calibration factor for Method 608 must be verified on each working day prior to sample analysis.

10.6.1.1. Methods 8082 and 8082A requires verification at the beginning, after every 12 hours or 20 samples (whichever is more frequent), and at the end of the analysis sequence.

10.6.1.2. Some programs, such as DOD, may require more frequent verification, or base the frequency on the number of samples instead of a time period. DOD requires a CCV for every ten injections of field samples.

10.6.1.3. At a minimum, the calibration verification includes analysis of the Aroclor 1260/1016 mix.

10.6.1.4. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

10.6.2. % Difference is used to compare the continuing calibration verification to the initial calibration when the average response factor is used.

***Equation 11***

$$\%Difference = \frac{\left|X - \overline{X}\right|}{X} \times 100\%$$

Where X = Calibration or response factor of the CCV

$\overline{X}$ = Average Calibration or response factor.

10.6.3. % Drift is used for comparing the continuing calibration to a liner or quadratic curve. The criterion for % drift is the same as for % difference.

***Equation 12***

$$\%\text{Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

10.6.4. Any individual compound with an average of the sum of required peaks with %D ≤ 20% (Method 8082A) or within ≤ 15% (Methods 8082, 608, TO-4A, and TO-10A) meet the calibration criteria. Some programs such as DOD may have other %D requirements.

10.6.5. It is not necessary to run a daily continuing calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration, i.e., the calibration curve and second source standard (initial calibration verification).

10.6.6. Samples must be bracketed by calibration verification standards that meet the criteria listed above.

10.6.7. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria and the preceding samples show no evidence of similar failure, the preceding samples have been successfully bracketed and no further action is required. If this CCV fails or adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue.

10.6.8. It is not allowed to analyze repeat CCVs on unattended runs.

10.6.9. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.6.10. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 20% (Method 8082A) or ± 15% (Methods 8082, TO-4A, and TO-10A) (or other specified requirements) corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than the criteria above, (or other specified requirements) a new calibration curve may be necessary.

10.6.10.1. Any samples injected after the last good continuing calibration standard must be reinjected, unless one of the following conditions is satisfied:

10.6.10.2. If the CCV response is elevated and the preceding samples are non-detect for all analytes of concern, an NCM may be filed and the data reported.

10.6.10.3. If the samples have already been analyzed twice, and it is apparent that matrix effects are causing the out of control event, an NCM may be filed and the data reported. Client consultation or dilution of samples may be necessary.

## 11. PROCEDURE

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified.
The Nonconformance memo will be filed in the project file. Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Extraction and cleanup – Extraction and cleanup procedures are described in SOP WS-OP-0002.

11.3. Suggested gas chromatographic conditions are given in Table 2.

11.4. Allow extracts to warm to ambient temperature before injection. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. The suggested analytical sequence is given in Table 5.

11.5.1. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a CCV.

11.5.2. Solvent blanks may be analyzed immediately following CCVs or suspect samples at any point during the run, however, they may not be routinely used to “clean out the instrument” immediately prior to CCVs.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an

injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte. For Aroclors use the retention time of the peaks used for calibration. The retention time window is defined as plus or minus three times the standard deviation of the retention times of each analyte.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid point of the initial calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than ± 0.03 minutes, use ± 0.03 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by initial calibration.

11.7. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP WS-OP-0013 for determination of percent moisture.

## 12. CALCULATIONIS/DATA REDUCTION

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Aroclors may be present. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence. Certain programs, such as the QSM, require second column confirmation of Aroclors.

12.1.3. If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect, and the results are qualified. If no anomalies, such as overlapping peaks, are noted, report the higher result, or both results.

12.2. Quantitation of Aroclors

12.2.1. If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1216 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative.

12.2.2. If well-separated Aroclor patterns are present, then both Aroclors are quantitated and reported.

12.2.3. Certain programs, such as those under the DOD QSM, may require that multiple mixtures be reported if identified. Refer to the DOD QSM policy WS-PQA-021 for further details.

12.3. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix or screening results.

12.4. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.4.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.4.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request. Refer to policy WS-PQA-021 for requirements for projects under the auspices of the DOD.

12.5. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in a nonconformance memo.

12.6. Dual channel quantitation – In the event that two columns are used for the analysis, used the guidance below to determine how to report the data. Certain programs, such as QSM, may have differing requirement for the reporting data from dual channel analyses. Refer to the QSM policy (WS-PQA-021) or the client QAPPs.

For confirmed results, two approaches are available to the analyst:

A) The primary column approach
B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.6.1. Primary column approach (A):

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true:

- There is obvious chromatographic interference on the primary column.
- The result on the secondary column is 40% greater than the result on the primary column, and there is no reason apparent on the chromatogram for rejecting the higher value (i.e., faulty integration, negative dips affecting the baseline, etc.)
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.) In this instance, only ND (not requiring deconfirmation) or spike results may be reported.

- LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly.

12.6.2. Better result approach (B):

The higher of the two results is normally reported unless obvious chromatographic interference is noted.
LCS, surrogates, and MS/MSD are reported from the primary column unless a standard failure dictates reporting a given analyte from the secondary column. This must be noted in an anomaly.

12.6.3. If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect, and the results are qualified. If no anomalies, such as overlapping peaks, are noted, report the higher result, or both results. RPD is calculated using the following formula:

***Equation 7***

$$RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$

Where R = Result

12.6.4. The experience of the analyst should weigh the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.7. Calculations

12.7.1. Concentration in the extract (single peaks):

***Equation 8***

$$Concentration(ng/mL) = \frac{A}{CF}$$

12.7.2. Where: A = Response for the analyte in the sample

12.7.3. CF = Calibration factor. Area (ng/mL), Section 10.1

12.7.4. Concentration in the extract, Aroclor mixture:

***Equation 9***

$$Concentration(ng/mL) = \frac{\sum_{i=1}^{n} C_{peak}}{n}$$

12.7.5. Where: $C_{peak}$ = Concentration of the peak, using Equation 8

12.7.6. n = number of peaks for the Aroclor mixture

12.7.7. Concentration in the sample

**Equation 10**

$$Concentration(ug/L\ or\ ug/kg) = \frac{C_{ex}\ x\ D\ x\ FV}{A_{sa}}$$

12.7.8. Where: $C_{ex}$ = Concentration in the extract (Equations 8 and 9)

12.7.9. $D$ = Dilution factor

12.7.10. $FV$ = Final extract volume (mL)

12.7.11. $A_{sa}$ = Amount of sample extracted (mL or g)

12.7.12. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:.

**Equation 11**

$$\%\ Recovery = \frac{Concentration\ found}{Concentration\ spiked}\ x\ 100$$

## 13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes

for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

## 14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. 1-2 milliliter autovials containing small amounts of solvent extract. These are collected at the instrument and immediately transferred to a satellite collection container in a fume hood. When full or after no more than one year, these satellite collection containers are transferred to the waste disposal area for disposal.

15.2. Extracts, extract dilutions, all disposable materials (filters, disposable glassware, etc.) with concentrations of Aroclors greater than 50 ppm. These must receive

special handling, shipping and disposal as PCB waste. Contact the Hazardous Waste Technician for specific instructions, as these cannot be disposed in normal waste streams.

## 16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8000B, Revision 0, December 1996, Determinative Chromatographic Separations

16.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8000C Revision 3, March 2003, Determinative Chromatographic Separations

16.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, Method 8082, Revision 0, December 1996, Polychlorinated Biphenyls (PCBs) by Gas Chromatography

16.4. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-4A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.5. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-10A, January 1999, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)

16.6. 40 CFR Appendix A Part 136 Method 608 April 8, 2014.

## 17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Reference Method 8082

17.2.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.3. Modifications from Reference Methods TO-4A and TO-10A

17.3.1. A minimum 5-point calibration is performed for Aroclor 1016/1260, and a single point calibration is performed for the remaining mixtures. An average response factor is determined for each analyte.

17.3.2. The calibration is verified daily or every 12 hours with the mid point standard.

17.3.3. The RT windows are set using the mid point standard and are updated daily or every 12 hours from the mid point standard.

17.3.4. Sample analysis is performed using a DB-608 and DB-1701, or other appropriate GC columns.

17.3.5. Instrument operating conditions will be determined on an instrument and column specific basis.

17.3.6. Method Blanks are deemed acceptable if no target analytes are present at or above the reporting limits or >10% of the measured concentration in the associated samples.

17.3.7. Sample results are not corrected for extraction efficiency, as determined by surrogate recovery.

## 18. ATTACHMENTS

18.1. Table 1 – Standard Analyte List and Reporting Limits

18.2. Table 2 – Recommended Conditions for DB-608, DB5 OR DB-1701

18.3. Table 3 – Calibration Levels

18.4. Table 4 – LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis with Acid Cleanup

18.5. Table 5 – Suggested Analytical Sequence

18.6. Table 6– QC Acceptance Criteria – Methods TO-4A and TO-10A

18.7. Table 7 - QC Acceptance Criteria – Method 608

18.8. Table 8– Method Accuracy and Precision as Functions of Concentrations – Method 608

18.9. Table 9– Summary of Method 608 QC Requirements.

## 19. REVISION HISTORY

19.1. WS-GC-0002, Revision 5.2, Effective 4/01/2016

19.1.1. Section 2 – Changed quantitation from external standard method to internal standard method.

19.1.2. Added Section 7.1.2 – Internal Standards

19.1.3. Added Section 9.9 – Internal Standards

19.1.4. Added Section 10.1.7 – Internal standard range.

19.1.5. Editorial changes.

19.2. WS-GC-0002, Revision 5.1, Effective 06/05/2015

19.2.1. Section 10.4, inserted, "The average of the sum of the maximum required peaks for each Aroclor of the second source calibration must be within ± 20% (Method 8082A) or within ± 15% (Methods 608, 8082, TO-4A, and TO-10A) of its expected value when calculated against the initial calibration."

19.2.2. Removed Section 10.6.1.3, "The initial calibration verification standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are given in Table 3."

19.2.3. Section 10.6.4, changed from 'Any individual compounds with…' to 'Any individual compound with an average of the sum of required peaks with …'.

19.2.4. Editorial changes

19.3. WS-GC-0002, Revision 5.0, Effective 04/15/2014

19.3.1. Section 1.1, appended, "This procedure is also applicable to the determination of PCBs in municipal and industrial discharges under 40 CFR 136.1."

19.3.2. Section 9.2.1, inserted, "Method 608 requires an LCS frequency equivalent to 10% of all samples analyzed."

19.3.3. Section 9.8, inserted "Method 608 requires a matrix spike on at least 10% of the samples from each sample site being monitored."

19.3.4. Added Section 9.8.3 and 9.8.3.1 regarding the spiking criteria for Method 608

19.3.5. Inserted Section 10.1.2.1, 10.1.2.2, and 10.1.2.3, which discuss the calibration curve set-up and criteria for the various references methods associated with this SOP.

19.3.6. Section 10.2, inserted, "Method 608 requires the %RSD to be <10% for the average response factor."

19.3.7. Section 10.2, appended, "Note: If a regression line is used, multi-level calibrations must be performed for each Aroclor, as the assumption of linearity through the origin for each Aroclor is no longer valid."

19.3.8. Section 10.2.2, first sentence, appended, "( < 10% for Method 608)."

19.3.9. Section 10.6.1, appended, " The working calibration curve or calibration factor for Method 608 must be verified on each working day prior to sample analysis."

19.3.10. Added Section 16.1, "40 CFR Appendix A Part 136 Method 608 April 8, 2014."

19.3.11. Added Tables 7, 8, and 9

19.4. WS-GC-0002 rev 4.6, Effective 07/03/2013

19.4.1. Amended Section 5 to accommodate the changes in source standards and resulting TSCA requirements.

19.4.2. Updated SOP to reflect change to TALS system

19.4.3. Updated to include Method 8000C/8082A criteria. Specific sections changed are the title and Sections 1.1, 9.7.5, 10.4, 10.6.4, and 10.6.10

19.5. WS-GC-0002 rev. 4.5 Effective 04/12/2013

19.5.1. Changed references from 'Target' to 'Chrom' and from 'Quantims' to 'TALS'.

19.5.2. Updated Tables 1, 2, and 3 with current operating parameters.

19.5.3. Editorial revisions.

19.6. WS-GC-0002 r4.4, Effective 02/17/2012

19.6.1. Section 11.6.3: Changed retention time window from 0.05 minutes to 0.03 minutes.

19.6.2. Editorial revisions.

19.7. WS-GC-0002 r4.3, Effective -1/07/2011

19.7.1. All references to QSM 4.1 were updated to QSM 4.x to encompass new revisions.

19.7.2. Changed Section 10.1.8 from "…select 5-10 major peaks in the analyte pattern. At least 5 peaks are required." to "…select 3-5 major peaks in the analyte pattern. At least 3 peaks are required."

**Attachments**

**Table 1**
**Standard Analyte List and Reporting Limits**

| Compound | Reporting Limit | | | | |
|---|---|---|---|---|---|
| | Water µg/L | Soil µg/kg | Waste µg/kg | Air µg/sample | Wipe µg/sample |
| Aroclor 1016 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1221 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1232 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1242 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1248 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1254 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1260 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1262 | 1.0 | 33 | 1000 | 0.75 | 1.0 |
| Aroclor 1268 | 1.0 | 33 | 1000 | 0.75 | 1.0 |

The following concentration factors are assumed in calculating the Reporting Limits:

**Table 1B**

| Matrix | Sample Volume | Final Extract Volume | Initial Extract Cleaned and submitted to GC group |
|---|---|---|---|
| Water | 1000 mL | 10 mL | 2 mL |
| Water by Reduced Volume Extraction (RVE) | 250mL | 5 mL | 2 mL |
| Low Level Soil/Solid | 30 g | 10 mL | 2 mL |
| Low Level Soil/Solid by EPA 3586 (MAE) | 15 g | 5 mL | 2 mL |
| High Level Solid/Waste Oil | 1 g | 10 mL | 2 mL |
| Air (trapped in PUFF or similar) | 1 Sample | 10 mL: | 2 mL |

| Table 2 | |
|---|---|
| **Parameter** | **Recommended Conditions for DB-608, DB5 or DB-1701** |
| Injection port temp | 230°C |
| Detector temp | 300°C |
| Temperature program | 180°C for 1 minute, 8°C /minutes to 280°C, hold 10 minutes |
| Column 1 | DB-608 30m x 0.53mm x 0.83µm |
| Column 2 | DB-1701 30m x 0.53mm x 1.0µm |
| Column 3 | DB-5 30m x 0.53mm x 1.0µm |
| Injection | 2µL |
| Carrier gas | Hydrogen |
| Make up gas | Nitrogen |
| Y splitter | Restek |
| | **Recommended Conditions for Stx-CLPesticides 1 and 2** |
| Injection port | 230°C, 70.0 psi for 0.5 min in pulsed splitless mode |
| Detector temp | 300°C |
| Temperature program | 100°C for 1 minute - 12.5°C /minutes to 300°C, hold 2 minutes - 30°C /minutes to 320°C, hold 1.33 minutes |
| Flow program | 2.8 mL/min for 19 min, 30 mL/min$^2$ to 15 mL/min for 1.6 min |
| Column 1 | Stx-CLPesticides-1 30m x 0.32mm x 0.5µm |
| Column 2 | Stx-CLPesticides-2 30m x 0.32mm x 0.25µm |
| Injection | 2µL |
| Carrier gas | Hydrogen |
| Make up gas | Nitrogen |
| Y splitter | Restek |

| Table 3 | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Calibration Levels µg/L** | | | | | | | |
| | **Level 1** | **Level 2** | **Level 3** | **Level 4** | **Level 5** | **Level 6** | **Level 7** |
| 1-Br-2-nitrobenzene (IS) | 200 | 200 | 200 | 200 | 200 | 200 | 200 |
| Aroclor 1016/1260 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1242 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1221 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1254 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1232 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1248 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1262 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| Aroclor 1268 | 25 | 50 | 100 | 200 | 500 | 1000 | 2000 |
| *Surrogates are included with all the calibration mixes at the following levels:* | | | | | | | |
| Tetrachloro-m-xylene | 2.5 | 5 | 10 | 20 | 50 | 100 | 200 |
| Decachlorobiphenyl | 2.5 | 5 | 10 | 20 | 50 | 100 | 200 |

*Note: It is recommended to use the Level 4 standard as the initial CCV of an analytical sequence (as described in Section 10 of this SOP), and the Level 5 for subsequent CCVs.*

| Table 4 | | | | | |
|---|---|---|---|---|---|
| **LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis with Acid Cleanup** | | | | | |
| | Aqueous µg/L | Soil µg/kg | Waste µg/kg | Air µg/sample | Wipes µg/sample |
| Aroclor 1016 | 2.0 | 66.7 | 2000 | 2.0 | 2.0 |
| Aroclor 1260 | 2.0 | 66.7 | 2000 | 2.0 | 2.0 |
| Tetrachloro-m-xylene (Surrogate) | 0.20 | 13.3 | 400 | 0.2 | 0.4 |
| Decachlorobiphenyl (Surrogate) | 0.20 | 13.3 | 400 | 0.2 | 0.4 |

**Table 5**
**Suggested Analytical Sequence (Method 8082/TO-4A/TO-10A)**

**Initial Calibration**

| Injection # | | |
|---|---|---|
| 1 | Solvent blank (optional) | |
| 2 | Aroclor 1016/1260 | Level 1 |
| 3 | Aroclor 1016/1260 | Level 2 |
| 4 | Aroclor 1016/1260 | Level 3 |
| 5 | Aroclor 1016/1260 | Level 4 |
| 6 | Aroclor 1016/1260 | Level 5 |
| 7 | Aroclor 1016/1260 | Level 6 |
| 8 | Aroclor 1016/1260 | Level 7 |
| 9 | Aroclor 1016/1260 | ICV Level 6 (1000 ppb) |
| 10 | Solvent Blank | |
| 11 | Aroclor 1221/1254 | Level 4 |
| 12 | Aroclor 1232/1262 | Level 4 |
| 13 | Aroclor 1242/1268 | Level 4 |
| 14 | Aroclor 1248 | Level 4 |
| 15 | Aroclor 1221/1254 | Level 4 |
| 16 | Solvent blank | |
| 17-33 | Sample 1-20 (or as many samples as can be analyzed in 12 hours) | |
| 34 | Aroclor 1016/1260 | Level 4 |
| etc | | |

**Daily Analysis**

| Injection # | | |
|---|---|---|
| 1 | Solvent blank (optional) | |
| 2 | Aroclor 1016/1260 | Level 3 |
| 3-22 | Sample 1-20 (or as many samples as can be analyzed in 12 hours) | |
| 23 | Aroclor 1016/1260 | Level 4 |
| 24-43 | Samples 21-40 (or as many samples as can be analyzed in 12 hours) | |
| 44 | Aroclor 1016/1260 | Level 4 |
| etc | | |

Note: Some programs and clients require that CCVs be analyzed after every 10 samples, rather than every 12 hours.

**TABLE 6**
**LCS and Surrogate Recovery Limits**

| | LIMITS | | |
|---|---|---|---|
| Method | LCS Native Recoveries | Surrogate | RPD |
| TO-4A | 65 – 125 | 60 – 120 | 30% |
| TO-10A | 65 – 125 | 60 – 120 | 30% |

**Table 7**
**QC Acceptance Criteria—Method 608**

| Parameter | Test conc. (µg/L) | Limit for s (µg/L) | Range for X (µg/L) | Range for P, $P_s$(%) |
|---|---|---|---|---|
| PCB-1016 | 50 | 10.0 | 30.5-51.5 | 50-114 |
| PCB-1221 | 50 | 24.4 | 22.1-75.2 | 15-178 |
| PCB-1232 | 50 | 17.9 | 14.0-98.5 | 10-215 |
| PCB-1242 | 50 | 12.2 | 24.8-69.6 | 39-150 |
| PCB-1248 | 50 | 15.9 | 29.0-70.2 | 38-158 |
| PCB-1254 | 50 | 13.8 | 22.2-57.9 | 29-131 |
| PCB-1260 | 50 | 10.4 | 18.7-54.9 | 8-127 |

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4 of Method 608).

X = Average recovery for four recovery measurements, in µg/L (Section 8.2.4 of Method 608).

P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2 of Method 608).

D = Detected; result must be greater than zero.

Note: These criteria are based directly upon the method performance data in Table 8 of Method 608. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 8.

**Table 8**
**Method Accuracy and Precision as Functions of Concentration—Method 608**

| Parameter | Accuracy, as recovery, X′ (µg/L) | Single analyst precision, $s_r'$ (µg/L) | Overall precision, S′ (µg/L) |
|---|---|---|---|
| PCB-1016 | 0.81C+0.50 | 0.13X+0.15 | 0.15X+0.45 |
| PCB-1221 | 0.96C+0.65 | 0.29X−0.76 | 0.35X−0.62 |
| PCB-1232 | 0.91C+10.79 | 0.21X−1.93 | 0.31X+3.50 |
| PCB-1242 | 0.93C+0.70 | 0.11X+1.40 | 0.21X+1.52 |
| PCB-1248 | 0.97C+1.06 | 0.17X+0.41 | 0.25X−0.37 |
| PCB-1254 | 0.76C+2.07 | 0.15X+1.66 | 0.17X+3.62 |
| PCB-1260 | 0.66C+3.76 | 0.22X−2.37 | 0.39X−4.86 |

X′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of X, in µg/L.

S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.

C = True value for the concentration, in µg/L.

**Table 9**
**Summary of Method 608 QC Requirements**

| QC Objective | Frequency | Criteria | Corrective Action |
|---|---|---|---|
| Initial Calibration (ICAL) | Prior to sample analysis; as needed thereafter when CCV fails or major maintenance; column change | <10% RSD to use average RF; Regression curve if >10% RSD | Re-prep and re-curve |
| Calibration Curve Verification (CCV) | Each work day prior to sample analysis | ± 15% of the expected response | Repeat once using a fresh calibration standard; New calibration curve. |
| Matrix Spike (MS) | 10% of the samples from each sample site | Compare recovery (P) with acceptance criteria in Table 7 | Analyze QC Check Standard (QCCS) and compare recovery (Ps) with acceptance criteria in Table 7. |
| Quality Control Check Standard (QCCS) | Analyzed if any analyte in matrix spike is outside QC acceptance limit. | Compare recovery (Ps) with acceptance criteria in Table 7 | Recovery for the QCCS is evaluated for any parameter that is outside the QC acceptance criteria in the matrix spike (MS). I fa parameter is outside acceptance criteria in both the MS and QCCS, that parameter can not be reported for regulatory purposes. |

# Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry

[Methods 200.8, 6020 and 6020A]

# Title: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry [Methods 200.8, 6020 and 6020A]

**Approvals (Signature/Date):**

| | |
|---|---|
| David Allameh<br>Operations Manager<br>9/9/15 Date | Joe Schairer<br>Health & Safety Manager / Coordinator<br>9/1/15 Date |
| Lisa Stafford<br>Quality Assurance Manager<br>9/1/2015 Date | Crystal Pollock<br>Laboratory Director<br>9/14/15 Date |

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## 1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 200.8, 6020 and 6020A.

1.2. Table 1 lists the analytes contained in the standard analyte list for ICPMS. These are the analytes for which the laboratory maintains certification in the form of PT samples, MDLs, IDLs, DOCs and linear range analyses. Elements not in this list may be analyzed with the client's knowledge that we do not maintain certification for analytes, only standards, outside the scope of our standard product.

1.3. Methods 200.8, 6020 and 6020A are applicable to the determination of total recoverable analyses in ground water, surface waters, and TCLP, STLC, and SPLP leachates/extracts. These methods are also applicable to the total analysis of soils, sludges, sediments and wastes. All matrices require digestion prior to analysis with the exception of STLC extracts. At a minimum, dissolved samples and STLCs are matrix matched under the guidelines of WS-IP-0008. STLCs are to be matrix matched at a 10x dilution. Silver concentrations must be below 0.1 mg/L in aqueous samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data

1.4. Reporting Limits

1.4.1. The standard reporting limits for metals analyzed by ICP-MS are listed in Table 1.

1.4.2. Reporting limits may differ from those listed if prescribed in a reviewed and finalized client specific QAPP.

1.4.3. Reporting limits for samples analyzed under QSM and AFCEE criteria usually differ from those shown in Table 1 and in many cases are specific to the client.

1.5. When undertaking projects for Department of Defense (DoD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

## 2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are introduced via a cross flow nebulizer into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio-frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber

that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

## 3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

## 4. INTERFERENCES

4.1. Isobaric interferences: Isobaric interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.

4.3. See Tables 8 and 9 for a list of common interferences and the isotopes affected.
**Note: Not all-possible interferences are listed.**

4.3.1. Matrix interferences: Internal standards will be used to correct for some matrix interferences.

4.3.2. A representative sample(s) of the project or projects being analyzed will be screened to avoid using an analyte as an internal standard that is present in the samples at a concentration greater than 10% of the internal standard concentration.

4.3.3. Internal standards should be added at a level to give approximately 100,000 - 2,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ±50 amu of the mass of the affected analyte.

4.3.4. Severe matrix effects are monitored by comparing the internal standard intensities of the sample to the internal standard intensities of the initial calibration blank. If any internal standard intensity is less than 30% or greater than 120% of the internal standard intensity of the calibration blank, a fivefold dilution will be performed on the sample to correct for severe matrix effects, and the sample reanalyzed.

## 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.1.2. This instrument produces RF energy. People with pacemakers should not go near the instrument while it is in operation.

5.1.3. Some extractions and digestions use hydrofluoric acid, which means that some samples to be analyzed are in an HF solution. The labels for all such extracts and digestates will be highlighted in yellow. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF acid solutions may not be used for any purposes except as prescribed in this or other TestAmerica Sacramento SOPs. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

**WARNING: The TestAmerica Sacramento Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response, the victim will be transported to the UCD Medical Center Emergency Room. Appendix 1 is a detailed first aid plan for working with HF.**

5.1.3.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Mylanta. If the burn site is too large to massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with the Epsom salt solution.

5.1.3.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.3.3. Glass reacts with HF to produce toxic silicon tetraflouride.

5.1.3.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.3.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3-3/4 full with an Epsom salts solution (1/2 cup of Epsom salts per quart of water). Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

5.2.1. Although no hazardous materials are used during this process, the following is a list of the materials which might be found in the digestates, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

5.2.2. A complete list of materials used in the method can be found in the reagents

and materials section of the extraction/digestion SOP related to the samples in question.

5.2.3. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

| Material (1) | Hazards | Exposure Limit (2) | Signs and symptoms of exposure |
|---|---|---|---|
| Nitric Acid (1) | Corrosive<br>Oxidizer<br>Poison | 2 ppm-TWA<br>4 ppm-STEL | Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| Hydrochloric Acid (1) | Corrosive<br>Poison | 5 ppm-Ceiling | Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| Hydrofluoric Acid (1) | Poison<br>Corrosive<br>Dehydrator | 3 PPM - TWA | Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE. |
| 1 – Always add acid to water to prevent violent reactions. | | | |
| 2 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

## 6. EQUIPMENT AND SUPPLIES

Preventive maintenance schedules for instrumentation may be found below.

| | | |
|---|---|---|
| INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)[1] | Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics.<br>Measure quartz torch for proper alignment when removed and cleaned.<br>Clean spray chamber and nebulizer.<br>Clean all filters and fans.<br>Check chiller coolant level.<br>Check and drain oil mist eliminator on roughing pumps. | As Needed |
| | Check sample waste container level.<br>Check quartz torch condition.<br>Check RF coil.<br>Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing.<br>Check condition of sampler and skimmer cones.<br>Check oil level of roughing pumps. | Daily[2] |
| | Replace oil in roughing pumps. | Every 2-3 Months |

6.1. ICP-MS Instrument, either:

6.1.1. Perkin Elmer/Sciex ELAN 6000 ICP-MS or equivalent, utilizing Elan software, version 2.3.2 or equivalent.

6.1.2. Agilent 7700x (G3281A) ICP-MS, equipped with:

6.1.2.1. Autosampler (ASX-5000 Series)

6.1.2.2. Integrated Sample Introduction System (ISIS)

6.1.2.3. MassHunter Software G7201B version B.01.03, Build 393.8

6.2. Argon gas: High-purity grade (99.99%).

6.3. Helium gas: High-purity grade (99.99%) for use with Agilent Instrument.

6.4. Hydrogen gas: High-purity grade (99.99%) for use with Agilent Instrument

6.5. Water re-circulator / chiller.

6.6. Adjustable air displacement pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes, 13 x 100 mm polystyrene or polypropylene.

## 7. REAGENTS AND STANDARDS

Use reagent grade chemicals in all tests. “Certificates of Analysis” should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate

and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.1. Reagent water is produced by a Millipore Nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Concentrated nitric acid ($HNO_3$), trace metal grade or better.

7.3. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.4. Instrument rinse: Add 2000 mL of reagent water to the 4000 mL rinse container. Add 80mL of concentrated $HNO_3$, 40mL of concentrated HCl, then dilute to volume with reagent water.

7.5. Stock standards are purchased as custom multi-element mixes and as single-element solutions. These must be plasma grade standards.

7.5.1. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.

7.5.2. Upon making a standard, the process must be documented in the standards log book and the standard given a specific ID according to laboratory procedure.

7.5.3. All intermediate and working standards must be labeled with the name of the preparer, the date prepared, the expiration date, acid concentrations, and the standard specific ID.

7.5.4. Standards containing silver must be protected from light. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer.

7.5.5. If no expiration date is provided, the stock solutions may be used for up to one year from opening and must be replaced sooner if verification from an independent source indicates a problem.

7.6. Calibration Standard/Continuing Calibration Standard: see Table 2 for instructions on making this standard.

7.7. Initial Calibration Verification (ICV) standard: see Table 3 for instructions on making this standard.

7.8. Low Level Check Intermediate Standard: see Table 4 for instructions on making this standard.

7.9. ICSA standard: see Table 5 for instructions on making this standard.

7.10. ICSAB standard: see Table 6 for instructions on making this standard.

7.11. Internal Standard A Intermediate: See Table 7 for instructions on making this standard.

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.2. All soils must be refrigerated to $3^{o}C \pm 3^{o}C$.

8.3. The analytical holding time for metals by ICP-MS is 6 months from date of collection.

## 9. QUALITY CONTROL

9.1. Batch - a quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards.

9.1.1. A batch must be prepared within the same time frame.

9.1.2. A method blank (MB) and a laboratory control sample (LCS) must be prepared as a part of every batch.

9.1.3. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/MSD).

9.1.4. An analytical batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples.

9.2.1. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples.

9.2.2. If a method blank exceeds ± the reporting limit for a given analyte then the samples associated with that batch must be re-prepared.

9.2.2.1. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo.

9.2.3. For samples requiring laboratory filtration and preservation, the method blank

must also be filtered and preserved. See policy WS-PQA-003 of the Quality Control Program for further details.

9.2.4. DOD/DOE projects including QSM Versions 4.2 and AFCEE Version 4.0: the results of the method blank must be ½ the reporting limit for a given analyte. The same exceptions and corrective actions apply as stated in section 9.2.2.1

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples.

9.3.1. A LCS for water samples consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. The LCS for solid samples consists of PTFE boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples.

9.3.2. The LCS recoveries are evaluated against in-house generated control limits, which are generated per policy WS-PQA-003 and found in the laboratory's LIM system.

9.3.3. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared for the out of control analyte/s except when a LCS exhibits high recovery.

9.3.3.1. For such a case, those samples with analyte concentrations less than the reporting limit can be reported.

9.3.3.2. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3.4. For samples analyzed under DoD QSM Version 4.2, and AFCEE criteria, there is no exception for reporting "ND" samples for a high LCS.

9.3.5. Random marginal exceedance: For analyte lists of 11-30 elements, 1 LCS random marginal exceedance is allowed. One out of control analyte may be accepted if the result is within 4 standard deviations of the mean. The QA department must validate all marginal exceedance incidents. A NCM must be filed when reporting a marginal exceedance.

9.4. The lower limit of quantitation (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value. This check should be

used to both establish and confirm the lowest quantitation limit. The LLQC going through preparation procedure will be called LLCS for TALs identification.

9.5. A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples.

For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

9.5.1. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations The MS/MSD pair must be processed in the same manner and at the same time as the associated samples.

9.5.2. Compare the percent recovery and relative percent recoveries to that in the historically generated control limits. These control limits can be found in the laboratory's LIM system and are generated per policy WS-PQA-003.

9.5.2.1. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS.

9.5.2.2. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5.3. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair, and batch precision is required by the client or program.

9.5.3.1. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details.

9.5.4. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/MSD analysis.

9.5.5. If the amount of an analyte found in the un-spiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply.

9.6. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard (ICV) immediately upon completion of instrument calibration.

9.6.1. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard.

9.6.2. The ICV must fall within +/- 10% of the true value of the standard solution. Table 3 shows the concentration of each standard target analyte in the ICV.

9.6.2.1. An ICB (initial calibration blank) prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

9.6.2.2. The ICB result must fall within +/- the reporting limit from zero.

9.6.3. If either the ICV or ICB fail to meet acceptance criteria the analyst should determine the cause and proceed with corrective action if warranted. This may require re-running the ICV or ICB, repouring and rerunning either the CCV or ICB, or remaking and reanalyzing the ICV or ICB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICV and ICB. The analyst may also recalibrate and rerun the ICV and ICB to verify the calibration.

9.6.4. AFCEE Version 4.0: The ICB and CCB results for all analytes must be less than or equal to 2X the MDL.

9.6.5. QSM Version 4.2: The ICB and CCB results for all analytes must be less than or equal to the LOD (limit of detection).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard, the continuing calibration standard, after every 10 samples and at the end of an analytical run.

9.7.1. Ten samples include any analysis that registers with a result, even if not used.

9.7.2. For method 6020 analysis, the calibration standard is also used as the CCV. The CCV result must fall within +/-10% of the true value for that solution.

9.7.3. A CCB (continuing calibration blank) is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero.

9.7.4. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples.

9.7.5. Results for given analytes may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs for the analytes.

9.7.6. If a mid-run CCV or CCB fails for any number of analytes, the possible cause of failure should be investigated, and all samples analyzed since the last acceptable CCV and or CCB must be reanalyzed for the unacceptable analytes.

9.7.7. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample.

9.7.8. During the course of an analytical run, the instrument may be "resloped" or recalibrated on a CCV/CCB pair to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV/CCB pair before any further samples may be analyzed. Any samples bracketed (including prior to the reslope) by a failed CCV/CCB must be reanalyzed for the unacceptable analytes.

9.8. Low Level Check Standard (LLSTD, LLICV, or LLCCV): Following analysis of the ICV/ICB pair the low level check standard/s is analyzed spiked with concentrations of each analyte of interest at or below the reporting limit. For method 6020A, it is also recommended to analyze the standard after samples (refer also to Section 10.9 for details and criteria).

9.8.1. The percent recovery of the low level standard results must be +/-50% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.2. QSM Version 4.2 and AFCEE Version 4.0: Low level check standard recovery must be ± 20%.

9.8.3. For Method 6020A the LLICV results must be ± 30% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.4. If the recovery is out of control, the analyst should determine the cause and proceed with corrective action. Appropriate corrective actions include rerunning the LLSTD, repouring and rerunning the LLSTD, or remaking and reanalyzing the LLSTD. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the LLSTD. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.9. For all analyses run under DOD/DOE criteria (QSM, AFCEE), linear range standards (LRSTD) are analyzed after the completion of calibration.

9.9.1. The linear range standards contain the analytes of interest at high levels.

9.9.2. The acceptance criterion for linear range standards is ± 10% of the true value.

9.9.2.1. If this criterion is not met for a given analyte/s, a lower concentration standard should be analyzed such that the criterion is met.

9.9.3. Once linear range has been established through the analysis of the linear range standards, no value can be reported that exceeds the concentration of the standards.

9.9.3.1. If sample concentrations exceed that of the linear range standards for any given analyte, the sample requires dilution at a level where all analytes fall below the level of the linear range standards.

9.10. Interference Check Analysis (ICSA/ICSAB): Interference check standards ICSA and ICSAB are analyzed directly after analysis of the low level check standard.

9.10.1. The ICSA/ICSAB are essentially remnants of method 6010 where the analytes spiked in the standard at high levels are considered interferents. For method 6020 these are not normally interferents, but represent analytes that might be seen at higher levels in real world samples.

9.10.2. ICSA (interference check standard A) contains only the high level analytes (interferents) without any spiking of the analytes more notably determined at lower relative concentrations. See Table 5 for the composition of the ICSA.

9.10.3. ICSA is also spiked with 10 ppm bromide to evaluate the correction required for bromide interference on selenium 82. Once the ICSA has been analyzed, the correction factor should be adjusted such that the result for selenium 82 is less than +/- the reporting limit.

9.10.4. Method 6020 does not place any acceptance criteria on ICSA, but only requires that it be analyzed.

9.10.4.1. For standard work, the ICSA concentration of non-spiked analytes should be less than two times the reporting limit to continue analysis. If it exceeds this limit, an NCM should be filed to explain the reason. Acceptable narrations include:

- All associated samples have high levels of the analyte (greater than 10x the level found in the ICSA)
- All associated samples are non-detect for the analyte.

Additional corrective actions include rerunning, repouring, and remaking and rerunning the ICSA.

9.10.4.2. AFCEE Version 4.0 requires the concentration of non-spiked analytes be less than 2X the MDL to continue analysis. Refer to Section 9.10.4.1 (above) for corrective action.

9.10.4.3. QSM 4.2 requires that the absolute value of the concentration of non-spiked analytes be less than the LOD to continue analysis. Refer to Section 9.10.4.1 (above) for corrective action.

9.10.5. The acceptance criteria for all of the methods does not apply when trace impurities are validated by the Certificate of Analysis for the ICSA solution that cause the non-spiked analytes to show positive concentrations outside of acceptance criteria. In such a case, a NCM is required that identifies the impurities and their certified concentrations in the standard.

9.10.6. ICSAB (interference check sample AB) is the same as ICSA, but all of the non-interfering analytes are spiked into the ICSA at the concentrations shown in Table 6.

9.10.7. The ICSAB results for the low level (non-interfering) analytes must fall within 80 – 120% (79.5 – 100.49 with rounding) of the true value. If the ICSAB does not meet these criteria for any analyte, the analyst should determine the cause and proceed with corrective action. Acceptable corrective actions include rerunning the ICSAB, repouring and rerunning the ICSAB, or remaking and reanalyzing the ICSAB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICSAB. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.10.8. For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.

9.11. Dilution test (Serial Dilution) – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.

9.11.1. One sample per preparation batch must be processed as a dilution test.

9.11.2. The test is performed by running a sample at a 5X (1+4) dilution.

9.11.3. Samples identified as field blanks cannot be used for dilution tests.

9.11.4. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 100X the MDL.

9.11.4.1. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.12. Post-digestion spikes: One sample in every batch may be spiked with all of the elements of interest at the instrument following sample preparation. The matrix spiking solution is generally used as the post-digestion spike resulting in the same concentration spike level as the matrix spikes.

9.13. Internal standards are added continuously to all analyses in an analytical run. The intensities must be monitored for every analysis.

9.13.1. For method 6020, The internal standard (IS) intensities of the ICV/CCVs and ICB/CCBs should be within 80 – 120% recovery relative to the internal standard intensities of the calibration blank. If the internal standards for the ICV/CCV/CCB exceed the criteria, terminate the analysis, correct the problem, and reanalyze affected samples.

IS intensities in samples must be within 30 to 120% of the IS intensities for the initial calibration blank, although suppression influencing sample results could be expected at levels less than 60-70% recovery. If the 30 – 120% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.13.2. For method 6020A, the internal standard (IS) intensities in field and QC (including ICV/CCV and ICB/CCB) samples must be greater than 30% and less than 150% of the IS intensities for the initial calibration standard. If the intensity in the sample is less than 30% of the intensity in the initial calibration standard, ensure that the instrument has not drifted by checking the intensities in the nearest calibration blank. If low intensities are also present in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples. If drift has not occurred, remove matrix effects by diluting the sample. Dilute at least 5x and reanalyze. Repeat if the first dilution is not sufficient to ameliorate the matrix effects. Narrate any findings.

9.13.3. For Method 200.8 the internal standard (IS) intensities in field and QC samples must be within 60-125 % of the IS intensities for the initial calibration blank. If internal standards for the ICV/CCV/CCB exceed the 60-125% criteria, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples, Otherwise, if the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample.

Narrate any findings.

9.13.4. The internal standard intensities of the ICV/CCVs and ICB/CCBs should be within 80 – 120% recovery relative to the internal standard intensities of the calibration blank.

9.13.4.1. If this criterion is not met, the analyst must recalibrate (or reslope) the instrument and rerun all samples associated with the out of control QC. Only analytes associated with the out of control internal standard require re-analysis.

9.13.5. Table 7 shows the composition of the internal standard A solution. This standard is diluted further at 2X or 3X prior to analysis.

9.13.6. The internal standard raw intensity counts must be printed on the raw data. The percent recovery of each internal standard relative to the calibration blank or the blank associated with a previous re-slope is printed on the raw data.

9.14. High Level Continuing Calibration Verification (High Level CCV) – This standard is used to verify the instrument's dual detector calibration.

9.14.1. The High Level CCV is analyzed at the beginning of the run to demonstrate whether the instrument is accurately measuring concentration at concentrations near the upper limit of the linear range. If the values are greater than ± 20%D a new dual detector calibration may be required.

9.14.2. Should values in the High Level CCV exceed the ± 20%D criteria, only values less than or equal to the nominal values of the mid-level continuing calibration may be reported.

## 10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Instrument start-up

10.2.1. Follow the instrument start-up procedure outlined in the Perkin Elmer Elan 6000 Operator's Manual and Agilent 7700X Operator's Manual.

10.3. Instrument Tuning

10.3.1. Perkin Elmer

10.3.1.1. Aspirate a 10 ppb tuning solution containing Li, Co, In, and Tl.

10.3.1.2. A mass calibration and resolution checks must be documented and included as part of the raw data package. If the following conditions are not met, re-tune the instrument.

10.3.1.3. Resolution must be < 0.9 amu full width at 10% peak height. The Elan 6000 resolution is maintained at approximately 0.7 amu.

10.3.1.4. Mass calibration must be within ± 0.1 amu from the true value.

10.3.1.5. A daily performance check must be performed as part of the daily "tune". This uses the same solution as in section 10.3.1, and this also contains Mg, Rh, and Ba. The elements Li, Co, In, and Tl must be below 5% RSD. Rh must be at or above 200,000 counts. The CeO and $Ba^{++}$ intensity readouts must be below 0.035 which shows the oxides and doubly charged ions are below 3.5%. .If any of these conditions are not met, maintenance or optimization should be performed until these specifications are met, or an NCM written to explain the affect on the analysis and clients results.

10.3.2. Agilent 7700X

10.3.2.1. A US EPA Tune Check Sample must be performed in the Helium [He] mode and Hydrogen [H2] mode. The US EPA Tune Check Sample contains Ba, Be, Ce, Co, In, Li, Mg, Pb, Rh, Tl, U and Y at 10 ppb. In the [He] mode the RSD% for mass 24, 59, 115, and 208 must be below 5%. In the [H2] mode mass 9, 24, 59, 115, and 208 must be below 5%.

10.3.2.2. Resolution must be < 0.9 amu full width at 10% peak height. The Agilent 7700X resolution is maintained at approximately 0.75 amu.

10.3.2.3. Mass calibration must be within ± 0.1 amu from the true value.

10.4. Initial calibration must be performed daily and each time the instrument is set up.

10.4.1. Calibration consists of a blank and a single calibration standard. Table 2 shows the concentration of all analytes in the calibration standard. Use the average of three integrations for both calibration and sample analyses. Initial calibration acceptance is based upon the ICV falling within +/- 10% recovery from the true value.

10.5. Following calibration, an ICV/ICB pair is analyzed to confirm the accuracy of the calibration (section 9.5).

10.6. Low level check standards are analyzed to confirm the sensitivity of the analytical run at the reporting limit for the samples (section 9.7).

10.7. For DOD/DOE, linear range standards are analyzed after the completion of the calibration as per section 9.8.

10.8. Continuing calibration verifications, CCV/CCB pair, are analyzed every ten (10) samples and at the end of the analytical sequence as per section 9.6.

10.9. For Method 6020A, the low level continuing calibration verification (LLCCV) standard should also be analyzed at the end of each analysis batch. A more frequent LLCCV analysis, i.e., every 10 samples may be necessary if low-level sample concentrations are anticipated and the system stability at low end of the calibration is questionable. The acceptance criteria for the LLCCV standard should be ± 30% of its true value.

## 11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All samples require a preparation prior to analysis depending on the matrix being evaluated. Preparation of each batch requires a method blank, LCS, and matrix spikes and/or matrix spike duplicates. The concentration of analytes in the LCSs and matrix spikes are listed in the individual preparation methods.

11.2.1. For method 6020 and 6020A soils analysis, EPA method 3050B is used to prepare all samples.

11.2.2. For method 200.8, 6020, 6020A aqueous samples, and TCLP leachate samples are prepared using the total recoverable digestion procedure, method 3005A (WS-IP-0001).

11.2.3. For method 200.8, 6020, 6020A dissolved samples, and STLC are prepared using the matrix matching procedure (SOP WS-IP-0008).

11.3. Before setting up an analytical run, read the QAS for all of the lots being analyzed. The QAS is created by the project managers and defines any special criteria required for client specific analyses.

11.4. An instrument backlog is generated from the TALs data system. This backlog lists the Job numbers that were assigned to each batch of samples. The backlog also shows project due date, number of samples and sample matrix. The instrument backlog aids the analyst in deciding the lot IDs to analyze on a given day.

11.5. An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.

11.6. Using utility software, the analyst can generate a sample run log directly from the instrument software.

11.7. In order to use the ICP-MS data upload program into TALs, the following naming conventions must be followed:

11.7.1. Samples are identified by the 5 character work order number.

11.7.2. Quality control parameters have a suffix after their sample ID.

| Parameter | Suffix |
|---|---|
| Method Blanks | MB |
| LCS | LCS |
| LCS Duplicate | LCSD |
| Matrix Spike | MS |
| Matrix Spike Duplicate | MSD |
| Sample Duplicate | DU |
| Serial Dilution | SD |
| Post-digestion Spike | PDS |

11.8. Set-up

11.8.1. Open a new dataset using the date and instrument in the title. For instance the first run (A) on instrument 2 on JAN 1, 2013 would be 070113A2.

11.8.2. Open the appropriate method if one already exists or create a new one for the analytes to be quantitated in the run. A method with all of the analytes has been generated and can be modified to the specific analytes required throughout any given analytical run.

11.8.3. All masses which could affect data quality should be monitored to determine potential interferences. Secondary masses are evaluated per SW 846 protocols.

11.8.4. Internal standards are added to all standards and samples by the instrument peristaltic pump and a mixing tube prior to analysis.

11.8.5. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.

11.9. The order of analysis for the initial QC samples and calibration should be:

1. Rinse
2. CAL Blank
3. STD1 (Calibration Standard)
4. ICV (Second source, must be ± 10% of true value)
5. ICB
6. LLSTD1
7. LLSTD2, etc
8. ICSA (Interference check solution with Bromine, for Selenium. ± RL for Se.)
9. ICSAB (Interference check solution, ± 20% of true value)
10. LRSTD1
11. LRSTD2, etc
12. Rinse (up to 3 rinses)
13. CCV1
14. CCB1
15. CCV2
16. CCB2

11.10. To continue the analytical run, add an additional 10 runs followed by CCV/CCB, and repeat for up to 24 hours.

11.11. Samples must be diluted and reanalyzed for any elements which are present at concentrations exceeding 90% of the upper linear range limit as determined in quarterly linear range studies.

## 12. CALCULATIONS/DATA REDUCTION

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left( \frac{Found(ICV)}{True(ICV)} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left( \frac{Found(CCV)}{True(CCV)} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left( \frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100 \left[ \frac{|MSD - MS|}{\left( \frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \times \left( \frac{Found(LCS)}{True(LCS)} \right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%\ \text{Difference} = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S =Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported in accordance with the TestAmerica Sacramento significant figure policy (WS-PQA-004).

## 13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits (Method 6020) or the static recovery limits of 85-115% (Method 200.8).

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

## 14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acid waste consisting of unused sample, analyzed sample solution and rinse solution. This is collected in four-gallon plastic carboys. When full or after no more than one year, the waste is dumped into an acid waste drum in the H-3 closet. When full to no less than one inch and no more than four inches of the top, or after no more than 75 days, the drum will be transferred to the waste collection area for disposal.

15.2. Miscellaneous disposable glassware, plastic vials with snap top caps, autosampler tubes and similar solid waste. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

## 16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, February 2007.

16.3. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma – Mass Spectrometry, Revision 5.4, 1994

16.4. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

16.5. Perkin Elmer/Sciex ELAN model 6000 Users Manual

16.6. Agilent Series 7700 ICP-MS Users Manual

16.7. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM), Version 4.2, October 2010.

16.8. AFCEE QAPP, Version 4.0, February 2005

16.9. Recommended ICP-MS References

16.9.1. K. E. Jarvis, A. L. Gray, and R. S. Honk, Handbook of Inductively Coupled Plasma Mass Spectrometry; Chapman & Hall, New York 1992.

16.9.2. A. R. Date and A. L. Gray, Applications of Inductively Coupled Mass Spectrometry, Chapman & Hall, New York 1989. ISBN # 0-41201721-0

## 17. METHOD MODIFICATONS

Deviations from Source Method and Rationale

17.1. As a conservation of space, the raw data will only contain the mean and standard deviation results on standard ELAN summary report. As long as the standard deviation data is shown, the analyst can make decisions on replicate quality on the standard ELAN summary report.

17.2. This SOP may be used to analyze for elements not included in Method 6020, as long as appropriate QC samples spiked with the non-routine analytes are run with acceptable results. Clients should be notified that we do not carry method performance indicators for these analytes.

17.3. For reporting consistency, an ICB/CCB is acceptable if the result is <RL. Method 6020 states that the results of the calibration blank (CCB) are to be less than 3x the IDL. If not, terminate the analytical sequence, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the

calibration is not drifting at the low end. TestAmerica Sacramento has adopted an absolute control limit of +/- RL from zero for calibration blank criteria for standard operations. Exceptions are defined in this SOP.

17.4. The ICSA and ICSAB are being made as needed with an expiration date of 5 months or less depending on stock solutions expiration dates. Method 6020 states that the ICSA and ICSAB need to be prepared fresh weekly from ultra pure reagents. TestAmerica Sacramento has adopted to make these standards as needed due to the stability of the purchased stock solutions (manufacturer's expiration date or <1 year).

## 18. ATTACHMENTS

Tables referenced in the body of the SOP

18.1. Table 1 -Standard Target Analyte List

18.2. Table 2 -Calibration and Calibration Check Standard Preparation

18.3. Table 3 -Initial Calibration Verification Standard

18.4. Table 4 -Low Level Check Intermediate Standard Preparation

18.5. Table 5 -Interference Check Solution A

18.6. Table 6 -Composition of the ICSAB Standard

18.7. Table 7 -Internal Standard A Stock

18.8. Table 8 -Common Molecular Ion Interferences in ICPMS

18.9. Table 9 -Recommended Analytical Isotopes and Additional Masses (Elements of Interest)

18.10. Table 10 -Recommended Analytical Isotopes and Additional Masses (Rare Earth Elements and other Elements)

18.11. Table 11 - Elemental Equations used in Calculations

## 19. REVISION HISTORY

19.1. WS-MT-0001, Revision 3.9, Effective 09/09/2015

19.1.1. Section 9.13.1, changed to include ICV/CCV and ICB/CCB criteria for internal standards (formerly section 9.13.4).

19.1.2. Section 9.13.2, changed the first two sentences to read, "For method 6020A,

the internal standard (IS) intensities in samples must be greater than 30% and less than 150% of the IS intensities for the initial calibration standard. If the intensity in the sample is less than 30% of the intensity in the initial calibration standard, ensure that the instrument has not drifted by checking the intensities in the nearest calibration blank."

19.1.3. Section 9.13.3, changed the first two sentences to read, "For Method 200.8 the internal standard (IS) intensities in field and QC samples must be within 60-125 % of the IS intensities for the initial calibration blank. If internal standards for the ICV/CCV/CCB exceed the 60-125% criteria, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples,"

19.1.4. Section 9.13.4 and 9.13.4.1 – removed. Incorporated into 9.13.1. These revisions are to be congruent with current TestAmerica interpretation of IS requirements for ICPMS methods.

19.1.5. Added references to DOD/DOE in place of DOD where applicable.

19.2. WS-MT-0001, Revision 3.8, Effective 12/12/2014

19.2.1. Added EPA Method 200.8 to Scope and Application Sections 1,1 and 1,3.

19.2.2. Section 6.4 – Changed Argon gas to Hydrogen gas.

19.2.3. Added Section 9.13.3 – "For Method 200.8 the internal standard (IS) intensities in the samples must be within 60-125 % of the IS intensities for the initial calibration blank. If the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

19.2.4. Added Section 10.3.2 for Agilent 7700X instrument parameters.

19.2.5. Added the following paragraph following Section 9.5 -"For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

19.2.6. Added the following to the end of Section 13.3.2 – "or the static recovery limits of 85-115% (Method 200.8)"

19.2.7. Editorial changes..

19.3. WS-MT-0001, Revision 3.7 Effective 08/29/2014

19.3.1. Added Section 9.10.8, "For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.)

19.3.2. Editorial changes.

19.4. WS-MT-0001, Revision 3.6, Effective 09/30/2013

19.4.1. Revised Section 9.5.3, 9.7.3 (number changed from 9.7.2) and 9.9.4 to reflect how analytical runs and decision making proceeds.

19.4.2. Removed Section 10.4 (Screening for Internal Standards)

19.4.3. Added Section 11.11 (regarding dilutions for analytes > linear range study)

19.4.4. Added Section 9.14 (High Level CCV) to reflect current practice to improve data quality.

19.4.5. Added references to method 6020A in Sections 1.3, 11.2, and 16.2

19.4.6. Added reference to preventive maintenance schedules in Section 6, and added Table 12 (Preventative Maintenance).

19.4.7. Deleted references to AFCEE 3.1 in Sections 9.3

19.4.8. Inserted Section 9.4 (low level QC Check sample)

19.4.9. Inserted Section 9.8.3 regarding the low-level standard frequency and criteria for method 6020A.

19.4.10. Sections 9.6.3, 9.8.4, 9.10.4.1, and 9.10.7 Clarified the corrective action procedures.

19.4.11. Section 10.3, removed elements not required in the 6020/6020A tuning solution.

19.4.12. Added Section 10.8, low-level calibration standard at the end of the run.

19.4.13. Section 11.4 through 11.7, minor edits to reflect procedure using TALS vs. the older LIMs.

19.4.14. Editorial changes.

19.5. WS-MT-0001, Revision 3.5, Effective 07/05/2013

19.5.1. Modified Section 10.3.3 to follow what is in the method regarding tuning.

19.5.2. Editorial changes.

19.6. WS-MT-0001, Revision 3.4, Effective 09/14/2012

19.6.1. Removed requirement for making ICSA and ICSAB standards weekly from Section(s) 7.9 and 7.10.

19.6.2. Removed references to AFCEE 3.1.

19.6.3. Editorial changes.

19.7. WS-MT-0001, Revision 3.3, Effective 06/10/2011

19.7.1. Inserted Cd (mass 114) in Table 1.

19.7.2. Deleted reference to 6020A in Method heading and reference (Section 19.2).

19.7.3. Removed Section 6.5: Class A volumetric pipettes.

19.7.4. Section 9.11: replaced “must be spiked” with “may be spiked”.

19.7.5. Section 10.4.1: replaced “representative samples must be screened” with “it is recommended that soil samples be screened”.

| TABLE 1 | | | |
|---|---|---|---|
| **Method 6020 Standard Target Analyte List** | | | |
| **Element** | **Symbol** | **Aqueous RL mg/L** | **Soil RL mg/Kg** |
| Aluminum | Al | 0.05 | 5.0 |
| Antimony | Sb | 0.002 | 0.2 |
| Arsenic | As | 0.002 | 0.2 |
| Barium | Ba | 0.001 | 0.1 |
| Beryllium | Be | 0.001 | 0.1 |
| Boron | B | 0.05 | 5.0 |
| Cadmium | Cd (mass 111) | 0.001 | 0.1 |
| Cadmium | Cc (mass 114) | 0.001 | 0.1 |
| Calcium | Ca | 0.05 | 30 |
| Chromium | Cr | 0.002 | 0.2 |
| Cobalt | Co | 0.001 | 0.1 |
| Copper | Cu | 0.002 | 0.2 |
| Iron | Fe | 0.05 | 5.0 |
| Lead | Pb | 0.001 | 0.1 |
| Lithium | Li | 0.01 | 1.0 |
| Magnesium | Mg | 0.05 | 5.0 |
| Manganese | Mn | 0.001 | 0.2 |
| Molybdenum | Mo | 0.002 | 0.2 |
| Nickel | Ni | 0.002 | 0.2 |
| Phosphorous | P | 0.05 | 20 |
| Potassium | K | 0.05 | 5.0 |
| Selenium | Se (mass 82) | 0.002 | 0.2 |
| Selenium | Se (mass 78) | 0.003 | 0.3 |
| Silver | Ag | 0.001 | 0.1 |
| Sodium | Na | 0.05 | 50 |
| Strontium | Sr | 0.005 | 0.5 |
| Thallium | Tl | 0.001 | 0.1 |
| Tin | Sn | 0.01 | 2.0 |
| Titanium | Ti | 0.005 | 0.5 |
| Vanadium | V | 0.01 | 1.0 |
| Zinc | Zn | 0.005 | 1.0 |

| Table 2 | | | | |
|---|---|---|---|---|
| **Calibration Standard and Continuing Calibration Verification Standard** | | | | **Final Volume = 500 mL** |
| **Element** | **Stock Standard ID** | **Stock Standard Concentration (mg/L)** | **Spike Volume (mL)** | **Final Concentration mg/L** |
| Aluminum | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Antimony | CPI-ICPMS-1B | 5.0 | 5.0 | 0.050 |
| Arsenic | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Barium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Beryllium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Boron | CPI-ICPMS-1B | 50 | 5.0 | 0.10 |
| Cadmium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Calcium | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Chromium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Cobalt | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Copper | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Iron | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Lead | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Lithium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Magnesium | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Manganese | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Molybdenum | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>20 | 5.0<br>2.5 | 0.20 |
| Nickel | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Phosphorous | Spex ZCAL-34 | 1000 | 2.5 | 5.0 |
| Potassium | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Selenium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Silver | CPI-ICPMS-1B | 5.0 | 5.0 | 0.050 |
| Sodium | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>1000 | 5.0<br>2.5 | 5.1 |
| Strontium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Thallium | CPI-ICPMS-1B | 5.0 | 5.0 | 0.050 |
| Tin | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Titanium | CPI-ICPMS-1B<br>Spex ZCAL-34 | 10<br>20 | 5.0<br>2.5 | 0.20 |
| Vanadium | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Zinc | CPI-ICPMS-1B | 10 | 5.0 | 0.10 |
| Sulfur | Spex ZCAL-34 | 1000 | 2.5 | 5.0 |
| Carbon | Spex ZCAL-34 | 2000 | 2.5 | 10 |
| Chloride | Spex ZCAL-34 | 7200 | 2.5 | 36 |

**TABLE 3**

**Method 6020 Initial Calibration Verification Standard**
**Stock: CPI CALLAB-3 Spike Volume: 4.0 mL**
**Final Volume: 500 mL**

| Element | Concentration mg/L | Element | Concentration mg/L |
|---|---|---|---|
| Aluminum | 0.8 | Manganese | 0.08 |
| Antimony | 0.04 | Molybdenum | 0.08 |
| Arsenic | 0.08 | Nickel | 0.08 |
| Barium | 0.08 | Phosphorous | 0.8 |
| Beryllium | 0.08 | Potassium | 0.8 |
| Boron | 0.8 | Selenium | 0.08 |
| Cadmium | 0.08 | Silver | 0.04 |
| Calcium | 0.8 | Sodium | 0.8 |
| Chromium | 0.08 | Strontium | 0.08 |
| Cobalt | 0.08 | Thallium | 0.04 |
| Copper | 0.08 | Tin | 0.08 |
| Iron | 0.8 | Titanium | 0.08 |
| Lead | 0.08 | Vanadium | 0.08 |
| Lithium | 0.08 | Zinc | 0.08 |
| Magnesium | 0.8 | | |

| Table 4 | | | | |
|---|---|---|---|---|
| **Low Level Check Intermediate Standard - Method 6020** | | | **Final Volume = 500 mL** | |
| **Element** | **Stock Standard ID** | **Stock Standard Conc'n (mg/L)** | **Spike Volume (mL)** | **Final Conc'n (mg/L)** |
| Aluminum | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Antimony | CALLAB-3 | 5.0 | 0.5 | 0.005 |
| Arsenic | CALLAB-3 | 10 | 0.5 | 0.01 |
| Barium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Beryllium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Boron | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Cadmium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Calcium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Chromium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Cobalt | CALLAB-3 | 10 | 0.5 | 0.01 |
| Copper | CALLAB-3 | 10 | 0.5 | 0.01 |
| Iron | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Lead | CALLAB-3 | 10 | 0.5 | 0.01 |
| Lithium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.02 | 0.05 |
| Magnesium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Manganese | CALLAB-3 | 10 | 0.5 | 0.01 |
| Molybdenum | CALLAB-3 | 10 | 0.5 | 0.01 |
| Nickel | CALLAB-3 | 10 | 0.5 | 0.01 |
| Phosphorous | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Potassium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Selenium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Silver | CALLAB-3 | 5.0 | 0.5 | 0.005 |
| Sodium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.2 | 0.5 |
| Strontium | CALLAB-3 | 10 | 0.5 | 0.01 |
| Thallium | CALLAB-3 | 5.0 | 0.5 | 0.005 |
| Tin | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.045 | 0.1 |
| Titanium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.02 | 0.05 |
| Vanadium | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.045 | 0.1 |
| Zinc | CALLAB-3<br>Single | 100<br>1000 | 0.5<br>0.02 | 0.05 |

| TABLE 5 | | | |
|---|---|---|---|
| *** Method 6020 Interference Check Solution A**<br><br>**Stock: Spex ZCAL-34 Spike volume: 10 mL**<br>**Bromide 1000 mg/L stock Spike volume: 1.0 mL**<br><br>**Final Volume: 100 mL** | | | |
| **Element** | **Concentration mg/L** | **Element** | **Concentration mg/L** |
| $Br^-$ | 100 | P | 100 |
| Al | 100 | S | 100 |
| Ca | 100 | C | 200 |
| Fe | 100 | $Cl^-$ | 720 |
| K | 100 | Mo | 2.0 |
| Mg | 100 | Ti | 2.0 |
| Na | 100 | | |

*** Make new standard weekly**

| TABLE 6<br>Composition of the ICSAB Standard | | | |
|---|---|---|---|
| Element | Concentration ug/mL | Element | Concentration ug/mL |
| Ag | 0.05 | Na | 100.1 |
| Al | 100.1 | Ni | 0.10 |
| As | 0.10 | Pb | 0.10 |
| B | 0.10 | Sb | 0.05 |
| Ba | 0.10 | Se | 0.10 |
| Be | 0.10 | Si | 0.10 |
| Ca | 100.1 | Sn | 0.10 |
| Cd | 0.10 | Sr | 0.10 |
| Co | 0.10 | Ti | 2.0 |
| Cr | 0.10 | Tl | 0.05 |
| Cu | 0.10 | U | 0.10 |
| Fe | 100.1 | V | 0.10 |
| K | 100.1 | Zn | 0.10 |
| Li | 0.1 | P | 100 |
| Mg | 100.0 | S | 100 |
| Mn | 0.10 | C | 200 |
| Mo | 2.10 | Cl- | 720 |

| Table 7 | | | |
|---|---|---|---|
| **Internal Standard A Stock - Method 6020** | | **Final Volume = 4000 mL** | |
| **Element** | **Stock Standard Concentration (mg/L)** | **Spike Volume (mL)** | **Final Concentration (mg/L)** |
| Lithium | 500 | 80 | 10 |
| Scandium | 1000 | 8.0 | 2.0 |
| Germanium | 10000 | 3.0 | 7.5 |
| Indium | 1000 | 3.0 | 0.75 |
| Thullium | 1000 | 2.0 | 0.5 |

| TABLE 8[1] | | | | | |
|---|---|---|---|---|---|
| **COMMON MOLECULAR ION INTERFERENCES IN ICP-MS** | | | | | |
| **Molecular Ion** | **Mass** | **Element Interferences[2]** | **Molecular Ion** | **Mass** | **Element Interferences[2]** |
| *BACKGROUND MOLECULAR IONS* | | | | | |
| $NH^+$ | 15 | | $^{38}ArH^+$ | 39 | |
| $OH^+$ | 17 | | $^{40}ArH^+$ | 41 | |
| $OH_2^+$ | 18 | | $CO_2^+$ | 44 | |
| $C_2^+$ | 24 | | $CO_2H^+$ | 45 | Sc |
| $CN^+$ | 26 | | $ArC^+$, $ArO^+$ | 52 | Cr |
| $CO^+$ | 28 | | $ArN^+$ | 54 | Cr |
| $N_2^+$ | 28 | | $ArNH^+$ | 55 | Mn |
| $N_2H^+$ | 29 | | $ArO^+$ | 56 | |
| $NO^+$ | 30 | | $ArOH^+$ | 57 | |
| $NOH^+$ | 31 | | $^{40}Ar^{36}Ar^+$ | 76 | Se |
| $O_2^+$ | 32 | | $^{40}Ar^{38}Ar^+$ | 78 | Se |
| $O_2H_+$ | 33 | | $^{40}Ar_2^+$ | 80 | Se |
| $^{36}ArH^+$ | 37 | | | | |
| *MATRIX MOLECULAR IONS – Chloride* | | | | | |
| $^{35}Cl0^+$ | 51 | V | $^{37}Cl0H^+$ | 54 | Cr |
| $^{35}Cl0H^+$ | 52 | Cr | $^{35}Cl0^+$ | 51 | V |
| $^{37}Cl0^+$ | 53 | Cr | $^{35}Cl0H^+$ | 52 | Cr |
| $Ar^{35}Cl^+$ | 75 | As | $Ar^{37}Cl^+$ | 77 | Se |
| *MATRIX MOLECULAR IONS – Sulfate* | | | | | |
| $^{32}SO^+$ | 48 | | $^{34}SOH^+$ | 51 | V |
| $^{32}SOH^+$ | 49 | | $SO_2^+$, $S_2^+$ | 64 | Zn |
| $^{34}SO^+$ | 50 | V, Cr | | | |
| $Ar^{32}S^+$ | 72 | | $Ar^{34}S^+$ | 74 | |
| *MATRIX MOLECULAR IONS – Phosphate* | | | | | |
| $PO^+$ | 47 | | $PO_2^+$ | 63 | Cu |
| $POH^+$ | 48 | | | | |
| $ArP^+$ | 71 | | | | |
| *MATRIX MOLECULAR IONS – Group I, II Metals* | | | | | |
| $ArNa^+$ | 63 | Cu | $ArCa^+$ | 80 | |
| $ArK^+$ | 79 | | | | |
| *MATRIX OXIDES[3]* | | | | | |
| TiO | 62-66 | Ni, Cu, Zn | MoO | 108-116 | Cd |
| ZrO | 106-112 | Ag, Cd | | | |

[1] *Table reference from Method 200.8, Section 13.2.6*

[2] *Method elements or internal standards affected by the molecular ions.*

[3] *Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.*

| TABLE 9<br>RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED [1] | | | |
|---|---|---|---|
| Isotope | Element of Interest | Isotope | Element of Interest |
| **27** | Aluminum [2] | 80,**78**,**82**,**76**,**77**,74 | Selenium |
| **121**,123 | Antimony [2] | **107**,**109** | Silver [2] |
| **75** | Arsenic [2] | **23** | Sodium [2] |
| 138,137,136,**135**,134,132,130 | Barium [2] | 203, **205** | Thallium [2] |
| 9 | Beryllium [2] | **51**,**50** | Vanadium [2] |
| **114**,112,**111**,110,113,116,106, **108** | Cadmium [2] | **66**, **67**, **68** | Zinc [2] |
| 42,**43**,**44**,46,48 | Calcium [2] | 83 | Krypton |
| **52**,**53**,**50**,54 | Chromium [2] | 72 | Germanium |
| **59** | Cobalt [2] | 139 | Lanthanum |
| **63**,**65** | Copper [2] | 140 | Cerium |
| **56**,**54**,**57**,58 | Iron [2] | 129 | Xenon |
| **206**,**207**,**208** | Lead [2] | **118** | Tin |
| 24,**25**,**26** | Magnesium [2] | 105 | Palladium |
| **55** | Manganese [2] | 47,**49** | Titanium |
| 202,**200**,199,201 | Mercury | 125 | Tellurium |
| 98,96,92,**97**,94 | Molybdenum | 69 | Gallium |
| 58,**60**,62,**61**,64 | Nickel [2] | 35,37 | Chlorine |
| **39** | Potassium [2] | | |

[1] *From Method 6020 CLP-M, Table 9*

[2] *Element approved for ICP-MS determination by SW846 Method 6020 CLP-M*

NOTE: Isotopes recommended for analytical determination are **bold**.

| TABLE 10 | | | |
|---|---|---|---|
| RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED | | | |
| **Rare Earth Elements** | **ICPMS Preferred Mass** | **Elemental Equations** | **Additional Masses** |
| Lanthanum | 138.906 | | |
| Cerium | 139.905 | | |
| Praseodymium | 140.907 | | |
| Neodymium | 141.908 | -0.125266 * $^{140}$Ce | 142.910, 144.912 |
| Samarium | 151.920 | -0.012780 * $^{157}$Gd | 144.912 |
| Europium | 152.929 | | |
| Gadolinium | 157.924 | -0.004016 * $^{163}$Dy | 156.934 |
| Terbium | 158.925 | | |
| Dysprosium | 163.929 | -0.047917 * $^{166}$Er | |
| Holmium | 164.930 | | |
| Erbium | 165.930 | | |
| Thulium | 168.934 | | |
| Ytterbium | 173.939 | -0.005935 * $^{178}$Hf | 171.937 |
| Lutetium | 174.941 | | |
| **Other Elements** | | | |
| Boron | 11.009 | | |
| Calcium | 43.956 | | |
| Cesium | 132.905 | | |
| Gallium | 68.926 | | |
| Germanium | 71.922 | | |
| Gold | 196.967 | | |
| Hafnium | 177.944 | | 176.944 |
| Holmium | 164.930 | | |
| Iridium | 192.963 | | |
| Lithium | 7.016 | | |
| Tungsten | 183.951 | -0001242* $^{189}$Os | |
| Uranium | 238.050 | | |
| Yttrium | 88.905 | | |
| Zirconium | 238.050 | | |
| Niobium | 92.906 | | |
| Palladium | 104.905 | | |
| Phosphorus | 30.994 | | |
| Platinum | 194.965 | | |
| Rhenium | 186.965 | -0.099379 * $^{189}$Os | |
| Rhodium | 102.905 | | |
| Rubidium | 84.912 | | |
| Ruthenium | 101.904 | -0.045678 * $^{105}$Pd | |
| Scandium | 44.956 | | |
| Strontium | 87.906 | | |
| Tantalum | 180.948 | | |
| Tellurium | 127.905 | -0.072348 * $^{129}$Xe | |
| Thorium | 232.03 | | |

**TABLE 11**
**ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS**

| Element | Elemental Equation | Note |
|---|---|---|
| Al | (1.000) ($^{27}$C) | |
| Sb | (1.000) ($^{121}$C) | |
| As | (1.000) ($^{75}$C) - (3.1278)[$^{77}$C) - (1.0177)($^{78}$C)] | Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank. |
| Ba | (1.000) ($^{137}$C) | |
| Be | (1.000) ($^{9}$C) | |
| Cd | (1.000) ($^{111}$C) - (1.073) [($^{108}$C) - (0.712) ($^{106}$C)] | Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present. |
| Cr | (1.000) ($^{52}$C) | In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank. |
| Co | (1.000) ($^{59}$C) | |
| Cu | (1.000) ($^{63}$C) | |
| Pb | (1.000) ($^{206}$C) + (1.000) ($^{207}$C) + (1.000) ($^{208}$C) | Allowance for isotopic variability of lead isotopes. |
| Mn | (1.000) ($^{55}$C) | |
| Mo | (1.000) ($^{98}$C) - (0.146) ($^{99}$C) | Isobaric elemental correction for ruthenium. |
| Ni | (1.000) ($^{60}$C) | |
| Se | (1.000) ($^{82}$C) | Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction. |
| Ag | (1.000) ($^{107}$C) | |
| Tl | (1.000) ($^{205}$C) | |
| Th | (1.000) ($^{232}$C) | |
| U | (1.000) ($^{238}$C) | |
| V | (1.000) ($^{51}$C) - (3.127) [($^{53}$C) - (0.113) ($^{52}$C)] | Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ratio may be determined from the reagent blank. |
| Zn | (1.000) ($^{66}$C) | |

* Method elements or internal standards affected by the molecular ions.

IH Air Monitoring Method for Polychlorobiphenyls

[Method No(s). NIOSH 5503]

# Title: IH Air Monitoring Method for Polychlorobiphenyls [Method No(s). NIOSH 5503]

**Approvals (Signature/Date):**

| | |
|---|---|
| 6/21/2015 | 6/26/2015 |
| Stephanie Stimson — Date | Lisa Maycock — Date |
| Industrial Hygiene Department Manager | Environmental Health & Safety Coordinator |
| 6/23/2015 | 6/21/2015 |
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## 1.0 Scope and Application

**1.1** This method is used for the determination of Polychlorobiphenyls (PCBs) in Air using NIOSH 5503.

**1.2** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

## 2.0 Summary of Method

**2.1** This standard operating procedure (SOP) is appropriate for the analysis of PCBs, such as Aroclors, on 13-mm glass fiber filters in series with 150-mg Florisil tubes. The appropriate sampling information for each chemical of interest is displayed in **Attachment 1.**

**2.2** The NIOSH method has been modified for use with a different analytical column and different instrument parameters.

## 3.0 Definitions

**3.1** Calibration Standard (CAL) – A solution prepared from the primary dilution standard solution(s) or stock standard solutions. Used to calibrate the instrument.

**3.2** Initial Calibration Standards (ICAL) – A series of CAL solutions used to initially establish instrument calibration and develop calibration curves.

**3.3** Continuing Calibration Verification Standard (CCV) – A CAL solution which is analyzed after every tenth field sample analysis, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous field samples analyzed. The CCV is at the highest calibration curve point concentration and may be from the same source as the calibration standards.

**3.4** Initial Calibration Verification (ICV) – A solution, which is analyzed after the initial standards and/or at the beginning of an analysis that is at the reporting limit concentration. A standard solution (or set of solutions) used to verify calibration standard levels. The ICV shall be prepared independently from the calibration standards.

**3.5** Laboratory Control Spike and Duplicate (LCS/LCSD) – Sorbent or media, to which a known quantity of analyte is added in the laboratory. The LCS and LCSD are analyzed exactly like the sample. LCS: A matrix-based reference material with an established concentration obtained from a source independent of the instrument calibration and traceable to NIST or other similar references materials. The LCS/LCSD is carried through the entire procedure from sample preparation through analysis as if it were a field sample. The purpose of the LCS/LCSD is the evaluate bias of the method.

**3.6** Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB) – The initial calibration blank and continuing calibration blank are zero concentration standards. They are in

essence the solvent/reagent solution (without analyte) in which instrument calibration standards are prepared. The initial calibration blank and continuing calibration blank are not subject to all of the handling steps applied to samples.

**3.7** Method Blank (MB) – Sorbent or media, that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples.

**3.8** Independently Prepared Calibration Standard – A standard prepared from a reference material other than that used for calibration. When using neat materials this may be a standard prepared from the same starting material but using a different dilution technique, i.e. prepared in a different final volume so as to utilize a different spike amount, prepared by a separate analyst using the same dilution or different dilution technique or from a stock solution having a different manufacturer or different manufacturer's lot identification.

**3.9** Refer to the Quality Assurance Manual for additional definitions and terms not defined in this section.

## 4.0 Interferences

**4.1** Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it may need to be followed by the analysis of solvent to check for cross-contamination.

## 5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe and closed-heeled, nonabsorbent shoes are a minimum PPE requirement.

### 5.1 Specific Safety Concerns or Requirements

**5.1.1** The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

**5.1.2** There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

**5.1.3** Use caution when scoring the glass end of the tube. Protect hand from cuts by using safe glass cutting techniques and wearing cut proof gloves as needed.

**5.1.4** The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: PCBs. Pure standard materials and stock standard solutions of these compounds must be handled in a hood wearing all appropriate PPE.

**I.** Additional information about the above listed analytes and all other compounds analyzed by this method is available via a MSDS. All MSDSs are available on the company's intranet site OASIS

## 5.2 Primary Materials Used

**5.2.1** The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material | Hazards | Exposure Limit (1) | Signs and symptoms of exposure |
|---|---|---|---|
| Hexane | Flammable<br>Irritant | 500 ppm-TWA | Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes. |
| 1 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |

# 6.0 Equipment and Supplies

## 6.1 Instrumentation

- Agilent 6890 Gas chromatograph with dual electron capture detectors (ECD) – An analytical system complete with a temperature-programmable gas chromatograph suitable for split / splitless injection and all required accessories, including syringes, analytical columns and gases. The capillary column should be directly coupled to the source.
- Dual analytical columns; Primary Column – RTX-CLPesticides, 15-m x 0.32-mm ID x 0.5-µm film thickness fused-silica capillary column. Secondary Column – RTX-CLPesticides2, 15-m x 0.32-mm ID x 0.25-µm film thickness fused-silica capillary column.
- Hewlett Packard Chem Station (Enviroquant).

## 6.2 Supplies

- Syringes, various sizes.
- Pipettes, various sizes
- 4-mL through 12-mL glass vials and caps.
- Gas-tight syringes.

- Vortex unit or mechanical shaker.
- 150-mg Florisil tube, SKC 226-39, or equivalent.
- 13-mm Glass Fiber Filter available from SKC, Zeflon, etc.
- Swinnex filter cassettes available from SKC, (catalog #225-32), or equivalent.
- Autosampler vials, caps and inserts.
- Tygon tubing, various sizes.

## 7.0 Reagents and Standards

**7.1** Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit use without lessening the accuracy of determination.

**7.2** Life of Standards and Reagents: Reference the current version of SOP PE-QAD-013.

**7.3** Hexane, pesticide residue analysis grade or equivalent.

Storage Requirements: Flammables cabinet.

**7.4** PCB Stock Standards

Storage Requirements: Store the stock standard in Teflon-sealed screw-cap bottles at 0 – 6°C and protect from light.

**7.5** Second source reagents of high purity should be obtained and analyzed to verify each primary source, if available.

**7.6** Certificates of Analysis should be obtained for every standard.

## 8.0 Sample Collection, Preservation, Shipment and Storage

**8.1** Active Sampling with the 13-mm glass fiber filters in series with 150-mg Florisil Tubes.

**8.1.1** Calibrate each personal sampling pump with a representative sampler in line.

**8.1.2** Break the ends of the sampler immediately before sampling, using a tube tip breaker supplied by TestAmerica. Alternatively, use a carbide blade or a metal file to score the glass ends of the tube. Place the red caps on the tube and snap off the tube ends. Keep the red caps in order to cap the tubes after sampling is finished. Attach sampler to personal sampling pump with flexible tubing. Connect the tube to the Swinnex filter cassette using the short piece of tubing. Air should be drawn into the cassette first and then through the tube.

**8.1.3** Sample at an accurately known flow rate for an appropriate total sample size. Refer to **Attachment 1** in this SOP or to NIOSH 5503 for exact sampling parameters (flow rates, duration of sampling, humidity, temperature concerns, etc.). After sampling the filter should be removed from the cassette and placed in a vial.

**8.1.4** Sample should be transported sealed to the laboratory following sampling. Upon sample receipt by the laboratory, samples will be stored at a refrigerated temperature of 2° - 6° C prior to extraction.

**8.1.5** The samples are stable for at least 60 days.

**8.2** Field Blanks. Clients should include at least one field blank with each sample set.

## 9.0 Quality Control

| QC Performed | Frequency | Acceptance Criteria | Correction Action |
|---|---|---|---|
| Minimum of 3 Point Calibration Curve, 6 points standard | When an opening or continuous CCV fails. | Coefficient of Determination $r^2 \geq 0.990$ | 1. Re-inject curve<br>2. Prepare new standards<br>3. Perform maintenance, if needed. |
| Primary Source CCV at Highest Calibration Concentration | Every 10 field samples and at the end of the analysis. | Within 75 – 125% of expected value | 1. Re-inject CCV<br>2. Re-inject curve<br>3. Prepare new standards<br>4. Run new curve<br>5. Perform maintenance, if needed. |
| Independently Prepared or Second Source ICV at Reporting Level | Immediately following calibration standards and also at beginning of analysis if re-calibration is not being performed | Within 50 – 150% of expected value | 1. Re-inject ICV<br>2. Re-inject curve<br>3. Prepare new standards<br>4. Run new curve<br>5. Perform maintenance, if needed. |
| Method Blank | One per batch of samples | < Report Limit | 1. Subtract blank from client and QC samples |
| LCS/LCSD (Independently Prepared or Second Source) | Every batch of samples | Within 75 – 125% recovery and ≤ 30% RPD or historical limits. | 1. Re-inject LCS/LCSD and/or qualify and report<br>2. Perform maintenance, if needed. |
| Calibration Blank | Beginning, every 10 field samples, and end of each analysis day, before or after ICV | < Report Limit is expected | 1. Subtract, if necessary and/or qualify and report<br>2. Recalibrate and reanalyze all samples since last compliant calibration blank, if needed. |

**9.1** It is the responsibility of the analyst to perform the analysis according to this SOP and complete the documentation required for review.

**9.1.1** Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this method perform extraction and preparation of extracts.

**9.2** Document all maintenance procedures in the instrument logbook assigned to each instrument.

**9.3 Method Blank and Calibration Blank**

**9.3.1** A method blank must be analyzed with each batch of samples.

**9.3.2** Most analytes found in the method blank will generally be below the reporting levels. However, if there is a value above the reporting level, then subtract the blank from the QC samples and the client's samples. If the method blank yields a result above the reporting level, but the client's samples do not show a background or the QC samples do not have an unusually high recovery, then the contamination is most likely isolated to that blank and further investigation should occur and an appropriate decision be made after further investigation. An appropriate decision may include, but not be limited to, re-analyzing the blank, extracting one or two additional blanks, analyzing an additional aliquot of the solvent used for extraction to check for contamination, ignoring the blank if it is obviously the only sample contaminated, etc.

**9.3.3** A calibration blank is used in establishing the analytical curve and to determine background levels of the solvents/reagents. Beginning, every 10 field samples, and end of each analysis day, before or after ICV.

## 10.0 Procedure

### 10.1 Sample Preparation

**10.1.1** Filter and Tube sample preparation.

**10.1.2** The filter portion of the sample should arrive at the laboratory in a vial. If it arrived in the Swinnex cassette, then document it in the prep batch, and transfer the filter to a vial. Swinnex cassettes can be rinsed and or sonicated with hexane, dried, and used again.

**10.1.3** Prepare another vial to hold the contents of the back tube section. The front section of the tube will be transferred to the same vial containing the filter. Therefore the sample will have two vials total, and both will be analyzed.

**10.1.4** Remove the glass wool from the tube and pour the contents of each section of each tube into the appropriately labeled vials.

**10.1.5** Slowly add 2 mL of hexane into each vial and cap them.

**10.1.6** Allow samples to stand 30 minutes with occasional agitation. The occasional agitation can be accomplished by shaking by hand for ~5 seconds every 10 to 15 minutes during 30-minute desorption procedure.

**10.1.7** Immediately following the desorption process, transfer some of the extract into an autosample vial containing an insert, and then optionally transfer the rest of the extract to a separate autosampler vial. The separate autosampler vial should be stored in the freezer in case the samples need to be re-analyzed. The remainder of the extract may remain in the original extraction vial, instead of being transferred, as no significant difference has been observed between initial analysis and reruns when stored in this manner.

**10.1.8** Dilutions – If the response for any peak exceeds the calibration range, make the appropriate dilution and reanalyze.

**10.1.9** Spike Solution Preparation.

**I.** The spiking solution is prepared as described in the table in section 10.2.3.

Therefore, 2 µL of spiking solution injected into Florisil in a vial would yield a spike of ~ 2 times the expected reporting level.

**10.1.10** In addition to spiking the sorbent, it is often advantageous to spike a 2 mL aliquot of fresh desorption solvent with the same spike stock that was used to spike the sorbent. By doing this, the spiked solution can be used as a "reference" that tests the accuracy of the spike stock itself and the actual spiking technique, however, at this time, references are not required.

## 10.2 Calibration

**10.2.1** According to the AIHA Policies manual, a minimum of 3 calibration standards need to be analyzed to establish a new calibration curve. A calibration blank must also be analyzed with each analysis.

**10.2.2** There are several primary sources available, depending on which PCBs are of interest. Generally, a primary source will contain 1 or 2 PCBs at 1000 µg/mL each. Calibrate with the PCB of interest, if known. Default to calibrating using PCB 1242, if no PCB of interest is provided by the client. For this standard preparation example, PCB 1242 will be shown using a primary source containing PCB 1242 (µg/2 mL = µg/sample).

| Standard ID | Preparation of Standard | PCBs (µg/mL) |
|---|---|---|
| Intermediate #1 | 200 µL of primary source + 1800 µL of hexane | 100 |
| Intermediate #2 | 300 µL of "Intermediate #1" + 2700 µL of hexane | 10 |
| #1 (CCV) | 600 µL of "Intermediate #2" + 2400 µL of hexane | 2 |
| #2 | 200 µL of "Intermediate #2" into 1800 µL desorption solvent | 1 |
| #3 | 100 µL of "Intermediate #2" into 1900 µL desorption solvent | 0.5 |

| | | |
|---|---|---|
| #1 x 10 | 200 µL of "#1" into 1800 µL desorption solvent | 0.2 |
| #2 x 10 | 200 µL of "#2" into 1800 µL desorption solvent | 0.1 |
| #3 x 10 | 200 µL of "#3" into 1800 µL desorption solvent | 0.05 |

**10.2.3** An independently prepared standard (i.e. prepared independently from the calibration standards from neat materials or prepared with a standard from a stock solution having a different manufacturer's lot identification) should be made at the same concentration as the reporting level and/or lowest point of the calibration curve and should be within ± 50% of the expected value. This standard is used as the ICV.

| Standard ID | Preparation of Standard | PCBs (µg/mL) |
|---|---|---|
| Intermediate #1 (Spike) | 200 µL of secondary source + 1800 µL of hexane | 100 |
| Intermediate #2 | 200 µL of "Intermediate #1" + 1800 µL of hexane | 10 |
| #3 | 100 µL of "Intermediate #2" into 1900 µL desorption solvent | 0.5 |
| #3 x 10 (ICV) | 300 µL of "#3" into 2700 µL desorption solvent | 0.05 |

**10.2.4** Pattern recognition of other PCBs not used for calibration are also run at the reporting level concentration. Should a hit of a PCB other than the calibrated PCB be present, the highest calibration standard will be run and utilized for a one-point calibration curve. This will be applied toward the sample hit in question, and used to quantify the targeted PCB. Example preparations for pattern recognition standards are as follows, with each Aroclor prepared independent of one another except Aroclors 1016 and 1260 and Aroclors 1221 and 1254, which are combined into those pairings.

| Standard ID | Preparation of Standard | PCBs (µg/mL) |
|---|---|---|
| Intermediate #2 | 20 µL of each source + 1980 µL of hexane<br>or<br>20 µL of each source + 1960 µL of hexane for paired Aroclors not already combined | 10 |
| #1 (if needed) | 400 µL of "Intermediate #2" into 1600 µL desorption solvent | 2.0 |
| #3 x 10 (Pattern recognition standard) | 10 µL of "Intermediate #2" into 1990 µL desorption solvent | 0.05 |

## 10.3 Sample Analysis

**10.3.1** Analytical Conditions (may be modified, if necessary).

Agilent 6890 Gas chromatograph with dual electron capture detectors (ECD)

| | |
|---|---|
| Detector Temperatures: | 320°C |
| Injection Port Temperature: | 220°C |

| | |
|---|---|
| Oven Temperature: | 100°C for 1.0 minutes, 30 °C/minute to 220 °C (hold 1.0 min) then 30 °C/minute to 235 °C (hold 3.0 min) then 30 °C/minute to 300 °C (hold 3.34 min). |
| Run Time: | 15.01 minutes |
| Equilibration Time: | 0.50 min |
| Injection Volume: | 2 µL |
| Carrier Gas: | Helium |
| Pressure: | ~7.4 psi |
| Injection Type: | Pulsed Splitless |
| Pulse: | Time 0.20 min, pressure 40.0 psi |
| Purge: | Purge on at 0.7minutes, off at 0.0 minutes, flow 20 mL/min |
| Gas Saver: | Time: 2.00 min, flow 20 mL/min |
| Column Flows: | 2.3 mL/min |
| Average Column Velocities: | 47 cm/sec |
| Detector Flow: | Combined flow: 30.0 mL/min |

**10.3.2**

| Quantitation & Confirmation |
|---|
| ICB or CCB |
| ICAL/CCB and/or ICV/CCB |
| Pattern Recognition Standards – Reporting level Aroclor standards other than the one used for calibration |
| CCB |
| Method Blank back section |
| Method Blank front section |
| LCS |
| LCSD |
| CCB |
| Sample back sections 1 - 10 |
| CCV |
| CCB |
| Sample front sections 1 – 10 (filter + front of tube) |
| CCV |
| CCB |

**10.4** Figure 1 and Figure 2 in Attachment 2 contain representative chromatograms of a standard and low spike containing the compounds listed in this SOP.

**10.5** An instrument that continually fails to properly perform is tagged with an “out of service” label until it has been serviced or maintenance is performed such that its proper function has been restored.

## 11.0 Calculations / Data Reduction

### 11.1 Reporting Results

**11.1.1** Glass Fiber Filters and Florisil Tubes.

**I.** Report the blank sorbent results as the method blank results.

**II.** Subtract the applicable method blank sections from the QC and the client's samples.

**11.1.2** Results for samples and all QC samples are reported to a minimum of two significant figures.

**11.1.3** A comment is included on the certification summary page of the client's report that the results were blank corrected. Samples and QC must be flagged if not blank corrected.

**11.1.4** Quantify PCB mixtures by comparison of the retention times, peak height, and/or peak area of 5 GC peaks with the corresponding peaks in the best-matching standard. If knowledge of the PCB of interest is not provided by the client or the analyst is not able to discern the PCB present, use PCB 1242 for reporting early eluting PCBs and either PCB 1254 or PCB 1260 as appropriate for reporting late eluting PCBs.

**11.1.5** Any manual integrations must be performed and documented in accordance with PE-QAD-009, Manual Integration/Data Integrity.

**11.2 Data Qualifiers**

**11.2.1** Use data qualifiers to qualify analytical results as needed to represent events that occurred during analysis that do not conform to SOP or Method criteria.

**11.2.2** Refer to SOP PE-QAD-018, Use of Data Qualifiers for the proper qualifiers to use and the procedure for completing the required Corrective Action Report.

**11.3 Accuracy**

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

**11.4 Precision (RPD)**

$$\textbf{Relative Percent Difference (RPD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

**11.5** Filter/Tube Samples: Determine the mass, µg (corrected for desorption efficiency) of each analyte found in the filter/tube sample front ($W_f$) and tube back ($W_b$) sorbent sections and the media blank sorbent sections. All calculations and desorption efficiency corrections are performed by the laboratory information management system.

$$\mu g/\text{sample} = (Wf - Bf) + Wb - Bb)$$

Where,

Wf = µg (corrected for desorption efficiency) front sorbent section
Wb = µg (corrected for desorption efficiency) back sorbent section
Bf = Media blank result front section

Bb = Media blank result back section

**11.6** Calculate the analyte air concentration as follows:

$$mg/m^3 = \frac{Ma}{R * T}$$

Where,

Ma = µg/sample
R = flow rate
T = # of minutes of sampling period

## 12.0 Method Performance

### 12.1 Method Detection Limit Study (MDL)

**12.1.1** The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

**12.1.2** MDL is also sometimes referred to as Limit of Detection (LOD). (NELAC Requirement)

**I.** "The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2 – 3X the LOD for single analyte tests and 1 – 4X the LOD for multiple analyte tests. The verification must be performed on every instrument that is to be used for analysis of samples and reporting of data." (2003 NELAC Standards, C.3.1.b)

**12.1.3** An important characteristic of expression of sensitivity is the difference in the MDL (LOD) and the Quantitation Limit or Reporting Limit (sometimes referred to as the Limit of Quantitation (LOQ)). (NELAC Requirement)

**I.** "The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1 – 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objective for accuracy. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ." (2003 NELAC Standards, C.3.2.c)

**12.1.4** Additional information can be found in the QA Manual.

### 12.2 Initial Desorption Efficiency/Validation Study

**12.2.1** Prior to ever analyzing a sample for a specific analyte, the DE for each analyte must be determined within the reporting range.

**12.2.2** To perform the initial DE study, prepare and analyze 3 replicates at a concentration at the expected reporting level, 3 replicates at a mid-level and 3 replicates at the highest level. Prepare three (3) media blanks to be analyzed with the DE spikes.

**12.2.3** Desorb the spikes and analyze together with calibration standards.

**12.2.4** Desorption efficiency is the ratio of the amount of the analyte recovered (µg) to the amount of the analyte injected (µg). The initial DE and future % recoveries should be control charted to track historical recoveries. Once enough historical data is generated, then acceptance criteria and acceptable ranges can be updated and perhaps tightened, if appropriate. In general, the largest variation occurs at the low end of the calibration range for spikes that are made near the reporting level.

**12.3 <u>Reporting Level Verification</u>**

**12.3.1** The reporting level must be established. This is accomplished by fortifying a media spike at a concentration at, or below, the expected reporting level, which is typically equal to the lowest standard concentration. Perform all calculations defined in the method and report the concentration values in the appropriate units.

**12.3.2** The reporting level media spike should be verified initially, annually, or if there is a significant change in the background or instrument response.

**12.3.3** Percent recovery for the reporting level verification spike should be within 50 – 150% of the expected value. If the percent recovery is outside this range, then an additional spike should be prepared at a higher level, which will result in a higher reporting level.

**12.4 <u>Training Requirements</u>**

**12.4.1** Refer to the QA Manual or to SOP PE-QAD-008 – Personnel Certification and Training. At a minimum before an analyst can perform the method independently, they must have:

a. Read the analytical method(s);
b. Read the applicable SOP(s); and
c. Acceptably performed and documented the data for four LCS' (normally two LCS/LCSD sets).

**12.4.2** TestAmerica's participation in the American Industrial Hygiene Association program requires the additional performance as listed in section 12.5 – "Demonstration of Capabilities".

**12.4.3** Additionally, training must be documented to include a description of the training content/activities and duration of the program provided to the analyst. The training course must last a minimum of 20 business days per the AIHA requirements. No independent work may be done prior to the completion of the training program.

## 12.5 Demonstration of Capabilities

**12.5.1** Refer to the QA Manual for general procedures and any specific concentrations that must be used.

**12.5.2** A Demonstration of Capability form shall be completed every six months per AIHA.

**12.5.3** **Initial Demonstration of Capability (IDOC)**: Prior to reporting any data, each analyst must have on file with the QA office information demonstrating proficiency with the analysis technique. Both precision and accuracy are measured for the target analytes. Make four replicate analyses of a daily working standard having a concentration between 2 – 5 times the RL for each analyte (generally at the LCS level). Calculate the average (X) and standard deviation (s). Where the LCS/LCSD replicates are utilized for IDOC evaluation, those same pass/fail control limits will demonstrate accuracy and precision capability.

**I.** If an analyst performs the desorption efficiency series of spikes, that analyst does not need to perform the IDOC in addition to the DE study. The DE study satisfies the IDOC.

**II.** An analyst also must be "authorized" to perform the analysis per AIHA. The approval is documented using the "Demonstration of Capability Authorization/Certification Statement", form PX-QAD-005. This document must be in the analyst's training file before they can analyze industrial hygiene samples.

**12.5.4** **Ongoing Demonstration of Capability:** Every six months an analyst must demonstrate ongoing proficiency. This can be accomplished through: a) acceptable analysis of a Performance Testing (PT) sample; b) by acceptable analysis of at least 2 pairs of LCS/LCSD that were analyzed during the six month period; or c) by repeating the IDOC as described in this SOP.

**I.** The acceptable demonstration for "b" or "c" is by acceptably performing and documenting the data of four LCS (normally defined as two LCS/LCSD sets), its approval by the respective department manager, and by approval of the QA Department. Acceptable demonstration is added to the analyst's training file.

**II.** Another "Demonstration of Capability Authorization/Certification Statement" must also be completed.

## 12.6 Control Limits (Procedure/Method Acceptance Criteria)

**12.6.1** Once control limits have been established (in-house or by method), they are verified, reviewed, and updated if necessary on an annual basis unless the method or regulatory authority requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**12.6.2** More information concerning Control Limits can be found in the QA Manual and the SOP PE-QAD-001 Control Limits and Statistical Process Control.

**12.6.3** Internal Quality Control Procedures. As part of the quality assurance program, the laboratory shall adhere to all stated QA/QC requirements in the method used. Any deviations from the method shall be documented. Deviations that result in nonconforming work shall be evaluated. The following QC checks will be performed per batch of samples:

**12.6.4** Accuracy and bias. Accuracy studies are performed to determine how close a measurement comes to an actual or a theoretical value. Accuracy can be expressed as percent recovery and evaluated by analysis of laboratory control samples (LCS). Bias is a systematic error manifested as a consistent positive or negative deviation from the true value. The bias can be found in the original validated method by NIOSH.

**12.6.5** Precision. Precision is evaluated by the reproducibility of analyses and it is commonly expressed as a relative percent difference. It can be evaluated by the analysis of laboratory control sample and laboratory control sample duplicate (LCS/LCSD).

**12.6.6** Blank sampling media and analytical reagents will be analyzed with each batch of samples. At least on field blank should be used for each day of field sampling, shipped and analyzed with each group of samples. The field blank is treated identically to the samples except that no air is drawn through the tube.

**12.6.7** Reporting Field Blanks. The final report shall state the measured quantitative result of the field blanks submitted to the laboratory. Also, the laboratory should disclose whether or not the sample results have been corrected for contamination based on the field blank or other analytical blank.

**12.7 <u>Measurement Uncertainty (NELAC/AIHA Requirement)</u>**

**12.7.1** Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurement” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**12.7.2** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 ± 0.5 mg/L.

**12.7.3** Refer to the Quality Assurance Manual for further discussion of uncertainty.

## 13.0 Pollution Control

**13.1.1** It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 14.0 Waste Management

**14.1** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Section 13 – Waste Management and Pollution Prevention of the Corporate Environmental Health and Safety Manual (CW-E-M-001) or the current Waste Management SOP.

**14.2** The following waste streams are produced when this method is carried out:

**14.2.1** **Autosampler vial waste** (Open Top Metal Drum): The used vials will be collected and stored in labeled containers located in the fume hood cabinets. When the containers are full, they will be transported in the same container to the storage area. The vials will then be safely and gently transferred to the drum, via a funnel to minimize fumes during removal of the drum lid, ensuring no vial breakage.

**14.2.2** **Mixed solvent waste** (Closed Top Metal Drum): Solvent waste is stored in appropriately labeled hazardous waste containers in satellite storage areas throughout the lab, and should be emptied as often as possible. The waste should be transferred to the storage area in these exact same containers. The snorkel valve on the fume hood exhaust tubing should be turned 90 degrees clockwise to direct flow to the snorkel. The drum openings should then be opened with a wrench. The carboy placed over one opening and the snorkel placed over the other opening. The valve for the carboy should then be opened thereby transferring the contents of the carboy into the waste drum.

**14.2.3** **Expired primary and working standards:**

- **I.** Low concentration (<1000 PPM): Dispose of in the solvent waste stream.
  - a. If the standard is prepared in water, dispose in the acidic waste stream.
- **II.** High concentration: (>1000 PPM) Lab pack.
- **III.** Dioxin Precursors (>50 PPM). Lab Pack
  - **1.** PCBs

**14.3** Any waste, which does not fit into any of the waste streams or cannot be disposed of in any of the other drums, should be brought directly to the department and/or waste manager's attention.

## 15.0 References / Cross-References

### 15.1 Test Methods

- NIOSH 5503, Polychlorobiphenyls in Air, Issue 2, 15 August 1994.
- Method TO-10 A, Determination of Pesticides and PCBS in Ambient Air Using Low Volume PUF Sampling Followed by Gas Chromatographic/Multi-Detector (GC/MD) Detection, January 1999.

**15.2** TestAmerica Laboratories, Inc.

- Environmental Health and Safety Manual, CW-E-M-001.

**15.3** TestAmerica Phoenix Policies and Procedures

- TestAmerica – Phoenix, Quality Assurance Manual, PX-QAD-011.
- SOP PE-SFT-001 - Laboratory Waste Disposal.
- SOP PE-QAD-008 – Personnel Certification and Training.
- PE-QAD-009 Manual Integrations
- PE-QAD-022 Good Calibration Practices
- PE-QAD-001 Control Limits and Statistical Process Control
- PE-QAD-013 Reagent and Standard Prep

**15.4** AIHA Policies for Laboratory Quality Assurance Programs, September 13, 2011.

## 16.0 Method Modifications:

| Item | Method | Modification |
|---|---|---|
| 1. | NIOSH 5503 | Dual column, dual detector analysis for confirmation utilizing the following analytical columns: Dual analytical columns; Primary Column – RTX-CLPesticides, 15-m x 0.32-mm ID x 0.5-µm film thickness fused-silica capillary column. Secondary Column – RTX-CLPesticides2, 15-m x 0.32-mm ID x 0.25-µm film thickness fused-silica capillary column. (Use of updated columns is not considered a modification to the method per AIHA.) |
| 2. | NIOSH 5503 | The desorption solvent volume has been modified from 5 mL front section; 2ml back section to 2 mL front section; 2ml back section. Desorption efficiency studies have been performed for validation. |
| 3. | NIOSH 5503 | Desorption time has been increased to 30 minutes from 20 minutes. Desorption efficiency studies have been performed for validation. |

## 17.0 Attachments

Attachment 1: Sampling Information

Attachment 2: Figure 1 / Figure 2 Representative Chromatographs

## 18.0 Revision History

Revision 0, dated April 3, 2008
- Integration of TestAmerica and STL operations.

Revision 1, dated January 28, 2011
- Conversion to the TestAmerica Laboratories, Inc. SOP template.

Revision 2, dated February 21, 2013
- Updated definition for Independently Prepared Standard
- Section 5.1.3 – added cut proof gloves to section
- Removed Data Review Checklist
- Added references to SOP PE-QAD-001 Control Limits and Statistical Process Control
- Updated AIHA policy reference

Revision 3, dated June 26, 2015
- Section 6.1 – Added Columns
- Section 9.0 – Revised QC Table
- Section 10.1.7 – Revised section to include storage of extracts
- Section 10.2.2/3 – Revised standard tables
- Section 10.2.4 – Added entire section and table
- Section 10.3.1 – Revised table on Instrument Parameters
- Section 10.3.2 – Revised table on Sample Sequence

## Attachment 1

## Sampling Information

| Chemical Name | Maximum Sample Storage | OSHA PEL TWA STEL | NIOSH REL TWA STEL | ACGIH TLV TWA STEL | Sampling Rates (L/min) | Air Volume Minimum Maximum | Reporting Level (µg/sample) |
|---|---|---|---|---|---|---|---|
| Aroclor 1016 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1221 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1232 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1242 | 60 days | 1 $mg/m^3$ | 0.001 $mg/m^3$ | 1 $mg/m^3$ | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1248 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1254 | 60 days | 0.5 $mg/m^3$ | 0.001 $mg/m^3$ | 0.5 $mg/m^3$ | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1260 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1262 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |
| Aroclor 1268 | 60 days | | | | 0.05 – 0.2 | 1 - 50 | 0.1 |

**Definitions of acronyms and abbreviations in Attachment 1:**
PEL = Permissible Exposure Limit
REL = Recommended Exposure Limit
STEL = Short Term Exposure Limit (usually based on 15-minute sampling period)
TLV = Threshold Limit Value
TWA = Time-Weighted Average (usually based on 8-hour workday)
OSHA = Occupational Safety and Health Administration
NIOSH = National Institute for Occupational Safety and Health
ACGIH = American Conference of Governmental Industrial Hygienists
LFC = lowest feasible concentration
mL/min = milliliters per minute
$mg/m^3$ = milligrams per cubic meter
Ceiling = the highest concentration allowable ever

**Attachment 2**

Figure 1. Representative Standard Solution Containing Aroclor 1016 and 1260.

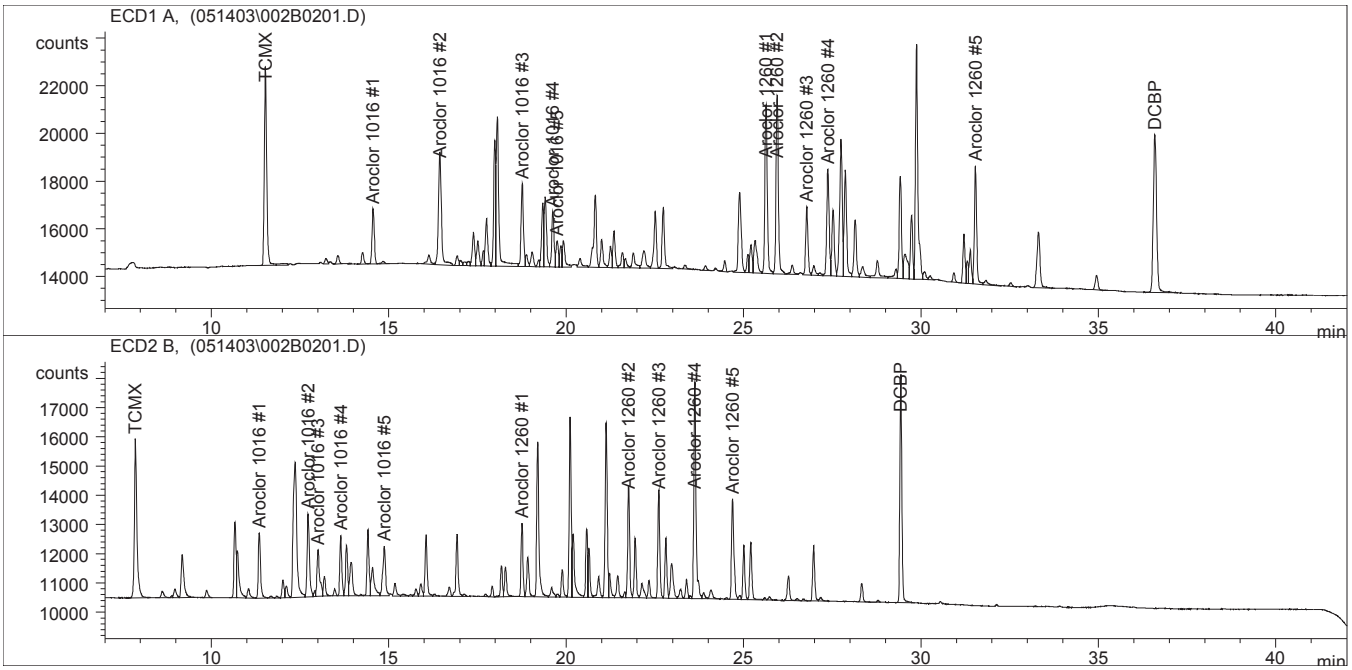
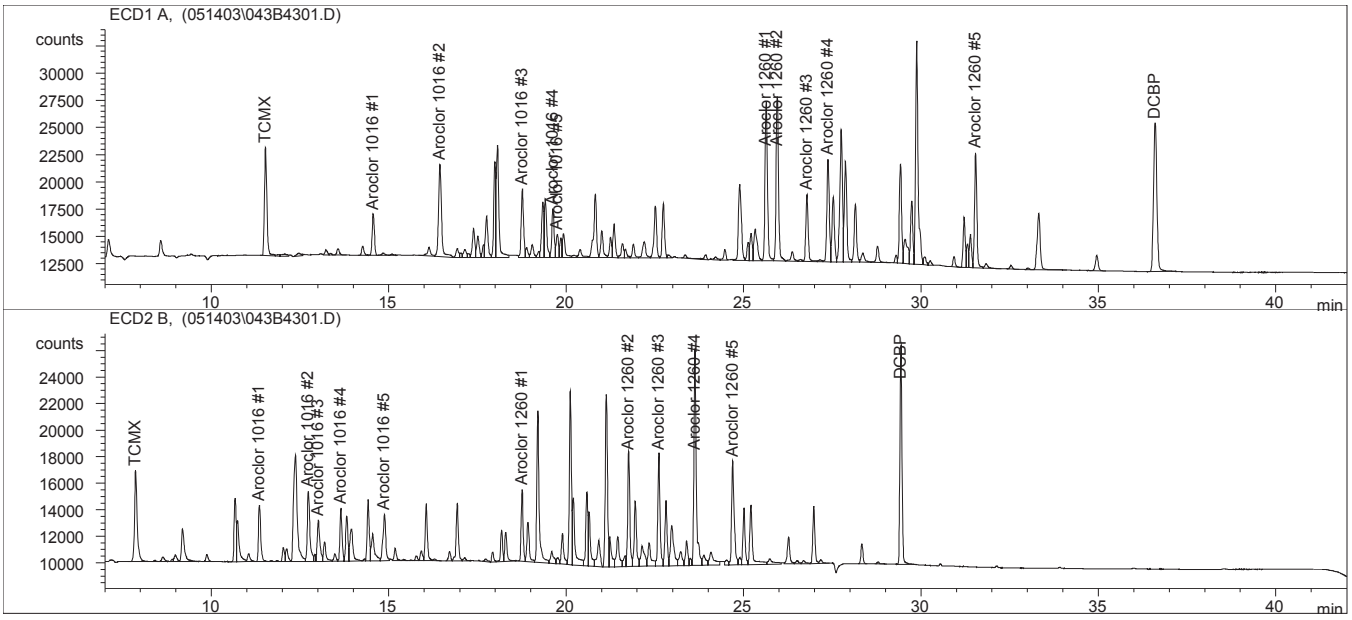



Figure 2. Representative Low Spike Containing Aroclor 1016 and 1260.



Metals Using ICP-AES and/or ICP-MS [NIOSH 7300, Issue 2 Modified, NIOSH 7303, OSHA ID-121 and OSHA ID-125G (Modified)]

# Title: Metals Using ICP-AES and/or ICP-MS [NIOSH 7300, Issue 2 Modified, NIOSH 7303, OSHA ID-121 and OSHA ID-125G (Modified)]

**Approvals (Signature/Date):**

| | |
|---|---|
| 4/6/2015 | 3/20/2015 |
| Stephanie Stimson — Date | Lisa Maycock — Date |
| IH Program/Technical Director | Environmental Health & Safety Coordinator |
| 3/24/2015 | 4/6/2015 |
| Ann Doerr — Date | Stephanie Stimson — Date |
| Quality Assurance Manager | Interim Laboratory Director |

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## 1.0 Scope and Application

This SOP supports metal air samples analyzed by NIOSH 7300 with both MCE and PVC filter media, NIOSH 7303 for MCE filter media, OSHA 121 for MCE filter media and OSHA 125G for both MCE and PVC via ICP and ICP-MS instrumentation under AIHA and NELAC certification as well as lead in air samples under ELLAP certification. Sample volumes and rates may vary by method.

### 1.1 Analytes, Matrix(s), and Reporting Limits

**1.1.1** This SOP is applicable to the determination of the concentration of trace elements in solution using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) and/or Inductively Coupled Plasma-Mass Spectrometry (ICP/MS). Digested samples are matrix matched with the standards. The following metals can be analyzed using this method:

| | | | |
|---|---|---|---|
| Aluminum | Chromium | Nickel | Tin |
| Antimony | Cobalt | **Phosphorus** | Titanium |
| Arsenic | Copper | Potassium | Uranium |
| Barium | **Iron** | **Selenium** | Vanadium |
| Beryllium | Lead | Silicon | Zinc |
| Bismuth | Lithium | Silver | Indium |
| Boron | **Magnesium** | Sodium | Gallium |
| Cadmium | Manganese | Strontium | |
| Calcium | Molybdenum | Thallium | |

**1.1.2** The reporting limit for each metal is listed in **Attachment 3**.

**1.1.3** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

## 2.0 Summary of Method

**2.1** Prior to analysis, samples must be digested in acid so that the metal content of the sample is detectable by the ICP and/or the ICP/MS instruments.

**2.2** Methods NIOSH 7300 and 7303, plus OSHA 121 and 125G describe the multi-elemental determination by ICP-AES using a simultaneous optical system with axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radiofrequency inductively coupled argon plasma. The spectra are dispersed by a grating spectrometer, and photosensitive devices monitor the intensities of the lines. Interference correction is required for trace element determination. We have applied this method to the ICP/MS due to the extreme similarities between the two (2) instruments.

## 3.0 Definitions

**3.1** Accuracy – The degree of agreement of a measured quality of concern.

**3.2** Calibration Standard (CAL) – A solution prepared from the primary dilution standard solution(s) or stock standard solutions. Used to calibrate the instrument.

**3.3** Continuing Calibration Verification Standard (CCV) – A CAL solution which is analyzed after every tenth field sample analysis, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous field samples analyzed. The CCV shall be from the primary source.

*Note: CCV's for IH/NELAC samples are at the Highest Calibration Standard and CCV's for ELLAP samples are at the Mid-point Calibration Standard.

**3.4** Calibration Blank/Continuing Calibration Blank (CB/CCB) – A calibration blank and continuing calibration blank are zero concentration standards. They are in essence the solvent/reagent solution (without analyte) in which instrument calibration standards are prepared. The calibration blank and continuing calibration blank are not subject to all of the handling steps applied to samples.

**3.5** Initial Calibration Standards (ICAL) – A series of CAL solutions used to initially establish instrument calibration and develop calibration curves.

**3.6** Initial Calibration Verification Standard (ICV) – A CAL solution, which is analyzed initially, prior to any field sample analysis, which verifies the previously established calibration curve. The concentration for the ICV should be at the lowest calibration level. The ICV solution must be made from a second source, independent of that of the calibration standards.

***Note: ICV for IH/NELAC/ELLAP samples is at the RL.**

**3.7** Initial Demonstration of Capability (IDOC) – A procedure to establish the ability of the analyst to generate acceptable accuracy and precision.

**3.8** Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS / LCSD) – An aliquot of filter matrix, to which a known quantity of analyte is added in the laboratory. This solution is comprised of analyte(s) that is obtained from a source external to the laboratory and different from the source of calibration standards. The LCS/LCSD are analyzed exactly like a sample.

**3.9** Method Blank (MB) – An aliquot of filter matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples.

**3.10** Precision – The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the closeness of results.

**3.11** Relative Percent Difference (RPD) – The difference between two values divided by the average of the values as expressed as a percent.

**3.12** Reporting Limit Verification (RLV) - An aliquot of filter matrix, to which a known quantity of analyte is added in the laboratory that is at or below the established reporting level and analyzed exactly like a sample. Annual requirement for IHLAP; Batch requirement for ELLAP.

**3.13** Interference Check Standards ICSA/AB– Solutions of selected method analytes of higher concentrations which are used to evaluate the routine for correcting known inter-element spectral interferences. They are also known as spectral interference check (SIC) solutions.

**3.14** Prep Blank (PB) (Digested Reagent Blank) – ELLAP only: An reagent blank that is treated exactly as a sample that is digested and analyzed. No filter matrix is added only digestion reagents. The results must fall below the reporting level, the MDL, or a multiplier of the MDL. PBs may not be used to blank correct ELLAP samples.

## 4.0 Interferences

**4.1** Inductively Coupled Plasma (ICP)

**4.1.1** Spectral interference is the most significant type of interference in ICP analyses. Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. The ICP has the ability to use multi-component spectral fitting (MSF) as a tool to correct for interferences. MSF uses mathematical models to distinguish analyte spectra from interfering spectra. As well as for correcting for interferences, MSF can improve detection limits and precision. MSF uses a multiple linear regression technique to fit the models created to unknown spectra during an analysis. Since only the peak shapes need to remain constant, typically the models are independent of concentration, plasma conditions and matrix effects.

**4.1.2** The ICP also has the ability to correct for spectral interferences using interelement correction (IEC) factors. The software is able to build a mathematical matrix that defines the concentrations observed for all the analytes contained in the active method. The matrix is a model of the spectral interferences expected from the elements analyzed.

**4.1.3** Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer.

**4.1.4** Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, etc.), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift.

Wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample, can control the problem.

**4.1.5** Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within the analytical run and suitable rinse times should be used to reduce them. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. The analyst may establish alternate rinse times.

**4.2** Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) – Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

**4.2.1** Isobaric elemental interferences – Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by a method of measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections would only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

**4.2.2** Abundance sensitivity – Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

**4.2.3** Isobaric polyatomic ion interferences – Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified. These interferences are listed in Table 2 of the EPA Method 200.8 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence, as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common Kr interference that affects the 82nd determination of both arsenic and selenium can be greatly reduced with the use of high purity krypton free argon.

**4.2.4** Physical interferences – Are associated with the physical processes, which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards ideally should have similar analytical behavior to the elements being determined.

**4.2.5** Memory interferences – Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to 10 times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of 10 of the method detection limit should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If memory interference is suspected, the sample should be reanalyzed after a long rinse time.

## 5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, closed-heel, nonabsorbent shoes are a minimum.

### 5.1 Specific Safety Concerns or Requirements

**5.1.1** The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

**5.1.2** Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

**5.1.3** Either nitrile or latex gloves may be used when performing this method.

**5.2 Primary Materials Used**

**5.2.1** The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material (2) | Hazards | Exposure Limit (1) | Signs and symptoms of exposure |
|---|---|---|---|
| Hydrochloric Acid | Corrosive Poison | 5 ppm-Ceiling | Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| Nitric Acid | Oxidizer Corrosive Poison | 2 ppm-TWA 4 ppm-STEL | Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. |
| 1 – Exposure limit refers to the OSHA regulatory exposure limit. | | | |
| 2 – Always add acid to water to prevent violent reactions. | | | |

## 6.0 Equipment and Supplies

### 6.1 Instrumentation

- Computer-controlled Inductively Coupled Plasma - Mass Spectrometer with Autosampler (Perkin Elmer® ELAN 6100 and Thermo X-Series) with radio frequency generator compliant with FCC regulations.
- Two Perkin Elmer® DV 5300 Computer-controlled Inductively Coupled Argon Plasma Emission Spectrophotometers.
- Hot Block digester capable of maintaining a temperature of 95°C.
- Vapor recovery device (i.e., ribbed watch glass, polypropylene ribbed watch cap).
- Analytical balance, capable of measuring to 0.01 g.
- Hood for ventilation with minimum face velocity of 100 cubic feet per minute (CFM).

**6.2 Supplies**

- Hot Block Digestion Cups, Polypropylene, graduated, with snap caps, 50 mL.
- Hot Block Digestion Cup Racks, 18-cup capacity.
- Volumetric flasks: Class A, 100 mL, for Metals use only.
- Adjustable Eppendorf pipette with appropriate tips.
- Filter paper - Hydrochloric/Hydrofluoric acid washed, Ultra pure water rinsed.
- Transfer pipettes, disposable, plastic.
- Specimen Cups, 100 mL, free of trace metals.
- Argon gas supply.
- Nitrogen gas supply (ICP only).
- Sampler: Mixed Cellulose Ester membrane filter (MCE), 0.8-µm pore size, 37-mm diameter, in cassette filter holder.
- Sampler: PVC filters with 5.0 µm pore and diameter.
- Personal sampling pump, 1 to 4 L/min., with flexible connecting tubing.

## 7.0 Reagents and Standards

**7.1** Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of determination.

**7.2** Nanopure water: Deionized water filtered through a Barnstead Nanopure II deionized water system to a resistivity greater than 17.0 megohm-cm. Water from the DI Polisher in the metals lab may be used as long as it meets the criteria listed at the unit.

**7.3** Concentrated Nitric Acid ($HNO_3$): Instra-analyzed, Trace Metal Grade, conc.

**7.4** 1:1 $HNO_3$ Acid. Add 40 mL Nanopure Water to 100 mL Volumetric Flask. Add 50 mL $HNO_3$. Bring up to volume with Nanopure Water.

**7.5** Concentrated Hydrochloric Acid (HCl): Instra-analyzed, Trace Metal Grade, conc.

**7.6** Internal Standard (ICP): 100 ppm Yttrium intermediate standard made from 1000 ppm Yttrium Stock Standard.

**7.7** Internal Standard (ICP/MS): A multi element standard stock solution is used to make a working solution.

**7.8** Stock standard solutions: There should be one source of standards for use in standardizing the instrument, and a second independent source for use as a quality control standard as indicated in the quality control sections of this SOP.

**7.8.1** The standards are prepared in an acid matrix that will match that of the samples as closely as possible.

**7.8.2** Mixed or single element calibration standard solutions: Purchased from reputable manufacturers/suppliers. All certificates of analysis are kept on file. Multiple calibration standard solutions are utilized due to spectral and chemical incompatibilities of some elements.

**7.8.3** The Interference Check Solution (ICS) contains known concentrations of interfering elements (such as Fe, Al, Ca, and Mg at 100 - 500 ppm). This is used to test the interference correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero. If the particular instrument will display over-correction as a negative number this spiking procedure will not be necessary.

## 8.0 Sample Collection, Preservation, Shipment and Storage

**8.1** Sampler: Filter (0.8-µm, 37-mm/47-mm, or equivalent cellulose ester membrane).

**8.2** Sampler: Filter (5.0-µm, 37-mm/47-mm, or equivalent polyvinylchloride filters).

**8.3** Flow rate: 1 to 4 L/minute. (Volume and rate varies by method).

**8.4** Air cassette samples have a holding time of six months (laboratory criteria). The filters should arrive at the laboratory in their plastic cassette cases with the covers closed. The cassettes can be shipped to the laboratory by being placed in a sturdy container (i.e. cardboard box). Icing is not needed.

**8.5** Two to ten (2 to 10) field blanks per set (clients should include a field blank with each set of samples).

## 9.0 Quality Control

### 9.1 QC Procedure Frequencies and Acceptance Limits for AIHA/NELAC

| QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Calibration Curve, 3 point (Minimum) | Each day of analysis | $r \geq 0.995$<br>Blank $\leq$ RL | 1. Evaluate Instrument and Standards.<br>2. Reanalyze curve<br>3. Prepare new standards and reanalyze curve |
| Initial Calibration Verification (ICV) at or below the Reporting Level | Immediately following calibration standards and also at beginning of analysis if re-calibration is not being performed | 50 - 150%; Si: 25 – 175% of predicted response following calibration | 1. Re-inject ICV.<br>2. Evaluate Standards.<br>3. Recalibrate. |

| QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Reporting Limit Verification (RLV) – Digested QC sorbent sample at or below the Reporting Level | Initially, annually and/or if there is a significant change in the background or instrument response. | 50 -150%; Si 25 – 175% of known value | Re-inject; Re-digest additional spike; raise RL and perform RLV at higher RL. |
| Calibration Blank/Continuing Calibration Blank | Beginning (before calibration curve), before or after ICV every 10 samples after CCV, and end of each analysis day | < RL | Recalibrate and reanalyze all samples since last compliant calibration blank. |
| Continuing Calibration Verification (CCV) @ Highest Calibration Point; Primary Source | Every 10 samples and at the end of the analysis | Within 90 – 110% of expected value | Re-analyze previous set of samples on either side of CCV. |
| Interference Check Standard (ICS & ICSAB) ICP only | Beginning, every 8 hours and the end of the analytical run | 80 – 120% recovery of the true value | Recalibrate and reanalyze all samples |
| Internal Standard | Each sample and MBLK, LCS/LCSD | Acceptable recovery is 70 – 130% of true value for ICP, 60 – 140% for ICP-MS | Dilute the sample, re-analyze and flag data with appropriate qualifier |
| Method Blank (MB) | Every sample batch of up to 20 samples | < RL | Report Method Blank result. Flag data with appropriate qualifier and complete NCM |
| Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Second Source | Every sample batch of up to 20 samples | Acceptable recovery is 80 – 120% of true value, and ≤ 20% RPD | 1. Re-inject to confirm.<br>2. Report result. Flag data with appropriate qualifier and complete NCM. |

*All QC must be matrix matched to acid content present in sample digestates.

## 9.2 Sample QC Procedure/Frequencies and Acceptance Limits for AIHA/NELAC

**9.2.1** All quality control information should be readily available for review. The information should be detailed enough so that someone reviewing the data can follow the analytical steps and calculations.

**9.2.2** Sample preparation, analysis, interpretation of results and quality control are done by qualified personnel who are familiar with ICP and ICP-MS theory and practice and have demonstrated the ability to generate acceptable results in metals analysis.

**9.2.3** The AIHA technical manager and/or her/his designee perform the final data review.

**9.2.4** Sample concentrations that exceed the concentration of the highest calibration point need to be diluted to within the calibration range and reanalyzed.

**9.2.5** An internal standard is added to samples in order to help compensate for matrix effects of high solids samples. Preferably, Yttrium should be used, but any analyte not being tested for that does not exhibit a spectral interference to the samples may be used. The recovery of the internal standard needs to be between 70 - 130% for ICP and 60 – 140% for ICP-MS. If recovery is not within this range, dilute the sample and re-analyze. If dilution does not bring the recovery within acceptance limits, flag with the appropriate data qualifier.

**9.2.6** It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

**9.2.7** Analyze a minimum of one method blank per 20 samples to determine if any contamination is occurring. The method blank must be carried through all sample prep procedures and contain the same acid concentration in the final solution as the sample solution used for analysis. The concentration in the method blank must be less than the reporting level. If the method blank is above the report level, then the samples should be blank corrected and this noted on the client report.

**9.2.8** Analyze two LCSs for every 20 samples. The LCS should be a solid matrix material (adding a known amount of liquid analyte to a MCE or PVC filter and then letting the analyte dry is an acceptable practice). Including the media, the LCS should be carried through all sample prep procedures and contain the same acid concentration in the final solution as the sample solution used for analysis. The LCS should have a recovery between 80 - 120%. The two LCSs should have a RPD of ≤ 20%. Should an LCS fail, samples should be flagged with the appropriate data qualifier and complete an NCM and submitted to QA along with the raw data (following the final review process).

**9.2.9** After calibration, every ten samples and at the end of a run a CCV and CCB are analyzed. The CCB concentration should be less than the report limit. The CCV should have a percent recovery between 90 - 110%.

**9.2.10** An Interference Check Standard should be analyzed at the beginning and end of each analysis and at least once every eight (8) hours during the analysis. The result must be within 20% of the true value. If the interference check sample is outside the acceptance limits, then the instrument should be recalibrated for that analyte.

**9.2.11** At the end of the analytical run, a check standard (high-level Calibration Check) must be analyzed that is at or above the highest reportable concentration for each analyte in any given sample. The result of the check standard must be within 10% of the true value for each analyte. If the standard is outside the acceptance limits for any analyte, then that analyte must be reanalyzed.

**9.2.12** A reporting limit verification spike: The reporting level must be established and verified be performing a reporting limit verification, which is done by fortifying a media spike at a concentration at or below the expected reporting level, which is typically equal to the lowest standard concentration. Perform all calculations defined in the method and report the concentration values in the appropriate units. The reporting level should be verified initially, annually and/or if there is a significant change in the background or instrument response.

The percent recovery for the reporting level verification spike should be within 50 – 150%; Si 25 – 175% of the expected value. . If the percent recovery is outside this range, then the spike should be confirmed by re-injecting, re-digesting and/or re-digesting analyzing at a higher level resulting in a higher reporting level.

9.2.13 Inter-element correction tables must be established for each individual analytical line and updated annually. The IEC's are not required as long as you can justify that they are not needed by running interference checks (ICSA & ICSAB) or MSF must be developed for analytical lines requiring corrections. See Attachment 6 for guidelines on the procedure. Reference the instrument method for information regarding which correction model is being used, either IEC or MSF, for each wavelength. All measurements must be within the instrument linear dynamic range where the correction equations are valid. See Attachment 7 for the current inter-element correction tables.

## 9.3 QC Procedure Frequencies and Acceptance Limits for ELLAP (Lead Only)

| QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Wipe sampling of laboratory surfaces associated with sample preparation and analysis | Quarterly wipe samples performed by QA department.<br><br>Quarterly wipe samples shall be logged in quarterly for lead wipe analysis via NIOSH 9100 or NIOSH 9102 and any corrective actions documented in the LIMS NCM system. | < lowest regulatory reporting level and/or laboratory reporting level. | Re-clean and re-sample to verify cleanliness before any analytical work is performed in the affected area. |
| Internal Standard | Each sample and MBLK, LCS/LCSD, etc. | Acceptable recovery is 70 – 130% of true value for ICP | Dilute the sample and flag data with appropriate qualifier |
| Calibration Curve, 3 point (Minimum) | Each day of analysis | $r \geq 0.995$<br>Blank ≤ RL | 1. Re-analyze curve<br>2. Prepare new standards and reanalyze curve |
| Initial Calibration Verification (ICV) – Second Source spiked at or below the reporting level | Once per run after calibration | Within ± 10% of the known value | 1. Re-inject ICV.<br>2. Evaluate Standards.<br>3. Recalibrate |
| Reporting Limit Verification (RLV) – Digested QC filter sample spiked at or below the Reporting Level | Once per day | Within ± 20% of known value | 1. Re-inject to confirm.<br>2. Report result. Flag data with appropriate qualifier and complete NCM. |

| | | | |
|---|---|---|---|
| Initial Calibration Blank (ICB) | Once per run at the beginning of the run (before the Calibration Curve) | Absolute value not more than 10% of the regulatory limit or minimum level of concern | Recalibrate and reanalyze all samples since last compliant calibration blank. |
| Continuing Calibration Blank (CCB) | After each ICS, ICV and CCV | Absolute value not more than 10% of the regulatory limit or minimum level of concern | Recalibrate and reanalyze all samples since last compliant calibration blank. |
| Continuing Calibration Verification (CCV) @ Mid-Level Calibration Point; Primary Source | Every 10 samples and at the end of the run | Within ± 10% of the known value | Re-analyze previous set of samples on either side of CCV. |
| Interference Check Sample (ICSA/ICSAB) for ICP analysis only | Beginning and end of each run or twice every 8 hours | Within ± 20% of the known value | Recalibrate and reanalyze all samples |
| Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – Second Source | Every sample batch of up to 20 samples | Acceptable recovery is 80 – 120% of true value, and ≤ 20% RPD | 1. Re-inject to confirm.<br>2. Report result. Flag data with appropriate qualifier and complete NCM. |
| Duplicate Sample | One per 20 samples or batch (5%), if available. | ≤ 25% RPD for the duplicate pair | 1. Re-inject for confirmation.<br>2. Report result. Flag data with appropriate qualifier and complete NCM.. |
| Method Blank (Matrix Blank)<br><br>And<br><br>Prep Blank (PB) (Reagents Only – No Filter - Digested Reagent Blank) | One per 20 samples or batch (5%) | Absolute value not more than 10% of the regulatory limit or the minimum level of concern | 1. Do not correct for results above the reporting level. Report and qualify as not subtracted from QC or sample results<br>2. Re-analyze<br>3. Determine cause of problem<br>4. Evaluate associated data<br>-If analyte ND in sample – flag with appropriate qualifier and complete NCM.<br>-If conc. in sample >10X conc. in blank flag with appropriate qualifier and complete NCM |

*All QC must be matrix matched to acid content present in sample digestates.

## 9.4 Sample QC Procedure/Frequencies and Acceptance Limits for ELLAP (LEAD Only)

**9.4.1** All quality control information should be readily available for review. The information should be detailed enough so that someone reviewing the data can follow the analytical steps and calculations.

**9.4.2** Sample preparation, analysis, interpretation of results and quality control are done by qualified personnel who are familiar with ICP and ICP-MS theory and practice and have demonstrated the ability to generate acceptable results in metals analysis.

**9.4.3** The ELLAP technical manager and/or her/his designee perform the final data review.

**9.4.4** Quarterly Wipe Sampling – Sampling of sample preparation and analysis surfaces shall be performed by the QA department. Quarterly wipe samples shall be logged in quarterly for lead wipe analysis via NIOSH 9100 or NIOSH 9102. Results not meeting the acceptance criteria of < lowest regulatory reporting level and/or laboratory reporting level shall be re-cleaned and re-sampled to verify cleanliness before any analytical work is performed in the affected area. Any corrective actions shall be documented in the LIMS NCM system.

**9.4.5** Sample concentrations that exceed the concentration of the highest calibration point need to be diluted to within the calibration range and reanalyzed.

**9.4.6** An internal standard is added to samples in order to help compensate for matrix effects of high solids samples. Preferably, Yttrium should be used, but any analyte not being tested for that does not exhibit a spectral interference to the samples may be used. The recovery of the internal standard needs to be between 70 - 130% for ICP. If recovery is not within this range, dilute the sample and re-analyze. If dilution does not bring the recovery within acceptance limits, flag with the appropriate data qualifier.

**9.4.7** It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

**9.4.8** Analyze a minimum of one method blank per 20 samples to determine if any contamination is occurring. The method blank must be carried through all sample prep procedures and contain the same acid concentration in the final solution as the sample solution used for analysis. The concentration in the method blank must be less than the reporting level. If the method blank is above the report level, then the samples should be not be blank corrected and this noted by using qualifiers or narrating on the client report. The absolute value should be ≤ 10% of the regulatory limit or the minimum level of concern.

**Regulatory limits in air:**
1.5 µg/m$^3$ – EPA National Ambient Air Quality Standard
30 µg/m$^3$ – OSHA Action Level (8 hr TWA)
50 µg/m$^3$ – OSHA Permissible Exposure Limit (General Industry)
50 µg/m$^3$ – OSHA Permissible Exposure Limit (Construction)

**9.4.9** Analyze two LCSs for every 20 samples. The LCS should be a solid matrix material (adding a known amount of liquid analyte to a MCE or PVC filter and then letting the analyte dry is an acceptable practice). Including the media, the LCS should be carried through all sample prep procedures and contain the same acid concentration in the final solution as the sample solution used for analysis. The LCS should have a recovery between 80 - 120%. The two LCSs should have a RPD of ≤ 20%. Should an LCS fail, samples should be flagged with the

appropriate data qualifier an NCM completed and submitted to QA along with the raw data (following the final review process).

**9.4.10** After calibration, every ten samples and at the end of a run a CCV and CCB are analyzed. The CCB concentration should be less than the report limit. The CCV should have a percent recovery between 90 - 110%.

**9.4.11** An Interference Check Standard should be analyzed at the beginning and end of each analysis and at least once every eight (8) hours during the analysis. The result must be within 20% of the true value. If the interference check sample is outside the acceptance limits, then the instrument should be recalibrated for that analyte.

**9.4.12** An Initial Calibration Verification, ICV, at the reporting limit from a second source shall be analyzed after instrumental calibration. The result must be within 90 -110% of the true value. If the standard is outside the acceptance limits, then the instrument must be recalibrated for that analyte, and the affected samples re-analyzed.

**9.4.13** Daily Reporting Limit Verification (RLV) (CRI/MRL): A reporting limit verification spike per batch of 20 samples: The reporting level must be established and verified be performing a reporting limit verification, which is done by fortifying a media spike at a concentration at or below the expected reporting level, which is typically equal to the lowest standard concentration. Perform all calculations defined in the method and report the concentration values in the appropriate units. The reporting level should be verified daily. The percent recovery for the reporting level verification spike should be within 80 - 120% of the expected value. If the percent recovery is outside this range, then the spike should be confirmed by re-injecting, re-digesting and/or re-digesting analyzing at a higher level resulting in a higher reporting level.

**9.4.14** At the end of the analytical run, a CCV, continuing calibration check standard must be analyzed that is around the mid-level calibration standard concentration for each analyte in any given sample. The result of the check standard must be within 10% of the true value for each analyte. If the standard is outside the acceptance limits for any analyte, then that analyte must be reanalyzed.

**9.4.15** The ICP instrument displays negative concentration, so the fortified interference check is not necessary. TestAmerica - Phoenix analyzes an ICSA, an interference check sample without the analytes spiked. Generally, the air samples analyzed do not have detectable analyte concentrations. Therefore, the ICS with no analytes spiked is representative of the routine samples received. The criteria used to evaluate the ICSA is plus or minus the reporting limit.

## 9.5 Method Blank, Prep Blank and Calibration Blank

**9.5.1** A method blank must be analyzed with each batch of samples. The method blank is used to identify possible contamination resulting from varying amounts of the acids used in sample processing and media. A prep blank is also required to be analyzed with each batch or ELLAP samples.

**9.5.2** The method blank must contain all the reagents in the same volumes as used in the processing of the samples including the MCE filter. The method blank must be carried

through the complete digestion procedure and contain the same acid concentration as that used in the sample solution. This method is also applicable to PVC filters and must be processed in the same manner as the MCE filters.

**9.5.3** Prep Blank (PB) (Digested Reagent Blank) – ELLAP only: An reagent blank that is treated exactly as a sample that is digested and analyzed. No filter matrix is added only digestion reagents. The results must fall below the reporting level, the MDL, or a multiplier of the MDL. PBs may not be used to blank correct ELLAP samples.

**9.5.4** Most analytes found in the method blank and prep blank for ELLAP samples will generally be below the reporting levels. However, if there is a value above the reporting level, then subtract the method blank from the QC samples and the client's samples for non-ELLAP samples. ELLAP samples are not to be blank corrected by the method blank or prep blank. If the method blank yields a result above the reporting level, but the client's samples do not show a background or the QC samples do not have an unusually high recovery, then the contamination is most likely isolated to that blank and further investigation should occur and an appropriate decision be made after further investigation. An appropriate decision may include, but not be limited to, re-analyzing the blank, extracting one or two additional blanks, analyzing an additional aliquot of the solvent used for extraction to check for contamination, ignoring the blank if it is obviously the only sample contaminated, etc.

**9.5.5** A calibration blank is used in establishing the analytical curve and to determine background levels of the solvents/reagents. Beginning, every 10 samples, and end of each analysis day, before or after ICV.

**9.5.6** The calibration blank/continuing calibration blank is prepared by acidifying reagent water to the same acid concentrations found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all continuing calibration blank determinations.

**9.5.7** The Interference Check Standards (ICS & ICSAB) contain known concentrations of interfering elements (such as Fe, Al, Ca and Mg at 100 – 500 ppm). This is used to test the interference correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, over-correction could go undetected since a negative value could be reported as zero.

## 9.6 Internal Quality Control Procedures

**9.6.1** As part of the quality assurance program, the laboratory shall adhere to all stated QA/QC requirements in the method(s) used. Any deviations from the method shall be documented. Deviations that result in non-conforming work shall be evaluated. The following QC checks will be performed per batch of samples:

**9.6.2** Accuracy and bias: Accuracy studies are performed to determine how close a measurement comes to actual or theoretical value. Accuracy can be expressed as percent recovery and evaluated by analysis of laboratory control samples (LCS). Bias is a systematic error manifested as a consistent positive or negative deviation from the true value. The bias can be found in NIOSH 7300 and 7303 as well as OSHA 121 and 125G.

**9.6.3** Precision: Precision is evaluated by the reproducibility of analyses and it is commonly expressed as a relative percent difference. It can be evaluated by the analysis of laboratory control sample and laboratory control sample duplicate (LCS/LCSD). Precision should be ≤20% for LCS/LCSD pair, or ≤10% for replicate injections of calibration standards.

**9.6.4** Blanks: Blank sampling media and analytical reagents will be analyzed, when applicable, with each batch of samples. At least one field blank should be used for each day of field sampling, shipped and analyzed with each group of samples. The field blank is treated identically to the samples except that no air is drawn through the cartridge.

**9.6.5** Reporting Field Blanks: The final report shall state the measured quantitative result of the field blanks submitted to the laboratory. Also, the laboratory should disclose whether or not the sample results have been corrected for contamination based on the field blank or other analytical blank. **Note: ELLAP samples are not to be blank corrected for method or field blanks.**

## 10.0 Procedure

### 10.1 Sample Preparation

**10.1.1** Locate samples and create a batch in the LIMS system. Confirm the analysis and metals needed via the backlog and/or chain of custody. If unclear, contact department manager, project manager and or technical program manager for guidance.

**10.1.2** Label sample digestion cups with accession numbers and Batch I.D. Similarly, label final sample containers such as specimen cups, if necessary.

**10.1.3** Place filter in appropriate digestion cup.

**10.1.4** Select a loose filter of the same filter type, MCE or PVC, and wet it with reagent water. Wipe each sample filter cassette to remove all visible material. This additional filter shall be added to the digestion tube of the wiped cassette. For QC samples 2 filters will be utilized to taken into account background levels.

**10.1.5** Pipette Spiking Solutions for LCS/LCSD and/or IDOCs. The Laboratory Control Samples (LCS-LCSD) are carried through the entire procedure, from preparation to analysis. One set should be prepared per type of media with associated samples per batch of 20 samples. MCE for samples on MCE filters, PVC for samples on PVC filters.

**10.1.6** The spike level shall be at a concentration level within the calibration curve of the applicable analysis. Approximately mid calibration level.

### 10.2 For ICP Analysis:

**10.2.1** Add 3.0 mL of concentrated $HNO_3$ and 3.0 mL of concentrated HCl to the sample. The sample in cup is then placed on the heat block, or other heating source at no more than 95℃ until the filter is completely dissolved. (TAKE CAUTION NOT TO BOIL THE SAMPLE).

**10.2.2** Remove samples from heat source when filter is dissolved. Allow samples to cool.

**10.2.3** Note: PVC filters will not dissolve. Digest samples for 30 minutes to ensure digestion is complete.

**10.3 For ICP/MS Analysis:**

**10.3.1** Add 6 mL of $HNO_3$ to the sample. The sample in the cup is then placed on the heat block, or other heating source, at no more than 95℃ until the filter is completely dissolved. (TAKE CAUTION NOT TO BOIL THE SAMPLE).

**10.3.2** Remove samples from the heat source when the filter is dissolved. Allow samples to cool.

**10.3.3** Add 0.25 mL of appropriate internal standard and pour up to a final volume of 25 mL with DI water.

**10.3.4** Cap the samples and place in the Metals Lab with a copy of the Digest Log.

**10.3.5** Note: PVC filters will not dissolve. Digest samples for 30 minutes to ensure digestion is complete.

**10.4 Calibration**

**10.4.1** As per the AIHA Policies manual, a minimum of 3 calibration standards need to be analyzed to establish a new calibration curve. This laboratory typically uses 5 calibration standards. A calibration blank must also be analyzed with each analysis.

**10.4.2** Add the appropriate amount of standard into each digestion tube labeled as a standard. Use the tables below. Calibration levels/ranges may be modified as needed as long as all QA/QC criteria are met and the MDLs support.

### 10.4.3 Calibration Standards for ICP-AES:

| Analyte | Cal. Blank | Std 1 (mg/L) | Std 2 (mg/L) | Std 3 (mg/L) | Std 4 (mg/L) | Std 5 (mg/L) | Std 6 (mg/L) |
|---|---|---|---|---|---|---|---|
| Al | | | 0.05 | 0.2 | 0.5 | 5 | 20 |
| Sb | | | | 0.02 | 0.05 | 0.5 | 2 |
| As | | | | 0.02 | 0.05 | 0.5 | 2 |
| Ba | | 0.001 | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Be | | 0.001 | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Cd | | 0.001 | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Ca | | 0.1 | 0.6 | 2.4 | 6.05 | 60.5 | 242 |
| Cr | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Co | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Cu | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Fe | | | 0.05 | 0.2 | 0.5 | 5 | 20 |
| Pb | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Li | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Mg | | | 0.1 | 0.42 | 1.05 | 10.5 | 42 |
| Mn | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Mo | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Ni | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| K | | | | 0.4 | 1 | 10 | 40 |
| Se | | | | 0.02 | 0.05 | 0.5 | 2 |
| Si | | | 0.025 | 0.1 | 0.25 | 2.5 | 10 |
| Ag | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Na | | 0.12 | 0.6 | 2.4 | 6 | 60 | 240 |
| Sr | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Tl | | | | | 0.05 | 0.5 | 2 |
| Sn | | | | | 0.05 | 0.5 | 2 |
| Ti | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| V | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| Zn | | | | 0.02 | 0.05 | 0.5 | 2 |
| Bi | | | | 0.02 | 0.05 | 0.5 | 2 |
| In | | | | | 0.025 | 0.25 | 1 |
| B | | | | 0.02 | 0.05 | 0.5 | 2 |
| P | | | | | 0.05 | 0.5 | 2 |
| Ga | | | | | 0.025 | 0.25 | 1 |
| V | | | 0.005 | 0.02 | 0.05 | 0.5 | 2 |
| In | | | | | 0.025 | 0.25 | 1 |

### 10.4.4 Calibration Standards for ICP- MS:

| Analyte | Cal. Blank | Std 1 (mg/L) | Std 2 (mg/L) | Std 3 (mg/L) | Std 4(mg/L) | Std 5(mg/L) | Std 6 (mg/L) | Std 7 (mg/L) | ICV/CCV (mg/L) |
|---|---|---|---|---|---|---|---|---|---|
| Antimony | Blank | - | - | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Arsenic | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Barium | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Cadmium | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Chromium | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Cobalt | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Copper | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Lead | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Manganese | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Molybdenum | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Nickel | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Selenium | Blank | - | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Silver | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Thallium | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Uranium | Blank | 0.001 | 0.002 | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Vanadium | Blank | - | - | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |
| Zinc | Blank | - | - | 0.003 | 0.01 | 0.05 | 0.1 | 0.2 | 0.1 |

Standards are made with the CCB solution: 2% Nitric, 1% HCl via serial dilution from 0.2 mg/L primary or secondary standard

The concentrations and number of standards may vary as long as the calibration contains a minimum of three (3) standards.

### 10.4.5 ISOTOPES MONITORED and EQUATIONS USED

| Analyte | Symbol | Isotopes Monitored | Correction Equations |
|---|---|---|---|
| Antimony | Sb | 121 | Sb 123 = Sb 123 – 0.127189 * Te 125 |
| Arsenic | As | 75 | As 75 = As 75 – 3.127 * [ArCl 77 – (0.815*Se 82)] |
| Barium | Ba | 135,**137** | |
| Cadmium | Cd | 111 | Cd 111 = Cd 111 – 1.073 * [MoO 108 –(0.712*Pd 106)] Cd |
| Chromium | Cr | 52 | |
| Cobalt | Co | 59 | |
| Copper | Cu | **63**,65 | |
| Lead | Pb | 206,207,**208** | Pb 208 = Pb 208 + 1* Pb 206 + 1* Pb 207 |
| Manganese | Mn | 55 | |
| Molybdenum | Mo | 98 | Mo 98 = Mo 98 – 0.110588 * Ru 101 |
| Nickel | Ni | 60 | |
| Selenium | Se | 77, 78, **82** | Se 82 = Se 82 – 1.008696 * Kr 83 |
| Silver | Ag | 109 | |
| Thallium | Tl | 205 | |
| Uranium | U | 238 | |
| Vanadium | V | **51** | – 3.127* Cr 53 + 0.353* Cr 52 |
| Zinc | Zn | **66**,67,68 | |
| Internal Standards | | | |
| Yttrium | Y | 89 | |
| Indium | In | 115 | -0.014032 * Sn118 |
| Terbium | Tb | 159 | |
| Germanium | Ge | 74 | -0.116645 * Se77 |
| **Alternate Internal Standards** | | | |
| Lithium | Li | 6 | |
| Scandium | Sc | 45 | |
| Rhodium | Rh | 103 | |
| Bismuth | Bi | 209 | |
| **(Information Only)** | | | |
| Aluminum | Al | 27 | |
| Calcium | Ca | 44 | |
| Krypton | Kr | 83 | |
| Magnesium | Mg | 24 | |
| Sodium | Na | 23 | |
| Potassium | K | 39 | |
| Tellurium | Te | 125 | |
| Tin | Sn | 118, 120 | |
| Ruthenium | Ru | 99, 101 | |
| Iron | Fe | 54 | Fe 54 = Fe 54 – 0.028226 * Cr 52 |

Note: Alternate isotopes are monitored but are not reported.

## 10.5 Sample Analysis

### 10.5.1 Instrument Operation

**I.** Turn instrument on according to the manufacturers' instructions. Argon needs to be at 80 –100 psi and is always left on. Nitrogen needs to be at 60 – 80 psi and is always left on (ICP only).

**II.** After igniting plasma, wait at least 30 minutes for plasma to stabilize before beginning analysis.

**III.** For the **ICP** a mercury alignment and manganese peak (using 1ppm Mn solution) should always be performed daily or at a minimum after the tubing and/or torch has been replaced. Greatest intensity of manganese, along with x-y alignment offset is recorded in the ICP Maintenance Log. For the **ICPMS** a Daily Performance or Tuning should be performed, use the criteria listed in the instrument book as a guideline only. When complete the printout will stay with the data.

**IV.** After selecting the appropriate method from the software, the sample information file or Dataset is created based on date: yymmdd-#, where # = run number for the day (+1 for each new file for that day).

**V.** Pour and place all calibration standards and samples in their proper places. (Note: Calibration standard positions and concentrations may change from time to time, but are recorded within the WinLab or Sciex software under 'Method Editor). A copy of this is printed and placed in the analytical data file. It is also on the Data Review Checklist. The required standards needed for calibration are a calibration blank and calibration standards 1, 2 and 3. One standard must be at or below the reporting limit.

**VI.** To calibrate go to the 'Automated Analysis Control' page. Choose 'Calibrate' to simply calibrate, or 'Analyze All' to calibrate and continue running all samples.

**VII.** Note that the analysis of all calibration check standards and the Interference Check Standard are also entered into the sample information file. Once the instrument is calibrated, analysis can begin. A minimum 60-second wash is mandatory between samples. There is a 30-second delay between the times the sample hits the plasma to when analysis begins to allow for plasma equilibrium with the sample matrix.

### 10.5.2 Sequence/Batch Sample Order:

| Quantitation & Confirmation |
|---|
| 1. Calibration Blank |
| 2. Calibration Standard 1 |
| 3. Calibration Standard 2 |
| 4. Calibration Standard 3 |
| 5. Calibration Standard 4 |
| 6. Calibration Standard 5 |

| Quantitation & Confirmation |
|---|
| 7. Calibration Standard 6 |
| 8. ICV secondary source at mid-point concentration for environmental samples and ICVL at or below Reporting Level for ELLAP and AIHA |
| 9. ICB |
| 10. ICSA |
| 11. ICSAB |
| 12. CCB |
| 13. CCV primary source |
| 14. CCB |
| 15. Method Blank |
| 16. Prep Blank (PB) (Digested Reagent Blank) – ELLAP Only |
| 17. MRL or RLV (if required) (CRI) |
| 18. LCS/LCSD (Second Source) |
| 19. MS/MSD (if applicable) |
| 20 – 29 10 samples |

**10.6 ICP Maintenance**

**10.6.1** As needed, the Nebulizer, torch, injector and spray chamber should be inspected, cleaned and/or replaced.

**10.6.2** Peristaltic pump tubing (3 pieces) needs to be changed when the analyst notices excessive wear, based on number of hours of usage and quality of samples run. The air and water filters are cleaned/changed when excessive build up on the filter is noticed.

**10.7 ICPMS Maintenance**

**10.7.1** The pump tubing is changed daily.

**10.7.2** Clean the cone/skimmer a minimum of once a week, or more often if needed. Clean the front of the skimmer daily with 4% nitric and the plasma turned off. Zn and Mo tend to plate out. When Zn and Mo begin to fail, the cone and skimmer should be changed.

**10.7.3** Clean nebulizer as needed using the correct syringe. Do not back flush the nebulizer as this will permanently damage the nebulizer rendering it un-repairable.

**10.7.4** Clean the lens as needed, when counts between the autolens and daily, i.e. Mg (~70, 000) become much different or approach 120, 000 counts and the DAC values on the autolens are higher than 10.

**10.7.5** Perform X-Y alignment calibration every time lens is cleaned and the skimmer and cone are cleaned/replaced. Or after any maintenance procedures in the torch chamber.

**10.7.6** A dual detector is performed after each time the system is cleaned prior to running a tune wizard.

**10.7.7** The nebulizer gas flow should be monitored daily and adjusted to maintain and lowered when CeO/Ce+ exceed 3% and/or the Ba++/Ba+ exceed 3.55% and the system is clean. The flow should be raised when sensitivity is poor and as the system becomes dirty. Once the flow can no longer be raised, the system should be taken down and thoroughly cleaned.

**10.7.8** For additional maintenance and trouble shooting information see Chapter 3: Tuning and Optimization in the ELAN 6100 software instrument manual.

## 11.0 Calculations / Data Reduction

**11.1** **Reporting Results**: Samples analyzed by NIOSH Method 7300, 7303 and OSHA 121 and 125G are reported in Total µg, or mg/m$^3$, to three significant figures. The quality control data is reported to a minimum of four significant figures. When a field blank is provided for a project, report the field blank as a sample result. The Method Blank is subtracted from all samples and QC except for ELLAP samples unless otherwise indicated.

**11.2** **Paperless Data Generation and Management:** If properly equipped, analysts have the option to use a digital document driver instead of a physical document printer. Data will be printed to a ".pdf" format to a properly labeled folder on a corporate server. As it is exported from the instrument software, the file name will consist of a unique instrument name, date, and time generated automatically by the digital driver. Multiple documents can then be merged into a fewer documents to facilitate attachment to the relevant digital data in LIMS software. After attachment, data will remain available on both the corporate server and LIMS software in accordance with the same requirements as printed data.

**11.3** **AIHA/NELAC**: **(Method Blank Corrected)**

**11.3.1** The analyst must input the sample result into LIMS in the units of mg/L (µg/mL). The LIMS then can perform the following calculations to yield µg and/or mg/m$^3$.

µg, Total = ((RV – MB) * 0.025 L * DF) * (1000 µg/1 mg)

Where:
RV = Raw Value in mg/L
MB = Method Blank Raw Value in mg/L
DF = Dilution Factor

Or

mg/m$^3$ = µg, Total/V

Where:

| | | |
|---|---|---|
| µg, Total | = | mass of analyte per sample volume |
| V | = | air sample volume in Liters |

Or

$$ppm = \frac{Ma * (24.45)}{V * MW}$$

Where:

Ma = µg, Total result
24.45 = ideal gas constant
V = air sample volume in liters
MW = molecular weight of a metal

**11.4 ELLAP: (Not Blank Corrected)**

**11.4.1** The analyst must input the sample result into LIMS in the units of mg/L (µg/mL). The LIMS then can perform the following calculations to yield µg and/or $mg/m^3$.

µg, Total = (RV * 0.025 L * DF) * (1000 µg/1 mg)

Where:

RV = Raw Value in mg/L
DF = Dilution Factor

Or

$mg/m^3$ = µg, Total/V

Where:

| | | |
|---|---|---|
| µg, Total | = | mass of analyte per sample volume |
| V | = | air sample volume in Liters |

Or

$$ppm = \frac{Ma * (24.45)}{V * MW}$$

Where:

Ma = µg, Total result
24.45 = ideal gas constant
V = air sample volume in liters
MW = molecular weight of lead

**11.5 Accuracy**

**ICV / CCV, LCS % Recovery** $= \frac{\text{observed concentration}}{\text{known concentration}} \times 100$

## 11.6 Precision (RPD)

**11.6.1** The relative percent difference between LCS/LCSD spiked duplicate determinations is calculated as follows:

$$\textbf{Relative Percent Difference (RPD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

## 12.0 Method Performance

Historical (in-house) QC limits must be examined for reasonableness. TestAmerica does not want to legitimize poor recoveries that are due to the incorrect choice of methods or spiking levels. Historical limits should also be compared with the objectives of the specific analytical method. For example, recovery limits (for LCS, MS, MSD, etc.) that include allowances for a relatively high positive bias (e.g. 70 – 170%) may be appropriate for determining that an analyte is not present in a sample. However, they would be less appropriate for the analyses of samples near, but below, a regulatory limit because of the potential high bias.

If available, the performance data that is listed in the determinative method should be compared to the QC limits generated from historical data. The SOPs for the determinative methods should have an attached appendix demonstrating those comparisons if available, or TestAmerica's single laboratory performance data will be included as an attachment. Refer to Attachments 1 through 3.

## 12.1 Method Detection Limit Study (MDL)

**12.1.1** The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

**12.1.2** MDL is also sometimes referred to as Limit of Detection (LOD). (NELAC Requirement)

**I.** "The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2 – 3X the LOD for single analyte tests and 1 – 4X the LOD for multiple analyte tests. The verification must be performed on every instrument that is to be used for analysis of samples and reporting of data." (2003 NELAC Standards, C.3.1.b)

**12.1.3** An important characteristic of expression of sensitivity is the difference in the MDL (LOD) and the Quantitation Limit or Reporting Limit (sometimes referred to as the Limit of Quantitation (LOQ)). (NELAC Requirement)

**I.** "The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1 – 2 times the claimed

LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objective for accuracy. This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ." (2003 NELAC Standards, C.3.2.c)

**12.1.4** Additional information can be found in the QA Manual.

**12.1.5** ELLAP requires an annual full MDL Study to be performed. For NELAC/AIHA an annual MDLV/RLV is satisfactory.

**12.2 <u>Reporting Level Verification</u> (AIHA and ELLAP)**

**12.2.1** The reporting level must be established. This is accomplished by fortifying a media spike at a concentration at, or below, the expected reporting level, which is typically equal to the lowest standard concentration. Perform all calculations defined in the method and report the concentration values in the appropriate units.

**12.2.2** **AIHA:** The reporting level media spike should be verified initially, annually, or if there is a significant change in the background or instrument response for AIHA.

**12.2.3** Percent recovery for the reporting level verification spike should be within 50 – 150% of the expected value. If the percent recovery is outside this range, then an additional spike should be prepared at a higher level, which will result in a higher reporting level.

**12.2.4** **ELLAP:** Each day that a matrix is analyzed, a matrix spiked sample (RLV/CRI), spiked at a concentration equal to or below the stated reporting limit with a liquid spike, shall be digested an analyzed to demonstrate acceptable recovery of lead.

**12.2.5** Percent recovery for the reporting level verification spike should be within 80 – 120% of the expected value. If the percent recovery is outside this range, then an additional spike should be prepared at a higher level, which will result in a higher reporting level.

**12.3 <u>Training Requirements</u>**

**12.3.1** Refer to the QA Manual or to SOP PE-QAD-008 – Personnel Certification and Training. At a minimum before an analyst can perform the method independently, they must have:

**a.** Read the analytical method(s);
**b.** Read the applicable SOP(s); and
**c.** Acceptably performed and documented the data for four LCS' (normally two LCS/LCSD (BS/BSD) sets).

**12.3.2** TestAmerica's participation in the American Industrial Hygiene Association and ELLAP program requires the additional performance as listed in **section 12.4** – "Demonstration of Capabilities". **(AIHA/ELLAP Methods only)**

**I.** Additionally, training must be documented to include a description of the training content/activities and duration of the program provided to the analyst. The training course

must last a minimum of 20 business days per the AIHA/ELLAP requirements. No independent work may be done prior to the completion of the training program.

**12.4 Demonstration of Capabilities**

**12.4.1** Refer to the QA Manual for general procedures and any specific concentrations that must be used.

**12.4.2** A Demonstration of Capability form shall be completed every six months per AIHA - IHLAP.

**I.** **Initial Demonstration of Capability (IDOC)**: Prior to reporting any data, each analyst must have on file with the QA office information demonstrating proficiency with the analysis technique. Both precision and accuracy are measured for the target analytes. Make four replicate analyses of a daily working standard having a concentration between 1 -4 times the RL for each analyte (generally at the LCS level). Calculate the average (X) and standard deviation (s). Where the LCS/LCSD replicates are utilized for IDOC evaluation, those same pass/fail control limits will demonstrate accuracy and precision capability.

**II.** If an analyst performs the desorption efficiency series of spikes, that analyst does not need to perform the IDOC in addition to the DE study. The DE study satisfies the IDOC.

**III.** An analyst also must be "authorized" to perform the analysis per AIHA/ELLAP. The approval is documented using the "Demonstration of Capability Authorization/Certification Statement", form PX-QAD-005. This document must be in the analyst's training file before they can analyze industrial hygiene samples.

**ELLAP Initial Demonstration of Capabilities (IDOCs)**

**I.** All analysts and technicians shall have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples.

**II.** For initial demonstration, analysts and technicians shall complete a minimum of four (4) independent test runs of sample preparation and/or instrumental analysis. Independent runs are defined as analytical runs consisting of at least five (5) samples of spiked with a CRM of known and varying concentrations within the calibration range, separated by a period of time sufficient to evaluate the performance of any previous independent run.

**III.** For sample preparation training, the recoveries of the associated reference materials or proficiency training materials for each run must be within ± 10% of the certified value, 75% of the time. For instrumental analysis training, the recoveries of the associated reference materials or proficiency training samples for each run must be within ± 10% of the certified value, 75% of the time.

**12.4.3** **Ongoing Demonstration of Capability:** Every six months for AIHA and ELLAP (annually for NELAC) each analyst must demonstrate ongoing proficiency. This can be accomplished through: a) acceptable analysis of a Performance Testing (PT) sample; b) by acceptable analysis of at least 2 pairs of LCS/LCSD that were analyzed during the six month period with

acceptance recovery of 80 – 120% and ≤ 20% RPD for precision; or c) by repeating the IDOC as described in this SOP with acceptance recovery of 80 – 120% and ≤ 20% RPD for precision.

**I.** The acceptable demonstration for "b" or "c" is by acceptably performing and documenting the data of four LCS (normally defined as two LCS/LCSD (BS/BSD) sets), its approval by the respective department manager, and by approval of the QA Department. Acceptable demonstration is added to the analyst's training file.

**II.** For AIHA/NELAC/ELLAP, another "Demonstration of Capability Authorization/Certification Statement" must also be completed.

## 12.5 Control Limits (Procedure/Method Acceptance Criteria)

**12.5.1** Once control limits have been established (in-house or by method), they are verified, reviewed, and updated if necessary on an annual basis unless the method or regulatory authority requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**12.5.2** More information concerning Control Limits can be found in the QA Manual and in the current version of SOP: PE-QAD-001 Control Charts and Statistical Process Control.

## 12.6 Measurement Uncertainty (NELAC/AIHA Requirement)

**12.6.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**12.6.2** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 ± 0.5 mg/L.

**12.6.3** Refer to the Quality Assurance Manual for further discussion of uncertainty.

## 13.0 Pollution Control

**13.1** It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 14.0 Waste Management

**14.1** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Section 13 – Waste Management and Pollution Prevention of the Corporate Environmental Health and Safety Manual (CW-E-M-001) or the current Waste Management SOP.

**14.2** The following waste streams are produced when this method is carried out.

**14.2.1** Acid waste consisting of sample, hydrochloric, nitric acid and rinse solution, is added to the existing facility acidic waste stream.

**I.** **Acidic waste:** Acidic waste is stored in appropriately labeled hazardous waste containers in satellite storage areas throughout the lab, and should be emptied as often as possible. The waste should be transferred to the storage area in these exact same containers. The snorkel valve on the fume hood exhaust tubing should be turned 90 degrees clockwise to direct flow to the snorkel. The drum openings should then be opened with a wrench. The carboy placed over one opening and the snorkel placed over the other opening. The valve for the carboy should then be opened thereby transferring the contents of the carboy into the waste drum.

**14.2.2 Expired primary and working standards:**

**I.** Low concentration: Dispose of in the solvent waste stream.
**II.** High concentration: Lab pack.

**14.3** Any waste, which does not fit into any of the waste streams or cannot be disposed of in any of the other drums, should be brought directly to the department and/or waste manager's attention.

## 15.0 References / Cross-References

**15.1** Perkin Elmer Optima 5300 Family Hardware Guide.

**15.2** Perkin Elmer ICP WinLab Software Guide.

**15.3** Perkin Elmer Sciex Elan 6100 ICP/MS Hardware and Software guides.

**15.4** Concepts, Instrumentation, and Techniques in Inductively Coupled Plasma Optical Emission Spectrometry (Perkin Elmer) Second Edition, 1997.

**15.5** NIOSH Method 7300 March 2003, "Elements by ICP", NIOSH Manual of Analytical Methods (NMAM), Fourth Edition.

**15.6** NIOSH Method 7303 March 2003, "Elements by ICP", NIOSH Manual of Analytical Methods (NMAM), Fourth Edition.

**15.7** Metal & Metalloid Particulates in Workplace Atmospheres (Atomic Absorption), OSHA ID-121, U.S. Department of Labor. Revised Feb. 2002

**15.8** Metal & Metalloid Particulates in Workplace Atmospheres (ICP Analysis), OSHA ID-125G, U.S. Department of Labor. Revised September 2002.

**15.9** AIHA Policies for Laboratory Quality Assurance Programs, April 1, 2010.

**15.10** TestAmerica Laboratories, Inc.
- Environmental Health and Safety Manual, CW-E-M-001.

**15.11** TestAmerica – Phoenix
- Quality Assurance Manual, PX-QAD-011.
- SOP PE-SFT-001 Sample Disposal and Waste Management.
- SOP PE-QAD-008 Personnel Certification and Training.
- SOP PE-QAD-019 Determination of Method Detection Limits.
- SOP PE-QAD-013 Reagent and Standard Preparation, Control and Documentation.
- PX-QAD-007 Training Documentation Form.
- PX-QAD-069 Group Training Documentation Form.

## 16.0 Method Modifications:

Digestions modified from NIOSH and OSHA methods.

## 17.0 Attachments

Attachment 1: NIOSH Method 7300 Table 3 Measurement Procedures and Data
Attachment 2: NIOSH Method 7300 Table 4 Measurement Procedures and Data
Attachment 3: Reporting Limits
Attachment 4: Perkin Elmer IEC, MSF Guidelines
Attachment 5: Inter-Element Correction Equations

## 18.0 Revision History

Revision 0, dated April 2, 2008

- Integration for TestAmerica and STL operations, SOP previously numbered SOP 07-018.01 in AEL scheme.

Revision 1, dated on February 17, 2009
- Transfer of the SOP into the corporate template.
- Minor revisions to clarify NELAC standards and AIHA policies.

Revision 2, dated on November 16, 2010
- Added the digestion procedures from SOP PE-MET-011 which eliminated that procedure.
- Added the requirements for the ELLAP (Lead) program to the SOP.

Revision 3, dated November 29, 2012
- Removed references to sections of old versions of the QA Manual.
- Section 1.1 – Removed Silver, Tellurium, Platinum, Thorium and Tungsten from the Analyte table.
- Section 3.3. – Changed CCV from secondary source standard to primary source. Added notes about CCV levels for ELLAP vs IH/NELAC.
- Section 9.1 and 9.2.5 – Changed acceptable Internal Standard recovery limits for ICPMS.
- Section 10.1.4 – Added procedure about wiping the cassette with a wet filter.
- Section 10.4.4 – Added an additional standard to the ICPMS calibration table.
- Section 10.5.2 – Changed placement of RLV on the sequence/batch sample order table.
- Section 10.7 – Added information about ICPMS Maintenance.
- Attachment 3 – Reporting Limit table updated.

Revision 4, dated April 7, 2015
- Replaced CAR with NCM throughout the document.
- Section 3.12 – Added frequency requirements for IHLAP and ELLAP.
- Section 3.13 – Added entire section
- Section 3.14 – Added entire section
- Section 5.0 – Added “closed-heel” to paragraph
- Section 7.11 – Added entire section
- Section 9.2.4 – Revised requirement for results that exceed calibration range
- Section 9.2.13 – Added new section
- Section 9.3 – Updated ELLAP QC Chart
- Section 9.4.5 – Revised requirement for results that exceed calibration range
- Section 9.5 – Added requirements for Prep Blank throughout section
- Section 10.4.4 – Updated Calibration Standards Chart for ICP-MS
- Section 10.4.5 – Added Isotopes Monitored and Equations Used Table
- Section 10.5.2 – Revised sequence table
- Section 11.2 – Added entire section
- Section 12.2.4 – Added ELLAP section to RLV section
- Section 12.4.2 – Added ELLAP IDOC requirements
- Section 17.0 – Added Attachments 4 and 5

## Attachment 1

## NIOSH Method 7300 Table 3 Measurement Procedures and Data

ELEMENTS (ICP): METHOD 7300, Issue 3, dated 15 March 2003 - Page 7 of 8

**TABLE 3. MEASUREMENT PROCEDURES AND DATA [1].**
**Mixed Cellulose Ester Filters (0.45 µm)**

| Element (a) | wavelength nm | Est. LOD µg/ Filter | LOD ng/mL | Certified 3x LOD (b) | % Recovery (c) | Percent RSD (N=25) | Certified 10x LOD (b) | % Recovery (c) | Percent RSD (N=25) |
|---|---|---|---|---|---|---|---|---|---|
| Ag | 328 | 0.042 | 1.7 | 0.77 | 102.9 | 2.64 | 3.21 | 98.3 | 1.53 |
| Al | 167 | 0.115 | 4.6 | 1.54 | 105.4 | 11.5 | 6.40 | 101.5 | 1.98 |
| As | 189 | 0.140 | 5.6 | 3.08 | 94.9 | 2.28 | 12.9 | 93.9 | 1.30 |
| Ba | 455 | 0.005 | 0.2 | 0.31 | 101.8 | 1.72 | 1.29 | 97.7 | 0.69 |
| Be | 313 | 0.005 | 0.2 | 0.31 | 100.0 | 1.44 | 1.29 | 98.4 | 0.75 |
| Ca | 317 | 0.908 | 36.3 | 15.4 | 98.7 | 6.65 | 64.0 | 100.2 | 1.30 |
| Cd | 226 | 0.0075 | 0.3 | 0.31 | 99.8 | 1.99 | 1.29 | 97.5 | 0.88 |
| Co | 228 | 0.012 | 0.5 | 0.31 | 100.8 | 1.97 | 1.29 | 98.4 | 0.90 |
| Cr | 267 | 0.020 | 0.8 | 0.31 | 93.4 | 16.3 | 1.29 | 101.2 | 2.79 |
| Cu | 324 | 0.068 | 2.7 | 1.54 | 102.8 | 1.47 | 6.40 | 100.6 | 0.92 |
| Fe | 259 | 0.095 | 3.8 | 1.54 | 103.3 | 5.46 | 6.40 | 98.0 | 0.95 |
| K | 766 | 1.73 | 69.3 | 23.0 | 90.8 | 1.51 | 96.4 | 97.6 | 0.80 |
| La | 408 | 0.048 | 1.9 | 0.77 | 102.8 | 2.23 | 3.21 | 100.1 | 0.92 |
| Li | 670 | 0.010 | 0.4 | 0.31 | 110.0 | 1.91 | 1.29 | 97.7 | 0.81 |
| Mg | 279 | 0.098 | 3.9 | 1.54 | 101.1 | 8.35 | 6.40 | 98.0 | 1.53 |
| Mn | 257 | 0.005 | 0.2 | 0.31 | 101.0 | 1.77 | 1.29 | 94.7 | 0.73 |
| Mo | 202 | 0.020 | 0.8 | 0.31 | 105.3 | 2.47 | 1.29 | 98.6 | 1.09 |
| Ni | 231 | 0.020 | 0.8 | 0.31 | 109.6 | 3.54 | 1.29 | 101.2 | 1.38 |
| P | 178 | 0.092 | 3.7 | 1.54 | 84.4 | 6.19 | 6.40 | 82.5 | 4.75 |
| Pb | 168 | 0.062 | 2.5 | 1.54 | 109.4 | 2.41 | 6.40 | 101.7 | 0.88 |
| **Sb** | 206 | 0.192 | 7.7 | 3.08 | 90.2 | 11.4 | 12.9 | **41.3** | 32.58 |
| Se | 196 | 0.135 | 5.4 | 2.3 | 87.6 | 11.6 | 9.64 | 84.9 | 4.78 |
| **Sn** | 189 | 0.040 | 1.6 | 0.77 | 90.2 | 18.0 | 3.21 | **49** | 21.79 |
| Sr | 407 | 0.005 | 0.2 | 0.31 | 101.0 | 1.55 | 1.29 | 97.3 | 0.65 |
| Te | 214 | 0.078 | 3.1 | 1.54 | 102.0 | 2.67 | 6.40 | 97.4 | 1.24 |
| Ti | 334 | 0.050 | 2.0 | 0.77 | 98.4 | 2.04 | 3.21 | 93.4 | 1.08 |
| Tl | 190 | 0.092 | 3.7 | 1.54 | 100.9 | 2.48 | 6.40 | 99.1 | 0.80 |
| V | 292 | 0.028 | 1.1 | 0.77 | 103.2 | 1.92 | 3.21 | 98.3 | 0.84 |
| **W** | 207 | 0.075 | 3.0 | 1.54 | **72.2** | 10.1 | 6.40 | **57.6** | 14.72 |
| Y | 371 | 0.012 | 0.5 | 0.31 | 100.5 | 1.80 | 1.29 | 97.4 | 0.75 |
| Zn | 213 | 0.310 | 12.4 | 4.60 | 102.2 | 1.87 | 19.3 | 95.3 | 0.90 |
| **Zr** | 339 | 0.022 | 0.9 | 0.31 | 88.0 | 19.4 | 1.29 | **25** | 57.87 |

(a) Bold values are qualitative only because of low recovery.
(b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD
(c) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.

NIOSH Manual of Analytical Methods (NMAM), Fourth Edition

## Attachment 2

## NIOSH Method 7300 Table 4 Measurement Procedures and Data



**TABLE 4. MEASUREMENT PROCEDURES AND DATA [1].**
**Polyvinyl Chloride Filter (5.0 µm)**

| Element (c) | wavelength nm | Est. LOD µg per filter | LOD ng/mL | Certified 3x LOD (b) | % Recovery (a) | Percent RSD (N=25) | Certified[17] 10x LOD (b) | % Recovery (a) | Percent RSD (N=25) |
|---|---|---|---|---|---|---|---|---|---|
| Ag | 328 | 0.042 | 1.7 | 0.78 | 104.2 | 8.20 | 3.18 | 81.8 | 18.9 |
| Al | 167 | 0.115 | 4.6 | 1.56 | 77.4 | 115.24 | 6.40 | 92.9 | 20.9 |
| As | 189 | 0.140 | 5.6 | 3.10 | 100.7 | 5.13 | 12.70 | 96.9 | 3.2 |
| Ba | 455 | 0.005 | 0.2 | 0.31 | 102.4 | 3.89 | 1.270 | 99.8 | 2.0 |
| Be | 313 | 0.005 | 0.2 | 0.31 | 106.8 | 3.53 | 1.270 | 102.8 | 2.1 |
| **Ca** | 317 | 0.908 | 36.3 | 15.6 | **68.1** | 12.66 | 64.00 | 96.8 | 5.3 |
| Cd | 226 | 0.0075 | 0.3 | 0.31 | 105.2 | 5.57 | 1.27 | 101.9 | 2.8 |
| Co | 228 | 0.012 | 0.5 | 0.31 | 109.3 | 4.67 | 1.27 | 102.8 | 2.8 |
| Cr | 267 | 0.020 | 0.8 | 0.31 | 109.4 | 5.31 | 1.27 | 103.4 | 4.1 |
| Cu | 324 | 0.068 | 2.7 | 1.56 | 104.9 | 5.18 | 6.40 | 101.8 | 2.4 |
| Fe | 259 | 0.095 | 3.8 | 1.56 | 88.7 | 46.82 | 6.40 | 99.1 | 9.7 |
| K | 766 | 1.73 | 69.3 | 23.4 | 96.4 | 4.70 | 95.00 | 99.2 | 2.2 |
| **La** | 408 | 0.048 | 1.9 | 0.78 | **45.5** | 4.19 | 3.18 | 98.8 | 2.6 |
| Li | 670 | 0.010 | 0.4 | 0.31 | 107.7 | 4.80 | 1.27 | 110.4 | 2.7 |
| **Mg** | 279 | 0.098 | 3.9 | 1.56 | **54.8** | 20.59 | 6.40 | **64.5** | 5.7 |
| Mn | 257 | 0.005 | 0.2 | 0.31 | 101.9 | 4.18 | 1.27 | 99.3 | 2.4 |
| Mo | 202 | 0.020 | 0.8 | 0.31 | 106.6 | 5.82 | 1.27 | 98.1 | 3.8 |
| Ni | 231 | 0.020 | 0.8 | 0.31 | 111.0 | 5.89 | 1.27 | 103.6 | 3.2 |
| P | 178 | 0.092 | 3.7 | 1.56 | 101.9 | 17.82 | 6.40 | 86.5 | 10.4 |
| Pb | 168 | 0.062 | 2.5 | 1.56 | 109.6 | 6.12 | 6.40 | 103.2 | 2.9 |
| **Sb** | 206 | 0.192 | 7.7 | 3.10 | **64.6** | 22.54 | 12.70 | **38.1** | 30.5 |
| Se | 196 | 0.135 | 5.4 | 2.30 | 83.1 | 26.23 | 9.50 | 76.0 | 17.2 |
| **Sn** | 189 | 0.040 | 1.6 | 0.78 | 85.7 | 27.29 | 3.18 | **52.0** | 29.4 |
| **Sr** | 407 | 0.005 | 0.2 | 0.31 | **71.8** | 4.09 | 1.27 | 81.2 | 2.7 |
| Te | 214 | 0.078 | 3.1 | 1.56 | 109.6 | 7.49 | 6.40 | 97.3 | 3.8 |
| Ti | 334 | 0.050 | 2.0 | 0.78 | 101.0 | 9.46 | 3.18 | 92.4 | 5.5 |
| Tl | 190 | 0.092 | 3.7 | 1.56 | 110.3 | 4.04 | 6.40 | 101.9 | 2.0 |
| V | 292 | 0.028 | 1.1 | 0.78 | 108.3 | 3.94 | 3.18 | 102.5 | 2.6 |
| **W** | 207 | 0.075 | 3.0 | 1.56 | **74.9** | 15.79 | 6.40 | **44.7** | 19.6 |
| Y | 371 | 0.012 | 0.5 | 0.31 | 101.5 | 3.63 | 1.27 | 101.4 | 2.5 |
| Zn | 213 | 0.310 | 12.4 | 4.70 | 91.0 | 68.69 | 19.1 | 101.0 | 9.6 |
| **Zr** | 339 | 0.022 | 0.9 | 0.31 | **70.7** | 54.20 | 1.27 | **40.4** | 42.1 |

(a) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.
(b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD [12].
(c) Bold values are qualitative only because of low recovery. Other digestion techniques may be more appropriate for these elements and their compounds.



UNCONTROLLED

## Attachment 3

## Reporting Limits

| Compound | ICP (mg/l) | ICP/MS (mg/l) |
|---|---|---|
| Aluminum | 0.1 | -- |
| Antimony | 0.1 | 0.003 |
| Arsenic | 0.1 | 0.001 |
| Barium | 0.02 | 0.003 |
| Beryllium | 0.001 | 0.01 |
| **Bismuth** | 0.2 | -- |
| Boron | 0.1 | -- |
| Cadmium | 0.001 | 0.001 |
| Calcium | 0.8 | -- |
| Chromium | 0.1 | 0.05 |
| Cobalt | 0.02 | 0.01 |
| Copper | 0.02 | 0.003 |
| Gallium | 0.1 | |
| Indium | 0.1 | |
| Iron | 0.1 | -- |
| Lead | 0.0125 | 0.001 |
| **Lithium** | 0.2 | -- |
| Magnesium | 0.8 | -- |
| Manganese | 0.02 | 0.003 |
| Molybdenum | 0.02 | 0.001 |
| **Nickel** | 0.02 | 0.003 |
| Phosphorus | 0.1 | -- |
| Potassium | 0.8 | -- |
| Selenium | 0.1 | 0.002 |
| Silicon | 0.1 | -- |
| Silver | 0.01 | 0.001 |
| Sodium | 2.0 | -- |
| **Strontium** | 0.02 | -- |
| Thallium | 0.1 | 0.001 |
| **Tin** | 0.2 | -- |
| **Titanium** | 0.1 | -- |
| Vanadium | 0.02 | 0.01 |
| Zinc | 0.1 | 0.05 |

Attachment 4
Perkin Elmer Inter-Element Correction Factors (IEC's), MSF Guidelines

The Instrument is calibrated as usual and analytes are analyzed.

Per Corporate Quality Memorandum CA-Q-QM-004 the following analytes need to be analyzed at the following concentrations.

| Element | Mg/L | Element | mg/L |
|---|---|---|---|
| Aluminum | 1000 | Silicon | 200 |
| Antimony | 50 | Silver | 2 |
| Arsenic | 50 | *Sodium | 10000 |
| Barium | 50 | Strontium | 20 |
| Beryllium | 20 | Thallium | 50 |
| Boron | 100 | Tin | 50 |
| Cadmium | 25 | Titanium | 25 |
| Calcium | 2000 | Vanadium | 50 |
| Chromium | 50 | Zinc | 50 |
| Cobalt | 50 | | |
| Copper | 50 | | |
| Iron | 2000 | | |
| Lead | 200 | | |
| Lithium | 25 | | |
| Magnesium | 1500 | | |
| Manganese | 20 | | |
| Molybdenum | 20 | | |
| Nickel | 50 | | |
| Phosphorous | 50 | | |
| Potassium | 500 | | |
| Selenium | 50 | | |

*Sodium cannot be analyzed at 10000 ppm without the instrument saturating. 2000 ppm Sodium will be used due to instrument limitations.

All analytes must be enabled when running the IEC's. The instrument must be calibrated and the pure standards analyzed.

Data is then examined to see if the pure analytes have false positives or negatives.

To build the IEC's table on the PE ICP:
Under tools; go to Interelement Correction Model Builder
Select the file that the IEC's were run in and the method that was used will be pulled in automatically.

Go to the Set Limits tab and set the limits to the corresponding reporting limit.
Go to the Calculate Factors tab and pull in the corresponding analyte. This will pull in the corresponding analytes and concentrations obtained.
Go to summarize factors and save factors obtained and save under a new method. DO NOT override current method. Print new factors and recalibrate and check ICSAB and ICSA recoveries.

## Attachment 5

Inter-Element Correction Equations

| Interfering Elements – ICP 2 | | | | | | |
|---|---|---|---|---|---|---|
| **Analytes** | **Al 396.153** | **As 188.979** | **Ca 317.933** | **Co 228.616** | **Cr 267.716** | **Cu 327.393** |
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 36.0741 | 0 | -4.06756 | 0.0550049 | 0 |
| **Cd9 226.502 Test Only** | -0.0567175 | 0 | 0 | 0 | -0.0836365 | 0 |
| **Fe 238.204** | 0 | 0 | 0 | -61.8411 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 3.83838 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -2.87154 | 0 |
| **Y-Axial** | 5332.69 | 144699 | 745.589 | 18907.5 | 5168.92 | 10826.6 |
| **Y Radial** | 5107.74 | 137982 | 712.112 | 18968.5 | 4992.94 | 10106.6 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | -17.07 | 0 |

| **Analytes** | **Fe 238.204** | **K 766.490** | **Mg 286.213** | **Mn 257.610** | **Mo 202.031** | **Ni 231.604** |
|---|---|---|---|---|---|---|
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0.11263 | 0 | 0 |
| **Cd9 226.502 Test Only** | 0.0567652 | -0.0697293 | 0 | 0 | 0 | -1.84753 |
| **Fe 238.204** | NA | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0.214893 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -10.3999 | 0 |
| **Y-Axial** | 1111.11 | 6614.6 | 509.263 | 10213 | 24213.7 | 34498 |
| **Y Radial** | 1055.17 | 6357.84 | 506.259 | 10189.3 | 2326.1 | 32570.7 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | 0 | 0 |

| **Analytes** | **Pb 220.353** | **Sn 189.927** | **Sr 421.552** | **Ti 334.940** | **V 290.880** |
|---|---|---|---|---|---|
| **Be 313.107** | 0 | 0 | 0 | -2.49635 | 0 |

| | | | | | |
|---|---|---|---|---|---|
| **Cd 228.802** | 0 | 0.306267 | 0 | 0 | 0.144568 |
| **Cd9 226.502 Test Only** | 0 | 0 | 0 | 0 | 0 |
| **Fe 238.204** | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 10.7209 |
| **V 290.880** | 0 | 0 | 0 | 0 | NA |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | 423.297 |
| **Y-Axial** | 21006.3 | 30732.1 | 0 | 19778.8 | 10269.4 |
| **Y Radial** | 20017.7 | 29538.7 | -2.58606e+008 | 0 | 9912.32 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | 0 |

**Interfering Elements – ICP 3**

| **Analytes** | **Al 396.153** | **As 188.978** | **Ca 317.933** | **Co 228.616** | **Cr 267.716** | **Cu 327.393** |
|---|---|---|---|---|---|---|
| **Al 396.153** | NA | 0 | 0 | 0 | 0 | 0 |
| **Ba 233.527** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Be 313.107** | 0 | 0 | 0 | 0 | -0.0624805 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Co 228.616** | 0 | 0 | 0 | NA | -0.401774 | 0 |
| **Cr 267.716** | 0 | 0 | 0 | 0 | NA | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0.258709 | 0 | NA |
| **Pb 220.353** | 0 | 0 | 0 | 0 | 0 | 0.329886 |
| **Sb 206.836** | -0.176763 | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Y-Axial** | 237.723 | 2071.65 | 301.519 | 1759.67 | 1019.69 | 1011.68 |
| **Y Radial** | 250.593 | 2103.71 | 316.149 | 1787.51 | 1045.07 | 1022.15 |

| **Analytes** | **Fe 238.204** | **K 766.490** | **Mg 286.213** | **Mn 257.610** | **Mo 202.031** |
|---|---|---|---|---|---|
| **Al 396.153** | 0 | 0 | 0 | 0 | 31.4537 |
| **Ba 233.527** | 0.0348397 | 0 | 0 | 0 | 0 |
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0.0549947 |
| **Co 228.616** | 0 | 0 | 0 | 0 | 0 |
| **Cr 267.716** | -0.0537566 | 0 | 0.106385 | 0 | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0 | 0 |
| **Pb 220.353** | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | -0.267406 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 9.58585 |
| **V 290.880** | 0.0692878 | 0 | -0.630446 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -9.85142 |
| **Y-Axial** | 323.615 | 222.825 | 960.459 | 2094.07 | 2039.61 |
| **Y Radial** | 330.127 | 230.086 | 985.197 | 2162.61 | 2064.3 |

| **Analytes** | **Ni 231.604** | **Pb 220.353** | **Sn 189.927** | **Ti 334.940** | **V 290.880** |
|---|---|---|---|---|---|
| **Al 396.153** | 0 | 0 | 0 | 0 | 0 |
| **Ba 233.527** | 0 | 0 | 0 | 0 | 0 |

| | | | | | |
|---|---|---|---|---|---|
| **Be 313.107** | 0 | 0 | 0 | -0.212013 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0.058736 |
| **Co 228.616** | 0.347667 | 0 | 0 | 0 | 0 |
| **Cr 267.716** | 0 | 0 | 0 | 0 | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0 | 0 |
| **Pb 220.353** | 0 | NA | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 24.3777 | 103.303 | 468.6 |
| **V 290.880** | 0 | 0 | 0 | 0 | NA |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0.4356 | 0 |
| **Y-Axial** | 1865.93 | 983.348 | 1892.45 | 2037.43 | 1843.2 |
| **Y Radial** | 1887.47 | 1000.28 | 1937.43 | 2074.41 | 1860.88 |

Attachment 4
Perkin Elmer Inter-Element Correction Factors (IEC's), MSF Guidelines

The Instrument is calibrated as usual and analytes are analyzed.

Per Corporate Quality Memorandum CA-Q-QM-004 the following analytes need to be analyzed at the following concentrations.

| Element | Mg/L | Element | mg/L |
|---|---|---|---|
| Aluminum | 1000 | Silicon | 200 |
| Antimony | 50 | Silver | 2 |
| Arsenic | 50 | *Sodium | 10000 |
| Barium | 50 | Strontium | 20 |
| Beryllium | 20 | Thallium | 50 |
| Boron | 100 | Tin | 50 |
| Cadmium | 25 | Titanium | 25 |
| Calcium | 2000 | Vanadium | 50 |
| Chromium | 50 | Zinc | 50 |
| Cobalt | 50 | | |
| Copper | 50 | | |
| Iron | 2000 | | |
| Lead | 200 | | |
| Lithium | 25 | | |
| Magnesium | 1500 | | |
| Manganese | 20 | | |
| Molybdenum | 20 | | |
| Nickel | 50 | | |
| Phosphorous | 50 | | |
| Potassium | 500 | | |

Selenium 50

*Sodium cannot be analyzed at 10000 ppm without the instrument saturating. 2000 ppm Sodium will be used due to instrument limitations.

All analytes must be enabled when running the IEC's. The instrument must be calibrated and the pure standards analyzed.

Data is then examined to see if the pure analytes have false positives or negatives.

To build the IEC's table on the PE ICP:

Under tools; go to Interelement Correction Model Builder
Select the file that the IEC's were run in and the method that was used will be pulled in automatically.
Go to the Set Limits tab and set the limits to the corresponding reporting limit.
Go to the Calculate Factors tab and pull in the corresponding analyte. This will pull in the corresponding analytes and concentrations obtained.
Go to summarize factors and save factors obtained and save under a new method.
DO NOT override current method. Print new factors and recalibrate and check ICSAB and ICSA recoveries.

## Attachment 5

Inter-Element Correction Equations

| Interfering Elements – ICP 2 | | | | | | |
|---|---|---|---|---|---|---|
| **Analytes** | **Al 396.153** | **As 188.979** | **Ca 317.933** | **Co 228.616** | **Cr 267.716** | **Cu 327.393** |
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 36.0741 | 0 | -4.06756 | 0.0550049 | 0 |
| **Cd9 226.502 Test Only** | -0.0567175 | 0 | 0 | 0 | -0.0836365 | 0 |
| **Fe 238.204** | 0 | 0 | 0 | -61.8411 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 3.83838 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -2.87154 | 0 |
| **Y-Axial** | 5332.69 | 144699 | 745.589 | 18907.5 | 5168.92 | 10826.6 |
| **Y Radial** | 5107.74 | 137982 | 712.112 | 18968.5 | 4992.94 | 10106.6 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | -17.07 | 0 |

| **Analytes** | **Fe 238.204** | **K 766.490** | **Mg 286.213** | **Mn 257.610** | **Mo 202.031** | **Ni 231.604** |
|---|---|---|---|---|---|---|
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0.11263 | 0 | 0 |
| **Cd9 226.502 Test Only** | 0.0567652 | -0.0697293 | 0 | 0 | 0 | -1.84753 |
| **Fe 238.204** | NA | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0.214893 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -10.3999 | 0 |
| **Y-Axial** | 1111.11 | 6614.6 | 509.263 | 10213 | 24213.7 | 34498 |
| **Y Radial** | 1055.17 | 6357.84 | 506.259 | 10189.3 | 2326.1 | 32570.7 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | 0 | 0 |

| **Analytes** | **Pb 220.353** | **Sn 189.927** | **Sr 421.552** | **Ti 334.940** | **V 290.880** |
|---|---|---|---|---|---|
| **Be 313.107** | 0 | 0 | 0 | -2.49635 | 0 |
| **Cd 228.802** | 0 | 0.306267 | 0 | 0 | 0.144568 |
| **Cd9 226.502 Test Only** | 0 | 0 | 0 | 0 | 0 |
| **Fe 238.204** | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 10.7209 |
| **V 290.880** | 0 | 0 | 0 | 0 | NA |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | 423.297 |
| **Y-Axial** | 21006.3 | 30732.1 | 0 | 19778.8 | 10269.4 |

| | | | | | |
|---|---|---|---|---|---|
| **Y Radial** | 20017.7 | 29538.7 | -2.58606e+008 | 0 | 9912.32 |
| **Zn 206.200** | 0 | 0 | 0 | 0 | 0 |

**Interfering Elements – ICP 3**

| Analytes | Al 396.153 | As 188.978 | Ca 317.933 | Co 228.616 | Cr 267.716 | Cu 327.393 |
|---|---|---|---|---|---|---|
| **Al 396.153** | NA | 0 | 0 | 0 | 0 | 0 |
| **Ba 233.527** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Be 313.107** | 0 | 0 | 0 | 0 | -0.0624805 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Co 228.616** | 0 | 0 | 0 | NA | -0.401774 | 0 |
| **Cr 267.716** | 0 | 0 | 0 | 0 | NA | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0.258709 | 0 | NA |
| **Pb 220.353** | 0 | 0 | 0 | 0 | 0 | 0.329886 |
| **Sb 206.836** | -0.176763 | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V 290.880** | 0 | 0 | 0 | 0 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | 0 | 0 |
| **Y-Axial** | 237.723 | 2071.65 | 301.519 | 1759.67 | 1019.69 | 1011.68 |
| **Y Radial** | 250.593 | 2103.71 | 316.149 | 1787.51 | 1045.07 | 1022.15 |

| Analytes | Fe 238.204 | K 766.490 | Mg 286.213 | Mn 257.610 | Mo 202.031 |
|---|---|---|---|---|---|
| **Al 396.153** | 0 | 0 | 0 | 0 | 31.4537 |
| **Ba 233.527** | 0.0348397 | 0 | 0 | 0 | 0 |
| **Be 313.107** | 0 | 0 | 0 | 0 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0.0549947 |
| **Co 228.616** | 0 | 0 | 0 | 0 | 0 |
| **Cr 267.716** | -0.0537566 | 0 | 0.106385 | 0 | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0 | 0 |
| **Pb 220.353** | 0 | 0 | 0 | 0 | 0 |
| **Sb 206.836** | -0.267406 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 0 | 0 | 9.58585 |
| **V 290.880** | 0.0692878 | 0 | -0.630446 | 0 | 0 |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0 | -9.85142 |
| **Y-Axial** | 323.615 | 222.825 | 960.459 | 2094.07 | 2039.61 |
| **Y Radial** | 330.127 | 230.086 | 985.197 | 2162.61 | 2064.3 |

| Analytes | Ni 231.604 | Pb 220.353 | Sn 189.927 | Ti 334.940 | V 290.880 |
|---|---|---|---|---|---|
| **Al 396.153** | 0 | 0 | 0 | 0 | 0 |
| **Ba 233.527** | 0 | 0 | 0 | 0 | 0 |
| **Be 313.107** | 0 | 0 | 0 | -0.212013 | 0 |
| **Cd 228.802** | 0 | 0 | 0 | 0 | 0.058736 |
| **Co 228.616** | 0.347667 | 0 | 0 | 0 | 0 |
| **Cr 267.716** | 0 | 0 | 0 | 0 | 0 |
| **Cu 327.393** | 0 | 0 | 0 | 0 | 0 |
| **Pb 220.353** | 0 | NA | 0 | 0 | 0 |
| **Sb 206.836** | 0 | 0 | 0 | 0 | 0 |
| **Si 251.611** | 0 | 0 | 24.3777 | 103.303 | 468.6 |
| **V 290.880** | 0 | 0 | 0 | 0 | NA |
| **V9 292.402 Test Only** | 0 | 0 | 0 | 0.4356 | 0 |

| | | | | | |
|---|---|---|---|---|---|
| **Y-Axial** | 1865.93 | 983.348 | 1892.45 | 2037.43 | 1843.2 |
| **Y Radial** | 1887.47 | 1000.28 | 1937.43 | 2074.41 | 1860.88 |

McCampbell

# METHOD 8082

# POLYCHLORINATED BIPHENYLS (PCBs)

# BY GAS CHROMATOGRAPHY

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# McCampbell Analytical Inc.

## METHOD 8082
## POLYCHLORINATED BIPHENYLS (PCBs)
## BY GAS CHROMATOGRAPHY

**Number:O – 8082** **Rev. No.: 2** **DATE: 11-4-13**

**Reviewed and Approved By:** _____ _____
Angela Rydelius Date

**Reviewed and Approved By:** _____ _____
Christine Horn Date

**Reviewed and Approved By:** _____ _____

## *Disclaimer*

The 2003 NELAC Standard, Chapter 5 Quality Systems, Section 5.5.4.1.1, Parts a), b), & f), (pg. 207 of 324) states with regard to laboratory SOPs,

> a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.
>
> b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual (see 5.5.4.1.2).
>
> f) The documents specified in 5.5.4.1.1 a) and 5.5.4.1.1 b) that contain sufficient information to perform the tests do not need to be supplemented or rewritten as internal procedures, if the documents are written in a way that they can be used as written. Any changes, including the use of a selected option must be documented and included in the laboratory's methods manual.

In accordance with these instructions, this SOP is an internally written document that acts as a supplement to the published method it references. This SOP does not stand alone and is to be used in conjunction with the published method. Instrument specific instructions, quality control summaries, as well as internal MAI policies are referenced in this SOP, including any deviations from the published method, if any such deviations exist. In the absence of a stated deviation, this SOP adheres strictly to all the requirements of the published method, regardless of whether or not those requirements are explicitly stated in this document.

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# Standard Operating Procedure:

## METHOD 8082
## POLYCHLORINATED BIPHENYLS (PCBs)
## BY GAS CHROMATOGRAPHY

### 1. Scope and Application

1.1. Method 8082 is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors or as individual PCB congeners in extracts from solid and aqueous matrices. Open-tubular capillary columns are employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target compounds listed below may be determined by either a single- or dual-column analysis system. The PCB congeners listed below have been tested by this method, and the method may be appropriate for additional congeners.

| Compound | CAS Registry No. | IUPAC # |
|---|---|---|
| Aroclor 1016 | 12674-11-2 | – |
| Aroclor 1221 | 11104-28-2 | – |
| Aroclor 1232 | 11141-16-5 | – |
| Aroclor 1242 | 53469-21-9 | – |
| Aroclor 1248 | 12672-29-6 | – |
| Aroclor 1254 | 11097-69-1 | – |
| Aroclor 1260 | 11096-82-5 | – |
| 2-Chlorobiphenyl | 2051-60-7 | 1 |
| 2,3-Dichlorobiphenyl | 16605-91-7 | 5 |
| 2,2',5-Trichlorobiphenyl | 37680-65-2 | 18 |
| 2,4',5-Trichlorobiphenyl | 16606-02-3 | 31 |
| 2,2',3,5'-Tetrachlorobiphenyl | 41464-39-5 | 44 |
| 2,2',5,5'-Tetrachlorobiphenyl | 35693-99-3 | 52 |
| 2,3',4,4'-Tetrachlorobiphenyl | 32598-10-0 | 66 |
| 2,2',3,4,5'-Pentachlorobiphenyl | 38380-02-8 | 87 |
| 2,2',4,5,5'-Pentachlorobiphenyl | 37680-73-2 | 101 |
| 2,3,3',4',6-Pentachlorobiphenyl | 38380-03-9 | 110 |
| 2,2',3,4,4',5'-Hexachlorobiphenyl | 35065-28-2 | 138 |
| 2,2',3,4,5,5'-Hexachlorobiphenyl | 52712-04-6 | 141 |
| 2,2',3,5,5',6-Hexachlorobiphenyl | 52663-63-5 | 151 |
| 2,2',4,4',5,5'-Hexachlorobiphenyl | 35065-27-1 | 153 |
| 2,2',3,3',4,4',5-Heptachlorobiphenyl | 35065-30-6 | 170 |
| 2,2',3,4,4',5,5'-Heptachlorobiphenyl | 35065-29-3 | 180 |
| 2,2',3,4,4',5',6-Heptachlorobiphenyl | 52663-69-1 | 183 |
| 2,2',3,4',5,5',6-Heptachlorobiphenyl | 52663-68-0 | 187 |
| 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl | 40186-72-9 | 206 |

1.2. Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

1.3. Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment. Therefore, this method provides procedures for the determination of selected individual PCB congeners. The 19 PCB congeners listed above have been tested by this method.

1.4. The PCB congener approach potentially affords greater quantitative accuracy when PCBs are known to be present. As a result, this method may be used to determine Aroclors, some PCB congeners, or "total PCBs," depending on regulatory requirements and project needs. The congener method is of particular value in determining weathered Aroclors. However, analysts should use caution when using the congener method when regulatory requirements are based on Aroclor concentrations.

1.5. Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS Method 8270 is also recommended as a confirmation technique when sensitivity permits.

## 2. Method Summary

2.1. A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique.

2.2. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), or micro extraction technique.

2.3. Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), ), or micro extraction technique.

2.4. Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds.

2.5. After cleanup, the extract is analyzed by injecting an aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

2.6. The chromatographic data may be used to determine the seven Aroclors in Sec. 1.1, individual PCB congeners, or total PCBs.

**3. Definitions**

3.1. ANALYTICAL BATCH – A group of up to 20 field samples analyzed on the same instrument. An analytical batch is not related to matrix, and may consist of samples from several preparatory batches.

3.2. CALIBRATION STANDARD – Prepared from the primary dilution standard solution or stock standard solutions.

3.3. CONTINUING CALIBRATION BLANK (CCB) – A blank matrix ran at the beginning of a sample sequence (i.e., before the CCV, degradation check, and sample analysis), to verify the instrumentation is free from contamination. A CCB is associated with an *analytical batch*, not a *preparatory batch.*

3.4. CONTINUING CALIBRATION VERIFICATION STANDARD (CCV) – A mid-level CAL solution analyzed before sample analysis in a sample sequence in order to confirm that the initial calibration is still valid. *At MAI, the term CCV is also used for the initial calibration verification (ICV) standard.*

3.5. INITIAL CALIBRATION STANDARDS – A series of CAL solutions used for initial calibration.

3.6. INITIAL CALIBRATION VERIFICATION STANDARD (ICV) – An individual CAL solution analyzed prior to any sample analysis, at the beginning of the day, which verifies initial/historical calibration curves. *At MAI, the term CCV is also used for the ICV standard.*

3.7. INITIAL DEMONSTRATION OF CAPABILITY (IDOC) – A method is prepared and determined which generates data of acceptable accuracy and precision for a reference sample containing the target analytes in a clean matrix. Establish the ability to generate acceptable accuracy and precision.

3.8. INSTRUMENT BLANK (IB) – A blank matrix ran at the beginning of a sample sequence (i.e., before the CCV, degradation check, and sample analysis), to verify the instrumentation is free from contamination. An IB is associated with an *analytical batch*, not a *preparatory batch* .

3.9. LABORARORY CONTROL SAMPLE (LCS*)* – An aliquot of clean matrix (reagent water, Ottawa sand, wipe, CT, etc.) are spiked with midrange levels of analyte and run like a sample. They are analyzed as samples, and % recoveries (accuracy) calculated in order to determine whether the methodology is in control.

3.10. MATRIX SPIKE AND MATRIX SPIKE DUPLICATE (MS/MSD) – Two aliquots of an environmental sample to which midrange levels of method analytes are added. They are analyzed as samples, and % recoveries (accuracy) and %RPD (precision) calculated with the purpose of determining whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes must be determined in a separate aliquot and subtracted.

3.11. METHOD BLANK (MB) – A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. Each *preparatory batch* requires a Method Blank. For aqueous samples, reagent water is treated exactly as a sample, including exposure to glassware, extraction, etc. For soil and solid samples, no matrix is used. For wipes, charcoal tubes, etc, an unused sample medium is extracted exactly as a sample. Its purpose is to determine if contamination is present from any of the sample preparation or analytical steps.

3.12. METHOD DETECTION LIMIT (MDL) – The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

3.13. PREPARATORY BATCH – A group of up to 20 field samples of the same matrix, that is associated with a set of Quality Control samples (for example, a MB, LCS /LCSD, and MS/MSD).

3.14. QUALITY CONTROL SAMPLE (QCS) – A control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

3.15. REPORTING LIMIT (RL) or *PRACTICAL QUANTITATION LIMIT (PQL)* – The lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions. The RL cannot be lower than the lowest calibration standard used in the initial calibration of the instrument and must be greater than the MDL.

3.16. DUPLICATE SAMPLE – Prepare by dividing a sample into two or more separate aliquots.

3.17. SPIKE – A known amount of an analyte added to a blank, quality control sample, field sample or sub-sample.

3.18. STANDARD CURVE – A curve that plots the concentrations of known standards of an analyte versus the instrument response to the analyte.

## 4. Interferences

4.1. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories.

4.1.1. Contaminated solvents, reagents, or sample processing hardware.

4.1.2. Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.1.3. Compounds extracted from the sample matrix to which the detector will respond.

4.2. Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

4.2.1. Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters.

4.2.2. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2.3. These materials can be removed through the use of Method 3665 (sulfuric acid/permanganate cleanup).

4.3. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned.

4.4. Elemental sulfur ($S_8$) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660.

4.5. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. A final solvent rinse with high purity grade solvent is done.

## 5. Safety

5.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the

chemicals specified in this method. Good laboratory technique dictates the use of appropriate dermal protection. A laboratory coat, eye protection, and gloves are the minimum requirements.

## 6. Equipment & Supplies

6.1. Gas Chromatographs (with MAI instrument ID numbers):

6.1.1. GC-5: Agilent Technologies 6890A Network GC System, Serial #: LR47359C

6.1.2. GC-15: Agilent Technologies 6890A Network GC System, Serial #: US00033554

6.1.3. GC-20: Agilent Technologies 6890N Network GC System, Serial #: US10317086

6.1.4. GC-23: Agilent Technologies 6890N Network GC System, Serial #: CN10612058

6.2. Autosampler: Agilent Technologies or HP 7683 Series

6.3. Injector: Agilent Technologies or HP 7683 Series

6.4. Columns: 1st Column ("Front"): DB35MS & 2nd Column ("Back"): DB5MS.

6.5. Electron Capture Detector (ECD)

6.6. Crimp-top vial, 2 mL

6.7. Volumetric Flasks, various sizes.

6.8. ChemStation Software.

## 7. Reagents & Standards

7.1. Surrogate Standard (SS):

7.1.1. The surrogate standard is Decachlorobiphenyl.

7.1.2. Dilute 1 mL of 200 ppm stock standard into 4 L solvent to produce a final concentration of 50 µg/L.

7.2. Internal Standard (IS):

7.2.1. The internal standard is Tetrachloro-m-xylene.

7.2.2. Dilute 1 mL of 200 ppm stock standard into 4 L solvent to produce a final concentration of 50 µg/L.

7.3. Calibration Standards:

7.3.1. Prepare the initial calibration standards as described in Table 3.

7.4. Continuing Calibration Verification (CCV) Standard:

7.4.1. Dilute 50 µL of the 16 ppm initial calibration stock standard solution into 40 mL of hexane plus IS and SS, to produce a final concentration of 20 µg/L.

7.5. Continuing Calibration Blank (CCB):

7.5.1. The solvent plus IS and SS.

7.6. Method Blank (MB):

7.6.1. Solvent plus IS and SS. Extract using the same procedure as for the samples. *Per NELAC, add appropriate amount of SS to reagent water, extract and concentrate like a sample, and add IS immediately before analysis.*

7.7. Laboratory Control Sample & Duplicate (LCS/LCSD):

7.7.1. Prepare as described in the following table:

| LCS/LCSD & MS/MSD Analyte List | Stock Std. Conc. (mg/L) | Vol Stock Std. (µL) | Vol Solvent (mL) | Intermediate Std. Conc. (mg/L) | Water QC Standards | | | | Soil QC Standards | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Vol Inter. Std. (µL) | Vol Solvent (mL) | Raw Conc. (µg/L) | Final Conc. (µg/L) | Vol Inter. Std. (µL) | Vol Solvent (mL) | Raw Conc. (µg/L) | Final Conc. (mg/Kg) |
| Aroclor1260 | 1,000 | 600 | 4 | 150 | 1 | 1 | 150 | 3.75 | 10 | 10 | 150 | 0.075 |

## 8. Sample Collection, Preservation, Shipment & Storage

8.1. Collect samples in contaminant free glass containers. Aqueous samples can be collected in 1L amber glass liter with TLE (Teflon lined enclosure), and 2 g to 30 g for solid samples in glass jars with TLE or metal liners (MAI).

8.2. For aqueous samples with no residual chlorine present, cool to 4$^{o}$C. For aqueous samples with residual chlorine present, add 3 mL 10% sodium thiosulfate solution per gallon (or 0.008%) and cool to 4$^{o}$C. NOTE: Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use.

8.3. Extract samples within 7 days of collection. Store extracts at 4$^{o}$C in the dark.

8.4. Analyze extracts within 40 days of extraction.

8.5. Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as high as a year.

## 9. Quality Control

9.1. IDOC: Each analyst for a given method must analyze at least 4 replicates of a mid-level standard extracted from a water matrix using the same procedure as for a water sample. Percent recoveries and RSDs for each analyte may be compared to values published in the method and should meet those criteria if they are from multi-laboratory studies, although this is not a strict requirement, and the development of in-house guidelines is strongly recommended. In the absence of recommended acceptance criteria the suggested rule of thumb is 70-130% recovery [MAI RPD ±20%].

9.2. An MDL study must be performed using a minimum of 7 extracted replicates for each matrix

9.3. One Instrument Blank (the CCB) must be analyzed at the beginning of every sequence, before the breakdown standard and CCV.

9.4. One Method Blank (MB) must be analyzed with each analysis batch, before processing any samples. The MB must be carried through the entire extraction process, including any clean up procedures. When samples that are extracted together are analyzed on separate instruments, the method blank associated that batch is analyzed on one instrument and a solvent blank on the other instrument(s). The results must be

below the Method RLs before continuing analysis. If not, corrective action must be taken. The only exception to this rule occurs if all samples in that sequence are ND less than the RL.

9.5. Add at least one Internal Standard (IS) to each sample, blank, standard or spiked sample prior to analysis.

9.6. Add at least one Surrogate Standard (SS) to each sample, blank, standard or spiked sample prior to its processing & extraction. SS recovery must be 70% – 130%. Dilution may cause the SS to be inaccurately measured, and if possible, less dilute injections may be used to evaluate SS recovery in these cases.

9.7. An LCS containing all analytes must be prepared and analyzed with each analysis batch to demonstrate the lab's ability to perform the analysis in a clean matrix. Spike at the same concentration as the MS/MSD. Recovery must be within laboratory established limits and the RPD should be = 30%. If the LCS fails to meet these criteria the source of the problem must be identified and resolved before continuing analyses.

9.8. An MS/MSD pair containing all analytes should be prepared and analyzed for every analysis batch. The percent recovery should be within laboratory established limits and the RPD should be = 30%, or matrix induced bias may be a factor.

9.9. Retention times for each analyte and the IS are established during every initial calibration. The analyst selects the RT using ChemStation by QEditing (i.e., manually integrating) the peak of each analyte in each calibration standard. Each analyte must have the same retention time in each of the calibration standards. A retention time that shifts during initial calibration indicates the presence of a serious problem (such as a leak) that requires maintenance. After each initial calibration, a new calibration method is saved in ChemStation, establishing that method's retention times. NOTE: Establishing a new method not only changes the retention time of an analyte, but also the instrument response of an analyte.

9.9.1. During the evaluation of the CCV standard, if the RT of any analyte shifts by greater than ± 0.05 minutes from the RT established during initial calibration, re-establish retention times by picking the peak in ChemStation and updating the current method. If the internal standard shifts one direction, all of the analytes should exhibit the same behavior (i.e., shift in the same direction and by the same amount). If the internal standard shifts, and the analytes retain the same retention time or shift in the opposite direction, this indicates a major problem exists. If the analytes shift retention times, but the internal standard does not, this also indicates a problem.

9.9.2. During sample analysis, if an analyte peak does not fall within ± 0.05 minutes of the established retention time (the "RT Window") for that analyte, that analyte is not considered present. However, if the RT of the internal standard shifts in a sample, then the RT of all sample analytes should shift by that time (i.e., if the IS shifts by + 0.15 minutes, and the established RT of analyte A is 3.0 minutes, then the new RT of A in that sample will become 3.15 ± 0.05 minutes). NOTE: The RT of the IS may shift in "dirty" samples (i.e., samples with cluttered chromatograms) or samples that contain a non-target analyte that elutes at the same time or near the IS.

9.9.3. Retention times often shift after a change in guard column, analytical column, injector liner, or after any major maintenance.

9.10. When confirmation is made on a second column, that analysis should meet all of the QC criteria described above, including calibration. Confirmation may not be necessary if the composition of the sample matrix is well established by prior analyses, (for instance, when a pesticide known to be produced or used in a facility is found in a sample from that facility).

9.11. When sample results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below:

$$RPD = 100 * |R1 - R2| / ( (R1+R2)/2 )$$

R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference.

9.11.1. If the RPD is >40%, check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result (peak coelution) or for coincidental peak identification based on pattern mismatch. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

9.11.2. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The higher result should always be reported with a narrative explaining the disparity between the results on the two columns.

9.11.3. MAI accepts overspiking of the unknown with an amount of known compound when there is a likely coelution mismatch. In the case when a compound is overspiked and it is observed that there is no increase in the peak of interest, that analyte may be reported as ND.

## 10. Calibration & Standardization

10.1. Initial Calibration:

10.1.1. A minimum five point calibration must be used to establish the calibration range of the method.

10.1.2. The lowest calibration point must be at the same concentration as the reporting limit.

10.1.3. Transfer an aliquot of each standard into an auto sampler vial and cap.

10.1.4. Load on auto sampler rack and analyze in order of least to most concentrated.

10.1.5. Calibration curves must be generated for each compound. These curves are generated by the ChemStation software.

10.1.6. ***Linear calibration using the average response factor*** – When the Relative Standard Deviation (RSD = [SD / $RF_{avg}$]) of the mean response factor for a single analyte is ≤ 20% over the entire calibration range, use the average response factor to determine sample concentrations (the calibration curve is assumed to be linear and to pass through the origin). Even if the RSD for one or more analytes exceeds 20%, linear calibration using the average RF may still be acceptable for these compounds if the following conditions are met:

10.1.6.1. The mean of the RSD values for ALL analytes in the calibration is ≤ 20%, and,

10.1.6.2. The user/client is provided a specific list of the compounds for which the RSD exceeded 20% and the results of the mean RSD calculations.

10.1.6.3. The alternative to using an average RF curve that exceeds 20% RSD is to adjust the calibration range until the RSD is ≤ 20%. Failing that, use a different calibration type.

10.1.7. ***Linear calibration using a least squares regression*** – If the RSD of the average response factor exceeds 20% over the calibration range, then linearity through the origin cannot be assumed. The analyst may use a linear regression fit that is not forced through the origin, but have the intercept calculated from the data points. The data points should be unweighted and not include the origin (0,0) as a calibration point. Only when combining replicate multi-point calibrations may data points be weighted.. The use of a linear regression may not be used as a rationale for reporting results outside of the calibration range. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purpose, r ≥ 0.99 is a strict requirement.

10.1.8. ***Non-linear calibration*** – To be suitable for non-linear calibration, the data point curve must be continuous, continuously differentiable, and monotonic over the calibration range. The fit equation may not have more than 4 parameters (for example, a third order polynomial). Do not force the line through the origin and do not include the origin (0,0) as a calibration point. Six calibration standards are required for a quadratic fit (this is the ChemStation curve fit) and seven for a cubic fit. The coefficient of the determination (COD) or 'goodness of fit' will equal 1.0 for a "perfect" fit model. In order to be an acceptable non-linear calibration, COD ≥ 0.99 is a strict requirement. The COD value is displayed in ChemStation on page 1 of the compound's calibration record.

10.1.9. Whichever curve fit option is used, it must yield a unique concentration for every response (area).

10.2. Calibration Verification:

10.2.1. Prior to sample analysis, verify calibration each 12 hour shift by injecting a Continuing Calibration Verification (CCV) Standard. The percent recovery of each analyte must be within ± 15%. However, if this criterion is exceeded for any analyte, the 'grand mean' (i.e., the average % recovery of *all* analytes in the CCV) may be used to verify calibration provided that the 'grand mean' is = 15%. If the 'grand mean' is employed, the user/client must be provided with the calibration verification data or a list of those analytes that exceeded ± 15%.

10.2.2. If the calibration does not meet the ±15% limit (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, perform corrective action, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within ±15%, then a new initial calibration must be prepared.

10.2.3. The CCV standard must also be analyzed every 20 samples and at the end of the analysis sequence and satisfy the criteria of recovery ± 15% or 'grand mean' = 15%. When a CCV fails to meet the QC criteria, all samples that were injected after the last CCV that last met the QC criteria must be re-injected to prevent mis-quantitations and possible false negative results.

10.2.3.1. The only exception occurs when the CCV analyzed after a group of samples exhibits a response for an analyte that exceed 115%, and that analyte was ND in the samples analyzed during the analytical shift. In this case (CCV fails high but all samples ND), the extracts for those samples do not need to be reanalyzed.

10.3 When confirmation is made on a second column, that analysis should meet all of the calibration criteria described above for initial calibration and calibration verification.

10.3.1 The only exception occurs when the CCV fails high on the second detector and the samples are ND.

## 11. Procedure

11.1. Samples, standards, and QC are received from the extractionist in 2 mL crimp-top vials. Each vial must have a label with the MAI sample ID and batch # printed on it.

11.2. Place samples on the autosampler, and type the corresponding sample ID in ChemStation.

11.3. The first sample in the sequence is the Continuing Calibration Blank (CCB), followed by the CCV, and finally samples and QC (in any order).

11.4. All integrations are performed using ChemStation.

## 12. Data Analysis & Calculations

12.1. Samples are analyzed in a set referred to as a sequence. The sequence always begins with the CCB and CCV. Once the CCV has passed the criteria, the instrument is considered to be in calibration and is ready to analyze samples and/or sample extracts.

12.2. All applicable QC samples including method blanks are included in the remainder of the sequence. In order for the sequence to be considered valid, the QC samples must pass all criteria outlined in Quality Control section above.

12.3. In the event that any of the CCVs or QC samples fail their criteria they should be immediately reanalyzed.

12.4. Whenever silica gel (method 3630) or florisil (method 3620) cleanups are used, the analyst must demonstrate that the fractionation scheme is reproducible.

12.5. All integrations are performed using ChemStation software.

12.6. Each Aroclor is identified by a series of characteristic peak patterns in the chromatogram:

12.6.1. Aroclors 1016, 1232, 1242, and 1248 each have 3 characteristic peaks. For a positive identification, at least 2 of the 3 peaks must be present in the chromatogram.

12.6.2. Aroclors 1221, 1254, and 1260 each have 4 characteristic peaks. For a positive identification, at least 3 of the 4 peaks must be present in the chromatogram.

**13. Method Performance**

13.1. Estimated quantitation limits for PCBs as congeners vary by congener, in the range of 5 - 25 ng/L in water and 160 - 800 ng/kg in soils, with the higher values for the more heavily chlorinated congeners.

13.2. The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

13.3. During method performance studies, the concentrations determined, as Aroclors were larger than those obtained using the congener method. In certain soils, interference prevented the measurement of congener 66. Recoveries of congeners from soils spiked with Aroclor 1254 and Aroclor 1260 were between 80% and 90%. Recoveries of congeners from environmental reference materials ranged from 51 - 66% of the certified Aroclor values.

**14. Pollution Prevention**

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14.2. The chemicals used in this method pose little threat to the environment when properly managed.

14.3. All standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of disposable waste.

14.4. For further information on pollution prevention consult *Less is better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington D.C. 20036, (202) 872-4477.

**15. Data assessment and acceptance criteria for quality control measures.**

15.1 Refer to section 9.

**16. Corrective Actions for out of control data.**

16.1. Refer to Nonconformance/Corrective Action Report (NC/CAR/PR) Procedure.

**17. Contingencies for handling out of control data or unacceptable data.**

17.1. Contact the laboratory manager or technical manager to assess out of control data.

**18. Waste Management**

18.1 All wastes must be disposed of safely, samples and extracts are disposed of following laboratory SOP.

**19. References**

19.1. United States Environmental Protection Agency. 1996. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods. EPA SW-846, 3rd Edition, Method 8082. U.S. EPA, Washington, D.C.

# Instrumental Analysis of Trace Metals by ICP-MS

(EPA Method 6020, Revision 0)

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## McCampbell Analytical Inc.

### Analytical Method

**Number: M- 6020 Rev. No.: 2 Date: 11-4-13**

# Instrumental Analysis of Trace Metals by ICP-MS

# (EPA Method 6020, Revision 0)

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## *Disclaimer*

The 2003 NELAC Standard, Chapter 5 Quality Systems, Section 5.5.4.1.1, Parts a), b), & f), (pg. 207 of 324) states with regard to laboratory SOPs,

> a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.
>
> b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual (see 5.5.4.1.2).
>
> f) The documents specified in 5.5.4.1.1 a) and 5.5.4.1.1 b) that contain sufficient information to perform the tests do not need to be supplemented or rewritten as internal procedures, if the documents are written in a way that they can be used as written. Any changes, including the use of a selected option must be documented and included in the laboratory's methods manual.

In accordance with these instructions, this SOP is an internally written document that acts as a supplement to the published method it references. This SOP does not stand alone and is to be used in conjunction with the published method. Instrument specific instructions, quality control summaries, as well as internal MAI policies are referenced in this SOP, including any deviations from the published method, if any such deviations exist. In the absence of a stated deviation, this SOP adheres strictly to all the requirements of the published method, regardless of whether or not those requirements are explicitly stated in this document.

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# Standard Operating Procedure:
# Instrumental Analysis of Trace Metals by ICP-MS
# (EPA Method 6020)

## 1. Scope and Application

1.1 This method is applicable to the determination of sub-µg/L concentrations of elements in soil, sludge and solid, water and wastewater samples and in waste extracts or digests by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

1.2 When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

1.3 The following elements are acceptable to be analyzed by method 6020. Metals not listed (ex: Mo) are permitted to be analyzed by 6020 if the analyst determines the accuracy and precision of the method for that element in the waste to be analyzed.

| | |
|---|---|
| Aluminum (Al) | Magnesium (Mg) |
| Antimony (Sb) | Manganese (Mn) |
| Arsenic (As) | Mercury (Hg) |
| Barium (Ba) | Nickel (Ni) |
| Beryllium (Be) | Potassium (K) |
| Cadmium (Cd) | Selenium (Se) |
| Calcium (Ca) | Silver (Ag) |
| Chromium (Cr) | Sodium (Na) |
| Cobalt (Co) | Thallium (Tl) |
| Copper (Cu) | Vanadium (V) |
| Iron (Fe) | Zinc (Zn) |
| Lead (Pb) | |

## 2. Summary of Method

2.1 Prior to analysis, samples which require total ("acid-leachable") values must be digested using appropriate sample preparation methods (such as Methods 3005 - 3051).

2.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

**3. Definitions**

3.1. ***Analysis Batch*** – A group of up to 20 field samples.

3.2. ***Calibration Check*** – Made from a stock solution which is different from the stock used to prepare the calibration standards to verify the ratio of instrument response to analyte amount.

3.3. ***Calibration Standard (CAL)*** – Prepared from the primary dilution standard solution or stock standard solutions.

3.4. ***Continuing Calibration Verification Standard (CCV)*** – Individual CAL solution analyzed.

3.5. ***Equipment Blank*** – Appropriate reagent grade media is opened in the field and poured appropriately over or through the sample collection device, collected in a sample container and returned to the lab as a sample. Its purpose is to check the cleanliness of the sampling device.

3.6. ***Field Blank*** – May be a trip blank or equipment blank.

3.7. ***Initial Calibration Standards*** – A series of Cal solutions used for initial calibration.

3.8. ***Initial Calibration Verification Standard (ICV)*** – An individual mid-range standard analyzed prior to any sample analysis, which verifies initial/historical calibration curves. EPA 6020A requires that it is a "second source standard".

3.9. ***Initial Demonstration Of Capability (IDOC)*** – A method is prepared and determined which generates data of acceptable accuracy and precision for a reference sample containing the target analytes in a clean matrix.

3.10. ***Instrument Check Standard (ICS)*** *or Interference Check Solution* – A solution is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections.

3.11. ***Instrument Detection Limits (IDLs)*** – The average calculation of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day reports in µg/L.

3.12. ***Instrument Performance Check Solution*** **(IPC)** – A midrange calibration standard.

3.13. ***Laboratory Control Sample (LCS )*** *or Laboratory Fortified Blank (LFB)* – An aliquot of reagent water spiked with midrange levels of analyte and run like a sample. Their purpose is to assess accuracy, and to determine whether the methodology is in control.

3.14. ***Method Blank (MB)*** *or Laboratory Reagent Blank (LRB)* – Reagent water treated exactly as a sample including exposure to glassware, etc. Its purpose is to determine if contamination is present in any of the sample preparation or analytical steps.

3.15. ***Method Detection Limit (MDL)*** – The minimum concentration of analyte that can be reported, which can be no lower than the lowest calibration standard.

3.16. ***Matrix Spike and Matrix Spike Duplicate (MS/MSD)*** *or Laboratory Fortified Matrix (LFM)* – Two aliquots of an environmental sample to which midrange levels of method analytes are added. They are analyzed as a sample, with the purpose of determining whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes must be determined in a separate aliquot and subtracted.

3.17. ***Practical Quantitation Limit (PQL)*** – The lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions.

3.18. ***Quality Control Sample (QCS)*** – A solution of method analytes of known concentration that is used to spike an aliquot of RLB or sample matrix. The QCS must be from an external source and different than that of the calibration standards.

3.19. ***Replicate Sample*** – Prepare by dividing a sample into two or more separate aliquots.

3.20. ***Standard Curve*** – A curve which plots concentrations of known anlayte standard versus the instrument response to the analyte.

3.21. ***Surrogate*** – Compounds that are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are added to all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.22. ***Trip Blank*** – Aliquot of reagent water or solvent in sealed containers that brought to the field and transported together with the field samples back to the lab.

## 4. Interferences

4.1. All known interferents (or their derivatives, such as alternate isotope of an interfering element) must be measured in addition to the target analyte. Table 3 shows a list of known isobaric interferents.

4.2. Where an interference source is present, the analyte impacted must be flagged for QA completeness to indicate: a) the percent of interference correction applied to the data, or b) an uncorrected interference by virtue of the elemental equation used for quantitation. Observed rather than theoretical (ex: $^{35}Cl/^{37}Cl = 3.13$) should be used to make interferent correction and chelation or collision cells can greatly diminish polyatomic interferents.

4.3. When intolerable physical interferences are present in a sample (due to sample nebulization, transport processes, and ion-transmission efficiencies), dilution of the sample fivefold (1+4) will usually eliminate the problem.

4.4. Memory interferences can be diminished with adequate rinse time between samples.

## 5. Safety

5.1. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a reference file of material safety data sheets for the chemicals specified in this method. Additional information on general laboratory safety is available from the Laboratory Safety Officer.

5.2. Good laboratory technique dictates the use of appropriate dermal protection. A laboratory coat, eye protection, and gloves are the minimum requirements.

## 6. Equipment & Supplies

6.1. Analytical balance

6.2. Beakers, volumetric flasks, and other glassware, as needed

6.3. 60 mL digestion bottles

6.4. Watch glass, ribbed

6.5. Fume Hood

6.6. Hot block

6.7. Pipettes

6.8. Instrumentation

| Instrument # | Equipment | Manufacturer | Model | Serial/ID No. |
|---|---|---|---|---|
| ICPMS1 | ICPMS | Agilent | G3162A | JP14100782 |
| | chiller | MERLIN | m75 | |
| | Autosampler | Agilent | ASX-520 | |
| | computer | HP | | |
| | computer | DELL | | |
| ICPMS2 | ICPMS | Agilent | 7500cx (G3272B) | JP51202111 |
| | chiller | Agilent | G1879B | |
| | Autosampler | Agilent | ASX-500 | |
| | computer | HP | | |

## 7. Reagents & Standards

7.1. Deionized (DI) Water

7.2. Hydrochloric acid (HCl), conc.

7.3. Nitric acid ($HNO_3$), conc.

7.4. Nitric acid ($HNO_3$), 1%.

7.5. Terbium (Tb) Stock Standard, 10,000 mg/L. Terbium is used as the Surrogate Standard.

7.5.1. Nitric acid/Terbium solvent: Carefully add 6.25 mL of the 10,000 mg/L Terbium stock standard into 4 L of Nitric acid, to produce a final concentration of 25 mg/L Terbium.

7.5.2. Per the solid digestion procedure referenced in Section 7.0, 10 mL of the 25 mg/L Terbium/ $HNO_3$ solution is included in the digestion.

**8. Sample Collection, Preservation and Storage**

8.1 Aqueous samples should be collected in polyethylene or fluorocarbon (TFE or PFA) plastic and soils in core tubes or glass jars.

8.2 Sample preservation and holding time are as follows:

A. *Metals excluding h exavalent chromium and mercury*:

a. Aqueous samples: (200 Series define aqueous samples as they have < 1% sediments)

1. Total: $HNO_3$ to pH<2, 6 months (200 Series call this Total Recoverable and requires a pH check on each sample. Also, if acid is added in the lab, 200 Series require that the sample be acidified 16-18 hours prior to analysis).
2. Dissolved: Filter on site, $HNO_3$ to pH<2, 6 months.
3. Suspended: Filter on site, 6 months

b. Solid samples:

1. Total: 6 months

**9. Quality Control**

**- Initial Demosntration -**

9.1. IDOC: Each analyst for a given method must analyze at least 4 replicates of a mid-level standard extracted from a water matrix using the same procedure as for a water sample. Percent recoveries and RSDs for each analyte may be compared to values published in the method and should meet those criteria if they are from multi-laboratory studies, although this is not a strict requirement, and the development of in-house guidelines is strongly recommended. In the absence of recommended acceptance criteria the suggested rule of thumb is 70-130% recovery [MAI RPD ±20%]. Please see SOP MAI-13 IDOC for detailed instructions.

9.2. An MDL study must be performed using a minimum of 7 extracted replicates for each matrix.

9.3. The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard in a sample falls below 30% of the intensity in the initial calibration standard, and instrument drift is not the cause, the sample must diluted until the 30% criteria is met. Nebulization efficiency varies with solution viscosity and IS is used to normalize this effect. Total Dissolved Solids < 0.2% is recommended for this reason as well as to prevent solids deposition at the cones.

9.4. To obtain analyte data of known quality, measure more than the analyte(s) of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantification and the concentration of interferents are insignificant, then the data may go uncorrected.

9.5. One dilution test (serial dilution) must be included for each twenty samples or less of each matrix in a batch. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank), an analysis of fivefold (1+4) dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected.

9.6. One MB must be analyzed with each analysis batch [MAI prefers to analyze it before processing any sample]. The results must be below the MDL before continuing the analysis. If not, corrective action must be taken.

9.7. Surrogate response – Terbium (Tb) is used by MAI as the Surrogate Standard for metals analysis. Tb is a rare Earth element that is not typically found in environmental samples at the level spiked. Each sample and standard that undergoes digestion is spiked with Tb during digestion, as described in 6.5. The response of the surrogate must be ± 30 % of the spike level.

9.8. An LCS must be prepared and analyzed with each analysis batch [MAI: if the recovery of any analyte falls outside the control limits of 75-125%, the source of the problem should be identified and resolved before continuing analyses].

*9.9.* An MS/MSD pair must be prepared and analyzed with each analysis batch. The spike may be added post-digestion. Calculate the percent recovery and RPD for the MS/MSD pair. The percent recovery should be 75-125% and less than 20 relative percent difference (RPD) for precision. Recovery outside of 75-125%, requires that the sample be diluted and the diluted values must be 10% of the original values to permit matrix effects to be assumed. Otherwise the MS/MSD must be redone.

*9.10. Post-digestions spike addition: Spike a prepared solution, or its dilution. Recovery should be ± 25%. If the spike is not recovered within the specified limits, dilute sample and reanalyze. Results must agree to within 10% of the original determination.*

**10. Calibration and Standardization**

10.1. After turning the plasma on, the mass calibration and resolution checks must be verified to be less than 0.9 amu full width at 10 percent peak height. If the mass calibration differs more than 0.1 amu from the true value, then it must be adjusted to the correct value. A 10 µg/L solution of Li, Co, In, and Tl may be used to cover the entire mass range. This same solution may be analyzed 4 times with RSD ≤ 5% for each analyte to demonstrate that the instrument has come to thermal equilibrium prior to the analyses of the calibration standard.

10.2. Mixed Calibration Standard Solutions: Dilute the stock-standard solutions to levels in the linear range for the instrument in a solvent consisting of 1% (v/v) $HNO_3$ in reagent water [MAI uses 2% $HNO_3$ plus 1% HCl for all standards and samples]. The calibration standard solutions must contain an appropriate internal standard for each analyte. An internal standard should be no more than 50 amu removed from the analyte and choices include $^{6}Li$, $^{45}Sc$ (not for soil), $^{89}Y$, $^{103}Rh$, $^{115}In$, $^{159}Tb$, $^{169}Ho$, and $^{209}Bi$. Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine spectral interferences or the presence of impurities. Transfer the mixed standard solutions to freshly acid-cleaned FEP fluorocarbon bottles for storage.

10.3. Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve and must be matrix matched to the standard. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure and is also matrix matched. The rinse blank is used to flush the system between all samples and standards and is generally 1-2% $HNO_3$ and must contain $AuCl_3$ if mercury is being analyzed [MAI uses 1% $HNO_3$ plus 0.5% HCl].

10.4. Interference Check Solution (ICS): The ICS (table 1, solution AB) must be analyzed initially and every 12 hours to demonstrate the magnitude of elemental and molecular ion isobaric interferences and the adequacy of any corrections used. The following table is the recommended ICS components and concentration:

| Solution Component | Solution AB Concentration (mg/L) |
|---|---|
| Aluminum (Al) | 100.0 |
| Calcium (Ca) | 300.0 |
| Iron (Fe) | 250.0 |
| Magnesium (Mg) | 100.0 |
| Sodium (Na) | 250.0 |
| Phosphorous (P) | 100.0 |
| Potassium (K) | 100.0 |
| Sulfur (S) | 100.0 |
| Carbon (C) | 100.0 |
| Chlorine (Cl) | 2000.0 |
| Molybdenum (Mo) | 2.0 |
| Titanum (Ti) | 2.0 |
| Arsenic (As) | 0.100 |
| Cadmium (Cd) | 0.100 |
| Chromium (Cr) | 0.200 |
| Cobalt (Co) | 0.200 |
| Copper (Cu) | 0.200 |
| Manganese (Mn) | 0.200 |
| Mercury (Hg) | 0.020 |
| Nickel (Ni) | 0.200 |
| Selenium (Se) | 0.100 |
| Silver (Ag) | 0.050 |
| Vanadium (V) | 0.200 |
| Zinc (Zn) | 0.100 |

10.5. Calibrate the instrument for the analytes using the calibration blank and at least a single initial calibration standard. Flush the system with the rinse blank between each standard solution. Use at least 3 integrations for both calibration and sample analyses. Rinse for 5 or 10 minutes before the calibration blank is run to improve performance.

10.6. The calibration must be verified initially with a second source ICV and then with a CCV after every ten samples and at the end of the run. Each analyte of interest must be within 10% of its true value or reanalysis or recalibation is required before any further samples may be analyzed. The calibration blank (CCB) must be analyzed at the same frequency [MAI: and it should follow the CCV to estimate carryover]. The CCB must be < 3 times the current IDL.

**11. Procedure**

11.1 Samples are digested following 3050 and 3010 methods.

11.2. The rinse blank should be used to flush the system between solution changes for blanks, standards and samples. Solution should be aspired 30 seconds prior to the acquisition of data to allow equilibrium to be established.

11.3 Over-range samples must be diluted and reanalyzed.

**12. Data Analysis and Calculations**

12.1. All calculations are performed using the ICP-MS instrument software, Microsoft EXCEL, and the Laboratory Information Management System (LIMS). EXCEL and LIMS calculate the final results independently using the instrumental result, and the two calculations must agree before submitting the data for final review. If dilutions were performed, the appropriate factors must be applied to sample values.

12.2. Final analytical results are reported in mg/Kg (wet weight). The instrument calculates raw results in µg/L, without taking the dilution factor into account. Therefore, calculate the final result by multiplying the raw (in mg/L) by the extraction factor (default 1 g sample to 1000 mL solvent), conversion factors of 1L/1000mL and 1000g/Kg and 1mg/1000µg, and the dilution factor DF, if any dilution(s) were performed. The calculation is as follows:

*Final Result (mg/Kg) = Raw Instrumental Result (µg/L) • (1000mL/1g) • (1L/1000mL) • (1000g/Kg) • (1mg/1000µg) • DF*

12.3. The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must be also be provided.

12.4. If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

12.5. By default, analytical results lower than the reporting limit are reported as ND<RL (that is, non-detect less than the reporting limit), replacing "RL" with the appropriate limit for the non-detected analyte. However, if a client requests quantitation down to the Method Detection Limit (MDL), or requests J-Flag, then results lower than the MDL are reported as ND<MDL, replacing "MDL" with the appropriate limit. Results between the MDL and RL are reported with a J descriptor.

12.6. Calibration and Quality Control calculations are described in their respective sections of this SOP.

**13. Method Performance**

12.1 Table 5 has precision and accuracy data.

**14. Nonconformance & Corrective Action**

14.1. Refer to SOP , Nonconformance/Corrective Action Report (NC/CAR) Procedure.

**15. Data assessment and acceptance criteria for quality control measures.**

15.1 Refer to Table 2 for acceptance criteria.

**16. Pollution Prevention**

16.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

16.2. The chemicals used in this method pose little threat to the environment when properly managed.

16.3. All standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of disposable waste.

16.4. For further information on pollution prevention consult *Less is better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington D.C. 20036, (202) 872-4477.

**17. Contingencies for handling out of control data or unacceptable data.**

17.1 Contact the laboratory manager or technical manager to assess out of control data.

**18. Waste Management**

17.1. All wastes must be disposed of safely, samples and extracts are disposed of following laboratory SOP.

**19References**

19.1United States Environmental Protection Agency. 1994. Method 6020, Rev 0. U.S. EPA, Washington, D.C.

**Table 1: Example Target List & Reporting Limits for EPA 6020**

| Target List | Soil |
|---|---|
| | RL (mg/Kg) |
| Aluminum | 50 |
| Antimony | 0.5 |
| Arsenic | 0.5 |
| Barium | 5 |
| Beryllium | 0.5 |
| Boron | 1.6 |
| Cadmium | 0.25 |
| Calcium | 100 |
| Chromium | 0.5 |
| Cobalt | 0.5 |
| Copper | 0.5 |
| Iron | 20 |
| Lead | 0.5 |
| Lithium | 5 |
| Magnesium | 20 |
| Manganese | 20 |
| Mercury | 0.05 |
| Molybdenum | 0.5 |
| Nickel | 0.5 |
| Potassium | 20 |
| Selenium | 0.5 |
| Silver | 0.5 |
| Sodium | 100 |
| Strontium | 20 |
| Thallium | 0.5 |
| Thorium | 0.5 |
| Tin | 0.5 |
| Titanium | 0.5 |
| Tungsten | 0.5 |
| Uranium | 0.5 |
| Vanadium | 0.5 |
| Zinc | 5 |

# Table 2: Quality Control Summary

| QC Area: | Acceptance Criteria: | Frequency: | Notes: |
|---|---|---|---|
| Tune | The analyst should follow the instructions provided by the instrument manufacturer. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. This must be verified by analyzing a tuning solution at least four times with relative standard deviations of =5% for the analytes contained in the tuning solution. | With initial calibration. | Solution containing elements representing all mass regions of interest (ex, 10 µg/L of Li, Co, In, and Tl). Verifies that resolution and mass calibration of instrument are within required specifications and that instrument has reached thermal stability. |
| Interference Check Sample | | Verify magnitude of elemental and molecular-ion isobaric interferences and adequacy of any corrections at beginning of analytical run or once every 12 hours, whichever is more frequent, by analyzing interference check solutions A and AB. | ICS is prepared to contain known concentrations of interfering elements that will demonstrate magnitude of interferences and provide test of any corrections. There are three mixes specified in the method. |
| QCS | *Nothing stated* | *Nothing stated* | *Nothing stated* |
| Surrogate Standard | *Nothing stated* | *Nothing stated* | *Nothing stated* |
| Internal Standard | When intensity of any IS is >30-120% of intensity of that IS in initial calibration standard, sample must be diluted fivefold and reanalyzed with addition of appropriate amounts of internal standards. Repeat until IS intensities fall within window.<br><br>Intensity levels of ISs for calibration blank and instrument check standard must agree within ± 20 percent of intensity level of the IS of original calibration solution. | | |
| Initial Calibraton | Calibrate the instrument for the analytes of interest, using the calibration blank and at least a single initial calibration standard according to the instrument manufacturer's procedure. | | During the course of an analytical run, the instrument may be "resloped" or recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis ofa CCV and CCB before any further samples may be analyzed. |
| Calibration Verification | Recovery ±10%<br><br>Calibration blank must be <3 times the current IDL for each element | Immediately following calibration, after every ten samples, and at the end of an analytical run. | Must be prepared in same acid matrix as CAL standards. Must be independent standard near midpoint of linear range at concentration other than that used for instrument calibration. |
| MB (LRB) | | | |
| LCS (LFB) | *No criteria stated* | One LCS per batch. | |
| MS (LFM) | *Nothing stated* | *Nothing stated* | *Nothing stated* |
| Duplicate | RPD =20% | Analyze one duplicate sample for every matrix in a batch (one per 20 samples). | *This is not a requirement for a matrix spike duplicate, as in 6010B.* |
| Dilution Test | If analyte concentration is sufficiently high (factor of 10 above instrumental detection limit after dilution), a 1:5 dilution should agree within ±10% of original determination. | One dilution test must be included for each twenty samples (or less) of each matrix in a batch. | |
| Post-Digestion Spike Addition | Recovery 75-125% | | Spike addition should produce minimum level of 10 x and maximum of 100 x instrumental detection limit. |

## Table 3: Known Isobaric Interferents

| Molecular Ion | Mass | Element Interference |
|---|---|---|
| $ArAr^+$ | 80 | Se |
| $Ar^{81}Br^+$ | 121 | Sb |
| $ArCa^+$ | 80 | Se |
| $Ar^{35}Cl^+$ | 75 | As |
| $Ar^{37}Cl^+$ | 77 | Se |
| $ArK^+$ | 79 | |
| $Ar^{26}Mg$ | 66 | Zn |
| $ArNa^+$ | 63 | Cu |
| $ArO^+$ | 56 | Fe |
| $ArP^+$ | 71 | |
| $Ar^{32}S^+$ | 72 | |
| $Ar^{34}S^+$ | 74 | |
| $^{81}BrH^+$ | 82 | Se |
| $^{79}BrO^+$ | 95 | Mo |
| $^{81}BrO^+$ | 97 | Mo |
| $^{81}BrOH^+$ | 98 | Mo |
| $^{35}ClO^+$ | 51 | V |
| $^{35}ClOH^+$ | 52 | Cr |
| $^{37}ClO^+$ | 53 | Cr |
| $^{37}ClOH^+$ | 54 | Cr |
| $^{44}CaO^+$ | 60 | Ni |
| MoO | 108-116 | Cd |
| $PO^+$ | 47 | |
| $POH^+$ | 48 | |
| $PO_2^+$ | 63 | Cu |
| $^{32}SO^+$ | 48 | |
| $^{32}SOH^+$ | 49 | |
| $^{34}SO^+$ | 50 | V, Cr |
| $^{32}SOH^+$ | 51 | V |
| $SO_2^+$, $S^{2+}$ | 64 | Zn |
| TiO | 62-66 | Ni, Cu, Zn |
| ZrO | 106-112 | Ag, Cd |

## Table 4: ICP-MS Instrument Parameters

| Parameter | ICPMS |
|---|---|
| **ASX500 Configuration** | |
| COM Port for ALS | COM1 |
| Racks | |
| Rack ID 1 | 21 |
| Rack ID 2 | 60 |
| Rack ID 3 | 60 |
| Rack ID 4 | 60 |
| | |
| LAN | 192 168 1 93 |
| Remote Start | Don't use |
| Sample Introduction | |
| Type | Peristalic Pump |
| Autosampler | ASX500 |
| Prohibited Masses | 14 16 17 18 19 |
| | 32 36 38 41 … |
| EM Protection | Auto setting of integ time in analog mode |
| QC Mode | IS_GEN. QCC |
| | |
| Plasma Ignition Mode | Aquenous Solution |
| Remote Shutdowr | Standard |
| Dead Time Calibration | Use Dead Time Calibration |

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**Table 5:**

ICP-MS MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR AQUEOUS SOLUTIONS

| Element | Comparability Range | %RSD Range |
|---|---|---|
| Aluminum | 95 - 100 | 11 - 14 |
| Arsenic | 97 - 114 | 7.1 - 48 |
| Barium | 91 - 99 | 4.3 - 9.0 |
| Beryllium | 103 - 107 | 8.6 - 14 |
| Cadmium | 98 – 102 | 4.6 - 7.2 |
| Calcium | 99 - 107 | 5.7 - 23 |
| Chromium | 95 - 105 | 13 - 27 |
| Cobalt | 101 - 104 | 8.2 - 8.5 |
| Copper | 85 - 101 | 6.1 - 27 |
| Iron | 91 - 900 | 11 – 150 |
| Lead | 71 - 137 | 11 - 23 |
| Magnesium | 98 - 102 | 10 - 15 |
| Manganese | 95 - 101 | 8.8 - 15 |
| Nickel | 98 - 101 | 6.1 - 6.7 |
| Potassium | 101 - 114 | 9.9 - 19 |
| Selenium | 102 - 107 | 15 - 25 |
| Silver | 104 - 105 | 5.2 - 7.7 |
| Sodium | 82 - 104 | 24 - 43 |
| Thallium | 88 - 97 | 9.7 - 12 |
| Vanadium | 107 – 142 | 23 - 68 |
| Zinc | 93 – 102 | 6.8 - 17 |

NIOSH 5503 PCBs

mixture: $C_{12}H_{10-x}Cl_x$ [where x = 1 to 10] MW: ca. 258 (42% Cl ; $C_{12}H_7Cl_3$); ca. 326 (54% Cl ; $C_{12}H_5Cl_5$) CAS: Table 1 RTECS: Table 1

**METHOD: 5503, Issue 2** **EVALUATION: PARTIAL**

**Issue 1: 15 February 1984**
**Revision #1: 15 August 1987**
**Issue 2: 15 August 1994**

**OSHA :** 1 mg/m$^3$ (42% Cl); 0.5 mg/m$^3$ (54% Cl)
**NIOSH:** 0.001 mg/m$^3$/10 h (carcinogen)
**ACGIH:** 1 mg/m$^3$ (42% Cl) (skin); 0.5 mg/m$^3$ (54% Cl) (skin)

**PROPERTIES:** 42% Cl: BP 325 to 366 °C; MP -19 °C; d 1.38 g/mL @ 25 °C; VP 0.01 Pa (8 x $10^{-5}$ mm Hg; 1 mg/m$^3$) @ 20 °C
54% Cl: BP 365 to 390 °C; MP 10 °C; d 1.54 g/mL @ 25 °C; VP 0.0004 Pa (3 x $10^{-6}$ mm Hg; 0.05 mg/m$^3$) @ 20 °C

**SYNONYMS:** PCB; 1,1'-biphenyl chloro; chlorodiphenyl, 42% Cl (Aroclor 1242); and 54% Cl (Aroclor 1254)

## SAMPLING

**SAMPLER:** FILTER + SOLID SORBENT (13-mm glass fiber + Florisil, 100 mg/50 mg)

**FLOW RATE:** 0.05 to 0.2 L/min or less

**VOL-MIN:** 1 L @ 0.5 mg/m$^3$
**-MAX:** 50 L

**SHIPMENT:** transfer filters to glass vials after sampling

**SAMPLE STABILITY:** unknown for filters; 2 months for Florisil tubes [1]

**BLANKS:** 2 to 10 field blanks per set

## ACCURACY

**RANGE STUDIED:** not studied

**BIAS:** none identified

**OVERALL PRECISION ($\hat{S}_{rT}$):** not evaluated

**ACCURACY:** not determined

## MEASUREMENT

**TECHNIQUE:** GAS CHROMATOGRAPHY, ECD ($^{63}$Ni)

**ANALYTE:** polychlorobiphenyls

**DESORPTION:** filter + front section, 5 mL hexane; back section, 2 mL hexane

**INJECTION VOLUME:** 4-µL with 1-µL backflush

**TEMPERATURE-INJECTION:** 250 to 300 °C
**-DETECTOR:** 300 to 325 °C
**-COLUMN:** 180 °C

**CARRIER GAS:** $N_2$, 40 mL/min

**COLUMN:** glass, 1.8 m x 2-mm ID, 1.5% OV-17/1.95% QF-1 on 80/100 mesh Chromosorb WHP

**CALIBRATION:** standard PCB mixture in hexane

**RANGE:** 0.4 to 4 µg per sample [2]

**ESTIMATED LOD:** 0.03 µg per sample [2]

**PRECISION ($\bar{S}_r$):** 0.044 [1]

**APPLICABILITY:** The working range is 0.01 to 10 mg/m$^3$ for a 40-L air sample [1]. With modifications, surface wipe samples may be analyzed [3,4].

**INTERFERENCES:** Chlorinated pesticides, such as DDT and DDE, may interfere with quantification of PCB. Sulfur-containing compounds in petroleum products also interfere [5].

**OTHER METHODS:** This method revises methods S120 [6] and P&CAM 244 [1]. Methods S121 [7] and P&CAM 253 [8] for PCB have not been revised.

**REAGENTS:**

1. Hexane, pesticide quality.
2. Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
3. Nitrogen, purified.
4. Stock standard solution of the PCB in methanol or isooctane (commercially available).*

* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6-mm OD, 4-mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. (SKC 226-39, Supelco ORBO-60, or equivalent) Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
2. Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
3. Tweezers.
4. Vials, glass, 4- and 7-mL, with aluminum or PTFE-lined caps
5. Gas chromatograph, electron capture detection ($^{63}$Ni), integrator and column (page 5503-1).
6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
7. Syringe, 10-µL.

**SPECIAL PRECAUTIONS:** Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [9-11].

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.

   NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [4]. Under these conditions, the limit of detection was 0.02 µg/m$^3$.
4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

**SAMPLE PREPARATION:**

5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.
6. In a 4-mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane.
7. Allow to stand 20 min with occasional agitation.

## CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 500 ng/mL PCB.
   a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB per sample).
9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
   a. Remove and discard back sorbent section of a media blank Florisil tube.
   b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
   c. Cap the tube. Allow to stand overnight.
   d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. µg PCB recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

## MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
    NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [12].
    NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
12. Sum the areas for five or more selected peaks.

## CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front ($W_f$) and back ($W_b$) sorbent sections, and in the average media blank filter (B) and front ($B_f$) and back ($B_b$) sorbent sections.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b)}{V}, \text{ mg/m}^3.$$

## EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL isooctane [8,13,14]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [1]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 mg/m $^3$ Aroclor 1016 [1].

**REFERENCES:**


[1] NIOSH Manual of Analytical Methods, 2nd ed., V. 1, P&CAM 244, U.S. Department of health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

[2] User check, Southern Research Institute, NIOSH Sequence #4121-U (unpublished, January 25, 1984).

[3] Kominsky, J. Applied Ind. Hyg. 1 (4), R-6 (1986).

[4] NIOSH Health Hazard Evaluation Report, HETA 85-289-1738 (unpublished, 1986).

[5] Hofstader, R. A., C. A. Bache, and D. J. Lisk. Bull, Environ. Contam. Toxicol., 11, 136 (1974).

[6] NIOSH Manual of Analytical Methods, 2nd ed., V. 4, S120, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).

[7] Ibid, V. 2, S121, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

[8] Ibid, Vol. 1, P&CAM 253

[9] Criteria for a Recommended Standard . . . Occupational Exposure to Polychlorinated Biphenyls, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-225 (1977).

[10] Current Intelligence Bulletin 7, Polychlorinated Biphenyls (PCBs), U.S. Department of Health and Human Services, Publ. (NIOSH) 78-127 (1975).

[11] Occupational Diseases, A Guide to Their Recognition, revised ed., 255-256, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-181 (1978).

[12] Dunker, J. C. and M. T. J. Hillebrand. Characterization of PCB Components in Clophen Formulations by Capillary GC-MS and GC-ECD Techniques, Environ. Sci. Technol., 17 (8), 449-456 (1983).

[13] Backup Data Report for S120, prepared under NIOSH Contract 210-76-0123, available as "Ten NIOSH Analytical Methods, Set 2," Order No. Pb 271-464 from NTIS, Springfield, VA 22161.

[14] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

[15] Hutzinger, O., S. Safe, and V. Zitko. The Chemistry of PCBs, CRC Press, Inc., Cleveland, OH (1974).


**METHOD REVISED BY:**

James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

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**Table 1. General Information.**

| Compound | CAS | RTECS |
|---|---|---|
| Polychlorinated Biphenyls | 1336-36-3 | TQ1350000 |
| Chlorobiphenyl | 27323-18-8 | DV2063000 |
| Aroclor 1016 (41% Cl) | 12674-11-2 | TQ1351000 |
| Aroclor 1242 (42% Cl) | 53469-21-9 | TQ1356000 |
| Aroclor 1254 (54% Cl) | 11097-69-1 | TQ1360000 |

**Table 2. Composition of some Aroclors [15].**

| Major Components | Aroclor 1016 | Aroclor 1242 | Aroclor 1254 |
|---|---|---|---|
| Biphenyl | 0.1% | <0.1% | <0.1% |
| Monochlorobiphenyls | 1 | 1 | <0.1 |
| Dichlorobiphenyls | 20 | 16 | 0.5 |
| Trichlorobiphenyls | 57 | 49 | 1 |
| Tetrachlorobiphenyls | 21 | 25 | 21 |
| Pentachlorobiphenyls | 1 | 8 | 48 |
| Hexachlorobiphenyls | <0.1 | 1 | 23 |
| Heptachlorobiphenyls | none detected | <0.1 | 6 |
| Octachlorobiphenyls | none detected | none detected | none detected |

| Table 3 | | | |
|---|---|---|---|
| **Spike Solution Components** | | | |
| **Solution** | **Solvent** | **Compounds** | **Conc. (µg/mL)** |
| Surrogate | Acetone | Decachlorobiphenyl<br>Tetrachloro-m-xylene | 0.2<br>0.2 |
| Pest Full (8081A) | Acetone | alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Aldrin, Heptachlor, Heptachlor Epoxide, Endosulfan I, Endosulfan II, Endosulfan sulfate, alpha-Chlordane, gamma-Chlordane, Dieldrin, 4,4'-DDE, 4,4'-DDD, 4,4'-DDT, Endrin, Endrin ketone, Endrin aldehyde, Methoxychlor | 1ug/mL (for all analytes) |
| Toxaphene | Acetone | Toxaphene | 5 |
| TChlor | Acetone | Technical Chlordane | 1 |
| PCB 1660 (8082) | Acetone | Aroclor 1016<br>Aroclor 1260 | 2.0<br>2.0 |

| Table 4 | | | | | | | |
|---|---|---|---|---|---|---|---|
| **West Sacramento Method 8081/8082 Extraction Summary** | | | | | | | |
| | Sample Amount | Surrogate | LCS1 | Extract Solvent | Extract Solvent Volume | Extraction Time | Comments |
| 1 L separatory funnel extraction | 1 L | 1.0 mL | See table 3 | DCM | 3 x 60 mL | 2 min shake, 10 min rest | |
| 250 mL LVI/RVE | 250 mL | 0.5 mL | See table 3 | | 3 x 30 mL | | |
| 125 mL LVI/RVE | 125 mL | 0.5 mL | See table 3 | | 3 x 15 mL | | |
| Leachate (SEPF) | 200 mL | 1.0 mL | See table 3 | | 3 x 60 mL | | Make the 200mL to 1L with DI water before extracting |
| Solid (Sonication) | 30 g | 2.0 mL | See table 3 | 1:1 DCM /Ace | 3 x 100mL | 3min | |
| Waste Dilution | 1 g | 2.0 mL | See table 3 | Hex | 10mL | NA | Don't concentrate. |
| Biologics (Sonication) | 10 g | 20 mL | See table 3 | 1:1 DCM /Ace | 3 x 100mL | 3 min | |
| Solid (Microwave) | 15g | 1.0mL | See table 3 | 1:1 Hex /Ace | 25mL | 25min | |

# ELEMENTS by ICP
# (Hot Block/HCl/$HNO_3$ Digestion)

**7303**

MW: Table 1 CAS: Table 2 RTECS: Table 2

**METHOD: 7303, Issue 1** **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

**OSHA:** Table 2
**NIOSH:** Table 2
**ACGIH:** Table 2

**PROPERTIES:** Table 1

**ELEMENTS:**

| | | | | | |
|---|---|---|---|---|---|
| aluminum | cadmium | indium | nickel | strontium | zinc |
| antimony* | calcium | iron | palladium | tellurium | |
| arsenic | chromium | lead* | phosphorus | thallium | |
| barium | cobalt | magnesium | platinum | tin* | |
| beryllium | copper | manganese | potassium | titanium | |
| bismuth* | gallium | molybdenum | selenium | vanadium | |
| boron | gold | neodymium | sodium | yttrium | |

* With certain restrictions (see Table 3)

## SAMPLING

**SAMPLER:** FILTER (0.8-µm, cellulose ester membrane)

**FLOW RATE:** 1 to 4 L/min

**VOL-MIN:** Table 1
-MAX: Table 1

**SHIPMENT:** Routine

**SAMPLE STABILITY:** Stable

**BLANKS:** 2 to 10 field blanks per set

## ACCURACY

**RANGE STUDIED:** 5,000 to 50,000 µg/sample

**BIAS:** Not determined

**OVERALL PRECISION:** Not determined

**ACCURACY:** Not determined

## MEASUREMENT

**TECHNIQUE:** INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY

**ANALYTE:** See element list above

**REAGENTS:** Conc. HCl, 1.25 mL; and conc. $HNO_3$, 1.25 mL

**FINAL SOLUTION:** 5% HCl and 5% $HNO_3$, 25 mL

**WAVELENGTH:** Element and instrument specific

**BACKGROUND CORRECTION:** Spectral wavelength shift

**CALIBRATION:** Elements in 5% HCl, 5% $HNO_3$

**RANGE:** LOQ to 50,000 µg/sample [1]

**ESTIMATED LOD:** Varies with element; Table 1

**PRECISION (Š):** Not evaluated

**APPLICABILITY**: The working range of this method is up to 100 mg/m$^3$ for each element in a 500-L sample (the minimum range depends on the LOD for each sample; see Table 1). The analysis is not compound specific. Certain elemental compounds are known to be acceptable or unacceptable by this method (see Table 3). For unverified compounds, a test run should be conducted using a known amount of the compound in question to determine acceptability.

**INTERFERENCES:** Interferences are spectral in nature and are accounted for by choosing appropriate wavelengths, applying interelement correction factors, and background correction.

**OTHER METHODS:** Alternative, more sensitive methods exist for some elements by graphite furnace atomic absorption spectroscopy. This method is similar to NIOSH Method 7301, differing only in the use of the hot block for digestion of the sampler.

**REAGENTS:**

1. Hydrochloric acid,* conc., ultra pure.
2. Nitric acid,* conc., ultra pure.
3. Calibration stock solutions, 50-1000 µg/mL. Commercially available single element solutions or multielement solutions prepared as instructed by the instrument manufacturer.
4. Argon, prepurified.
5. Distilled, deionized, Type II water.
6. Diluting solution: 5% HCl : 5% $HNO_3$. To about 600 mL of deionized water in a 1-L volumetric flask, slowly add 50 mL conc. HCl and 50 mL conc. HNO3. Dilute to the mark with deionized water.

* See SPECIAL PRECAUTIONS

**EQUIPMENT:**

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled argon plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Hot block apparatus at 95 °C.
5. Digestion vessels and caps, 50-mL.
6. Watchglasses.
7. Pipettes, electronic and mechanical.
8. Regulator, two-stage, for argon.
9. Forceps.

**SPECIAL PRECAUTIONS:** Concentrated acids are powerful oxidizers, toxic, and corrosive liquids. Wear protective clothing and work in a fume hood.

**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

**SAMPLE PREPARATION:**

3. Open the cassette filter holder and with forceps remove the sample filter. Fold the filter into quarters taking care not to lose any sample, and transfer to a clean, 50-mL hot block digestion tube.
4. Add 1.25 mL HCl. Cover with a plastic watchglass. Place in the hot block and heat at an internal temperature of 95 °C for 15 minutes.
   NOTE: The internal temperature may vary from the digital readout. Calibrate the hot block prior to digestion.
5. Remove the sample from the hot block and cool for 5 minutes. Remove watchglass and add 1.25 mL $HNO_3$. Replace watchglass and return to hot block at 95 °C for 15 minutes.
6. Remove the sample from the hot block and cool for at least 5 minutes. Rinse watchglass into the sample container and discard watchglass.
7. Dilute to 25-mL final volume with distilled, deionized Type II water.

**CALIBRATION AND QUALITY CONTROL:**

8. Calibrate the spectrometer according to the manufacturer's recommendations. Use standards consisting of the same 5% HCl : 5% $HNO_3$ matrix as the samples.
9. Analyze a standard every 10 samples.
10. Analyze a media blank every 20 samples, and a reagent blank every 10 samples.
11. Analyze a set of two laboratory control samples every 40 samples of a given matrix for a given analyte.
12. Check recoveries with at least two spiked media blanks per ten samples.
    NOTE: In the determination of lead, there may be a measurement interference (for example, samples with high aluminum levels). More recent instruments have a correction for this.

**MEASUREMENT:**

13. Set spectrometer to conditions specified by manufacturer.
14. Analyze standards, samples and quality control checks.
    NOTE: If the elemental value for a sample is above the linear range of the element(s) in question, dilute the sample solution with 5% HCl : 5% $HNO_3$ diluting solution, reanalyze and apply the appropriate dilution factor in the calculations.

**CALCULATIONS:**

15. Obtain the solution concentrations for the sample, $C_s$ (µg/mL), and the average media blank, $C_b$ (µg/mL), from the instrument.
16. Using the solution volumes of sample, $V_s$ (mL), and media blank, $V_b$ (mL), calculate the concentration, C ($mg/m^3$), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, mg/m^3$$

NOTE: µg/L ≡ $mg/m^3$

**EVALUATION OF METHOD:**

The method was evaluated for all elements and compounds listed in Table 1 and Table 2 between 1999 and 2001 using known amounts of bulk material [4]. Evaluation is ongoing for additional elements and compounds. The limits of detection and quantitation were also determined for each element. Two ICP instruments were used in the evaluation, a Thermal Jarrell Ash Model 61E [5] and a TJA IRIS [6], operated according to the manufacturer's instructions.

**REFERENCES:**

[1] WOHL [2001]. Metals validation using hot block digestion, Unpublished data. Wisconsin Occupational Health Laboratory, Madison, WI.
[2] NIOSH [1994]. Method 7300: Elements by ICP, NIOSH Manual of Analytical Methods, Fourth Edition, Issue 2, Aug. 15, 1994.
[3] WOHL [2001]. Metals Manual 2001, WOHL Internal Document, Updated Apr. 1, 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
[4] WOHL [2001]. WOHL General Operations Procedures Manual, WOHL Internal Document, Updated 2001. Wisconsin Occupational Health Laboratory, Madison, WI.
[5] Thermal Jarrell Ash [1991]. ICAP 61E Plasma Spectrometer Operator's Manual, Thermal Jarrell Ash Corp., Part No. 128832-01, Feb., 1991.
[6] Thermal Jarrell Ash [1997]. IRIS Plasma Spectrometer User's Guide, Thermal Jarrell Ash Corp., Part No. 135811-0, Feb. 4, 1997.

**METHOD WRITTEN BY:**

Jason Loughrin, Lyle Reichmann, Doug Smieja, Shakker Amer, Curtis Hedman
Wisconsin Occupational Health Laboratory (WOHL).

**TABLE 1: ANALYTE INFORMATION FOR VALID ELEMENTS AND COMPOUNDS**

| Analyte | Properties MW | Properties MP (°C) | LOD (µg/mL) | LOQ (µg/mL) | Estimated LOQ (µg/sample)* | Minimum** air vol. (L) | Maximum*** air vol. (L) |
|---|---|---|---|---|---|---|---|
| Al | 26.98 | 660 | 0.111 | 0.37 | 9.25 | 2 | 10,000 |
| As | 74.92 | 817 | 0.009 | 0.03 | 0.075 | 8 | 5,000,000 |
| Au | 196.97 | 10.63 | 0.015 | 0.05 | 1.25 | 1 | 3,300 |
| B | 10.81 | 2177 | 0.0094 | 0.0283 | 0.71 | 1 | 3,300 |
| Ba | 137.34 | 3.51 | 0.0018 | 0.006 | 0.15 | 1 | 100,000 |
| Be | 9.01 | 2178 | 0.00075 | 0.0025 | 0.062 | 35 | 25,000,00 |
| Bi | 208.98 | 271 | 0.025 | 0.085 | 2.12 | 1 | 10,000 |
| Ca | 40.08 | 842 | 0.099 | 0.33 | 8.25 | 2 | 10,000 |
| CaO | 56.08 | 2927 | 0.139 | 0.462 | 11.6 | 3 | 10,000 |
| Cd | 112.4 | 321 | 0.0037 | 0.012 | 0.30 | 3 | 500,000 |
| Co | 58.93 | 1495 | 0.003 | 0.011 | 0.27 | 3 | 500,000 |
| Cr | 52.00 | 1890 | 0.009 | 0.03 | 0.75 | 8 | 500,000 |
| Cu | 63.54 | 1083 | 0.020 | 0.060 | 1.50 | 15 | 500,000 |
| Fe | 55.85 | 1535 | 0.070 | 0.20 | 5.00 | 1 | 5,000 |
| $Fe_2O_3$ (as Fe) | 159.69 | 1462 | 0.070 | 0.20 | 5.00 | 1 | 5,000 |
| Ga | 69.72 | 29.75 | 0.03 | 0.09 | 2.25 | 1 | 3,300 |
| In | 114.82 | 156.3 | 0.015 | 0.05 | 1.25 | 15 | 500,000 |
| Mg | 24.31 | 651 | 0.047 | 0.14 | 3.50 | 1 | 10,000 |
| MgO | 40.32 | 2825 | 0.078 | 0.23 | 5.75 | 5 | 33,000 |
| Mn | 54.94 | 1244 | 0.0012 | 0.004 | 0.10 | 0.05 | 10,000 |
| Mo | 95.94 | 651 | 0.0072 | 0.024 | 0.60 | 0.5 | 10,000 |
| Nd | 92.906 | 2477 | 0.01 | 0.03 | 0.75 | 0.1 | 3,300 |
| Ni | 58.71 | 1453 | 0.012 | 0.039 | 0.98 | 1 | 50,000 |
| P | 30.97 | 44 | 0.3 | 1.0 | 25 | 250 | 500,000 |
| Pb | 207.19 | 328 | 0.023 | 0.07 | 1.75 | 35 | 100,000 |
| Pd | 106.4 | 1550 | 0.009 | 0.03 | 0.75 | 0.1 | 3,300 |
| Pt | 195.09 | 1769 | 0.0045 | 0.015 | 0.38 | 200 | 25,000,000 |
| Sb | 121.75 | 630.5 | 0.018 | 0.06 | 1.50 | 3 | 100,000 |
| Se | 78.96 | 217 | 0.021 | 0.064 | 1.60 | 8 | 250,000 |
| Sn | 118.69 | 232 | 0.015 | 0.05 | 1.25 | 1 | 25,000 |
| Sr | 87.62 | 769 | 0.002 | 0.006 | 0.15 | 300 | 100,000,000 |
| Te | 127.60 | 450 | 0.15 | 0.5 | 12.5 | 125 | 500,000 |
| Ti | 47.90 | 1675 | 0.005 | 0.016 | 0.40 | 0.1 | 10,000 |
| Tl | 204.37 | 304 | 0.044 | 0.133 | 3.32 | 35 | 500,000 |
| V | 50.94 | 1890 | 0.003 | 0.01 | 0.25 | 2.5 | 500,000 |
| Y | 88.91 | 1495 | 0.001 | 0.003 | 0.075 | 0.1 | 50,000 |
| Zn | 65.37 | 419 | 0.022 | 0.066 | 1.65 | 0.5 | 10,000 |
| ZnO | 81.37 | 1970 | 0.027 | 0.082 | 2.05 | 0.5 | 10,000 |

* Value based on a 25-mL sample volume.

** The minimum sampling volume needed to obtain the OSHA PEL at the LOQ for the element/compound at a sample digestion volume of 25 mL.

*** The maximum sampling volume for a given sample, calculated by taking 50,000 µg as the limit for the element/compound per sample.

NOTE: The LOD and LOQ values are dependent on the particular analytical instrument used. Also, LOD and LOQ values may vary for a particular element due to certain interelement interferences.

**TABLE 2. EXPOSURE LIMITS, CAS #, RTECS**

| Element (Symbol) | CAS # | RTECS | Exposure Limits, $mg/m^3$ (Ca = carcinogen) OSHA | NIOSH | ACGIH |
|---|---|---|---|---|---|
| Silver (Ag) | 7440-22-4 | VW3500000 | 0.01 (dust, fume, metal) | 0.01 (metal, soluble) | 0.1 (metal)<br>0.01 (soluble) |
| Aluminum (Al) | 7429-90-5 | BD0330000 | 15 (total dust)<br>5 (respirable) | 10 (total dust)<br>5 (respirable fume)<br>2 (salts, alkyls) | 10 (dust)<br>5 (powders, fume)<br>2 (salts, alkyls) |
| Arsenic (As) | 7440-38-2 | CG0525000 | varies | C 0.002, Ca | 0.01, Ca |
| Barium (Ba) | 7440-39-3 | CQ8370000 | 0.5 | 0.5 | 0.5 |
| Beryllium (Be) | 7440-41-7 | DS1750000 | 0.002, C 0.005 | 0.0005, Ca | 0.002, Ca |
| Calcium (Ca) | 7440-70-2 | -- | varies | varies | varies |
| Cadmium (Cd) | 7440-43-9 | EU9800000 | 0.005 | lowest feasible, Ca | 0.01 (total), Ca<br>0.002 (respir.), Ca |
| Cobalt (Co) | 7440-48-4 | GF8750000 | 0.1 | 0.05 (dust, fume) | 0.02 (dust, fume) |
| Chromium (Cr) | 7440-47-3 | GB4200000 | 0.5 | 0.5 | 0.5 |
| Copper (Cu) | 7440-50-8 | GL5325000 | 1 (dust, mists)<br>0.1 (fume) | 1 (dust)<br>0.1 (fume) | 1 (dust, mists)<br>0.2 (fume) |
| Iron (Fe) | 7439-89-6 | NO4565500 | 10 (dust, fume) | 5 (dust, fume) | 5 (fume) |
| Potassium (K) | 7440-09-7 | TS6460000 | -- | -- | -- |
| Lanthanum | 7439-91-0 | -- | – | – | -- |
| Lithium (Li) | 7439-93-2 | -- | -- | -- | -- |
| Magnesium (Mg) | 7439-95-4 | OM2100000 | 15 (dust) as oxide<br>5 (respirable) | 10 (fume) as oxide | 10 (fume) as oxide |
| Manganese (Mn) | 7439-96-5 | OO9275000 | C 5 | 1; STEL 3 | 5 (dust)<br>1; STEL 3 (fume) |
| Molybdenum (Mo) | 7439-98-7 | QA4680000 | 5 (soluble)<br>15 (total insoluble) | 5 (soluble)<br>10 (insoluble) | 5 (soluble)<br>10 (insoluble) |
| Nickel (Ni) | 7440-02-0 | QR5950000 | 1 | 0.015, Ca | 0.1 (soluble)<br>1 (insoluble, metal) |
| Phosphorus (P) | 7723-14-0 | TH3500000 | 0.1 | 0.1 | 0.1 |
| Lead (Pb) | 7439-92-1 | OF7525000 | 0.05 | 0.05 | 0.05 |
| Antimony (Sb) | 7440-36-0 | CC4025000 | 0.5 | 0.5 | 0.5 |
| Selenium (Se) | 7782-49-2 | VS7700000 | 0.2 | 0.2 | 0.2 |
| Tin (Sn) | 7440-31-5 | XP7320000 | 2 | 2 | 2 |
| Strontium (Sr) | 7440-24-6 | – | – | – | -- |
| Tellurium (Te) | 13494-80-9 | WY2625000 | 0.1 | 0.1 | 0.1 |
| Titanium (Ti) | 7440-32-6 | XR1700000 | -- | -- | -- |
| Thallium (Tl) | 7440-28-0 | XG3425000 | 0.1 (skin) (soluble) | 0.1 (skin) (soluble) | 0.1 (skin) |
| Vanadium (V) | 7440-62-2 | YW240000 | -- | C 0.05 | -- |
| Tungsten | 7440-33-7 | – | 5 | 5<br>10 (STEL) | 5<br>10 (STEL) |
| Yttrium (Y) | 7440-65-5 | ZG2980000 | 1 | N/A | 1 |
| Zinc (Zn) | 7440-66-6 | ZG8600000 | – | -- | -- |
| Zirconium (Zr) | 7440-67-7 | ZH7070000 | 5 | 5, STEL 10 | 5, STEL 10 |

**TABLE 3: VALIDATION SUMMARY**

| Analyte | Status[1] | Analyte | Status | Analyte | Status |
|---|---|---|---|---|---|
| Ag | Not Valid | CuO | Valid | S | Not Valid |
| Al | Valid | Fe | Valid | Sb | Partially Valid[4] |
| $Al_2O_3$ | Not Valid | $Fe_2O_3$ | Valid | $Sb_2O_3$ | Partially Valid[5] |
| As | Valid | Ga | Valid | Se | Valid |
| Au | Valid | In | Valid | Si | Not Valid |
| B | Valid | KCl | Pending | Sn | Partially Valid[6] |
| Ba | Pending | Mg | Valid | SnO | Pending |
| BaO | Pending | MgO | Valid | $SnO_2$ | Pending |
| $BaO_2$ | Pending | Mn | Valid | Sr | Valid |
| $BaCl_2$ | Valid | MnO | Valid | $SrCrO_4$ | Valid (by Cr) |
| $BaSO_4$ | Pending | Mo | Valid | Te | Valid |
| Be | Valid | NaCl | Pending | Ti | Valid |
| Bi | Partially Valid[2] | Nd | Valid | Tl | Valid |
| Ca | Valid | Ni | Valid | V | Valid |
| $CaCO_3$ | Valid | P | Valid | $V_2O_5$ | Valid |
| CaO | Valid | Pb | Partially Valid[3] | Y | Valid |
| Cd | Valid | $PbCrO_4$ | Valid (by Cr) | Zn | Valid |
| Co | Valid | PbO | Valid | ZnO | Valid |
| Cr | Valid | Pd | Valid | Zr | Not Valid |
| Cu | Valid | Pt | Valid | ZrO | Not Valid |

[1] Status definitions

Valid: The method is suitable for samples up to at least 0.0500 g bulk material with recoveries of between 90 and 110 percent. This weight exceeds most expected levels encountered in work environments.

Partially Valid: The method is suitable with bulk-material recoveries of between 90 and 110 percent under certain conditions (as footnoted above).

Not Valid: The method procedure is not suitable for samples at any weight with recoveries of between 90 and 110 percent. An alternative method should be used.

[2] Valid up to 10,000 µg/sample and within 7 days of sample digestion.
[3] Valid up to 50,000 µg/sample and at least 24 hours after sample digestion; Valid up to 15,000 µg/sample within 24 hours of sample digestion.
[4] Valid up to 25,000 µg/sample and within 7 days of sample digestion.
[5] Valid up to 25,000 µg/sample and within 7 days of sample digestion.
[6] Valid up to 30,000 µg/sample and within 7 days of sample digestion.

NOTE: The upper limits of the method can be extended by serial dilution of the samples at the time of analyses.

# Attachment F

# Equipment Performance Information

# Performance Characteristic Sheet

**EFFECTIVE DATE:** **September 24, 2004** **EDITION NO.: 1**

**MANUFACTURER AND MODEL:**

Make: *Niton LLC*

Tested Model: *XLp 300*

Source: $^{109}$Cd

Note: This PCS is also applicable to the equivalent model variations indicated below, for the Lead-in-Paint K+L variable reading time mode, in the XLi and XLp series:

XLi 300A, XLi 301A, XLi 302A and XLi 303A.
XLp 300A, XLp 301A, XLp 302A and XLp 303A.
XLi 700A, XLi 701A, XLi 702A and XLi 703A.
XLp 700A, XLp 701A, XLp 702A, and XLp 703A.

Note: The XLi and XLp versions refer to the shape of the handle part of the instrument. The differences in the model numbers reflect other modes available, in addition to Lead-in-Paint modes. The manufacturer states that specifications for these instruments are identical for the source, detector, and detector electronics relative to the Lead-in-Paint mode.

## FIELD OPERATION GUIDANCE

**OPERATING PARAMETERS**:

Lead-in-Paint K+L variable reading time mode.

**XRF CALIBRATION CHECK LIMITS**:

| |
|---|
| 0.8 to 1.2 mg/cm$^2$ (inclusive) |

The calibration of the XRF instrument should be checked using the paint film nearest 1.0 mg/cm$^2$ in the NIST Standard Reference Material (SRM) used (e.g., for NIST SRM 2579, use the 1.02 mg/cm$^2$ film).

If readings are outside the acceptable calibration check range, follow the manufacturer's instructions to bring the instruments into control before XRF testing proceeds.

**SUBSTRATE CORRECTION:**

For XRF results using Lead-in-Paint K+L variable reading time mode, substrate correction is <u>not</u> needed for:

Brick, Concrete, Drywall, Metal, Plaster, and Wood

**INCONCLUSIVE RANGE OR THRESHOLD:**

| K+L MODE READING DESCRIPTION | SUBSTRATE | THRESHOLD (mg/cm$^2$) |
|---|---|---|
| Results not corrected for substrate bias on any substrate | Brick | 1.0 |
| | Concrete | 1.0 |
| | Drywall | 1.0 |
| | Metal | 1.0 |
| | Plaster | 1.0 |
| | Wood | 1.0 |

## BACKGROUND INFORMATION

### EVALUATION DATA SOURCE AND DATE:

This sheet is supplemental information to be used in conjunction with Chapter 7 of the HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing* ("HUD Guidelines"). Performance parameters shown on this sheet are calculated from the EPA/HUD evaluation using archived building components. Testing was conducted in August 2004 on 133 testing combinations. The instruments that were used to perform the testing had new sources; one instrument's was installed in November 2003 with 40 mCi initial strength, and the other's was installed June 2004 with 40 mCi initial strength.

### OPERATING PARAMETERS:

Performance parameters shown in this sheet are applicable only when properly operating the instrument using the manufacturer's instructions and procedures described in Chapter 7 of the HUD Guidelines.

### SUBSTRATE CORRECTION VALUE COMPUTATION:

Substrate correction is not needed for brick, concrete, drywall, metal, plaster or wood when using Lead-in-Paint K+L variable reading time mode, the normal operating mode for these instruments. If substrate correction is desired, refer to Chapter 7 of the HUD Guidelines for guidance on correcting XRF results for substrate bias.

### EVALUATING THE QUALITY OF XRF TESTING:

Randomly select ten testing combinations for retesting from each house or from two randomly selected units in multifamily housing. Use the K+L variable time mode readings.

Conduct XRF retesting at the ten testing combinations selected for retesting.

Determine if the XRF testing in the units or house passed or failed the test by applying the steps below.

- Compute the Retest Tolerance Limit by the following steps:
  - Determine XRF results for the original and retest XRF readings. Do not correct the original or retest results for substrate bias. In single-family housing a result is defined as the average of three readings. In multifamily housing, a result is a single reading. Therefore, there will be ten original and ten retest XRF results for each house or for the two selected units.
    - Calculate the average of the original XRF result and retest XRF result for each testing combination.
    - Square the average for each testing combination.
    - Add the ten squared averages together. Call this quantity C.
    - Multiply the number C by 0.0072. Call this quantity D.
    - Add the number 0.032 to D. Call this quantity E.
    - Take the square root of E. Call this quantity F.
- Multiply F by 1.645. The result is the Retest Tolerance Limit.
- Compute the average of all ten original XRF results.
- Compute the average of all ten re-test XRF results.
- Find the absolute difference of the two averages.

If the difference is less than the Retest Tolerance Limit, the inspection has passed the retest. If the difference of the overall averages equals or exceeds the Retest Tolerance Limit, this procedure should be repeated with ten new testing combinations. If the difference of the overall averages is equal to or greater than the Retest Tolerance Limit a second time, then the inspection should be considered deficient.

Use of this procedure is estimated to produce a spurious result approximately 1% of the time. That is, results of this procedure will call for further examination when no examination is warranted in approximately 1 out of 100 dwelling units tested.

**TESTING TIMES:**

For the Lead-in-Paint K+L variable reading time mode, the instrument continues to read until it is moved away from the testing surface, terminated by the user, or the instrument software indicates the reading is complete. The following table provides testing time information for this testing mode. The times have been adjusted for source decay, normalized to the initial source strengths as noted above. Source strength and type of substrate will affect actual testing times. At the time of testing, the instruments had source strengths of 26.6 and 36.6 mCi.

| **Testing Times Using K+L Reading Mode (Seconds)** | | | | | | |
|---|---|---|---|---|---|---|
| | All Data | | | Median for laboratory-measured lead levels ($mg/cm^2$) | | |
| Substrate | 25th Percentile | Median | 75th Percentile | Pb < 0.25 | 0.25 ≤ Pb<1.0 | 1.0 ≤ Pb |
| Wood Drywall | 4 | 11 | 19 | 11 | 15 | 11 |
| Metal | 4 | 12 | 18 | 9 | 12 | 14 |
| Brick Concrete Plaster | 8 | 16 | 22 | 15 | 18 | 16 |

**CLASSIFICATION RESULTS:**

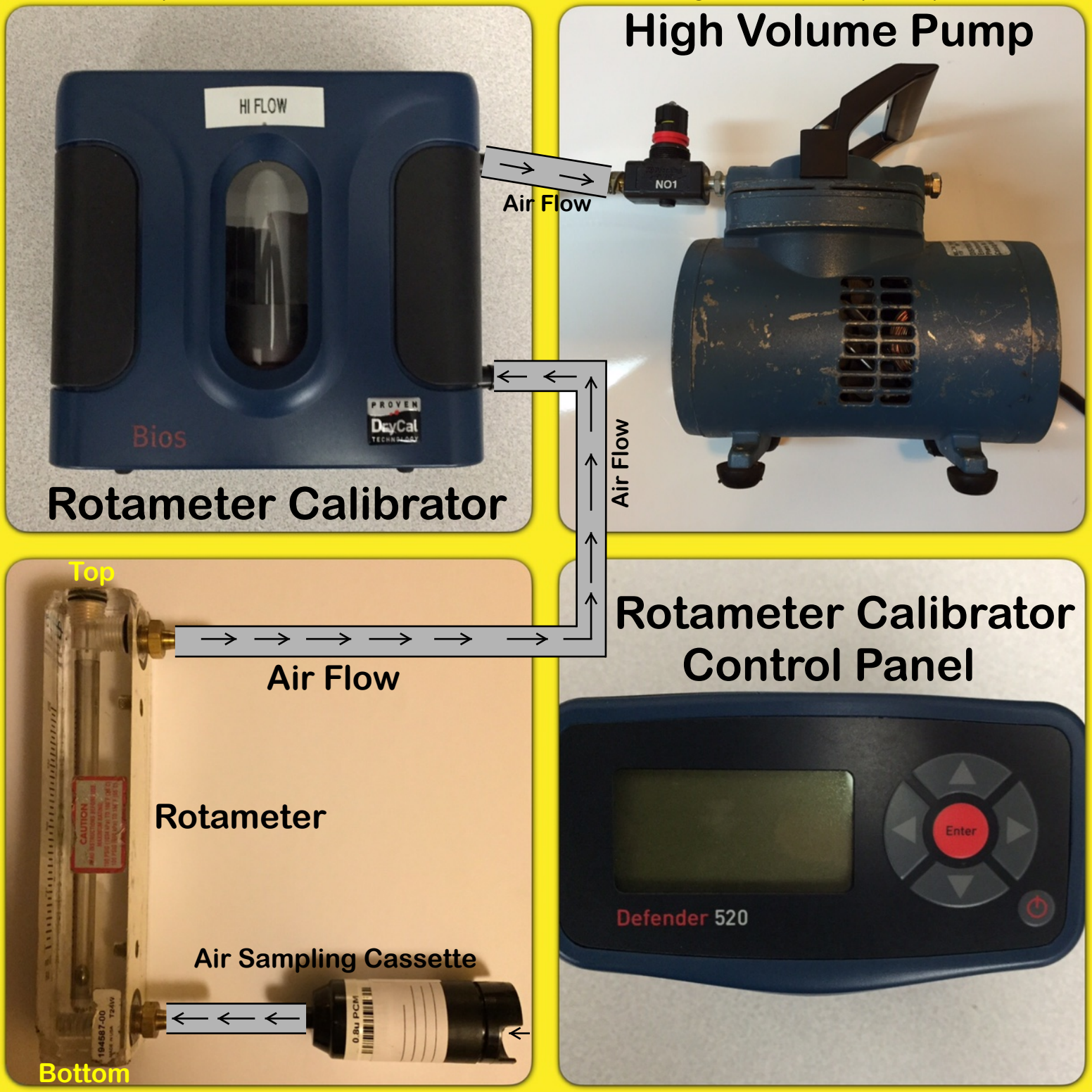
XRF results are classified as positive if they are greater than or equal to the threshold, and negative if they are less than the threshold.

**DOCUMENTATION:**

A document titled *Methodology for XRF Performance Characteristic Sheets* provides an explanation of the statistical methodology used to construct the data in the sheets, and provides empirical results from using the recommended inconclusive ranges or thresholds for specific XRF instruments. For a copy of this document call the National Lead Information Center Clearinghouse at 1-800-424-LEAD.

This XRF Performance Characteristic Sheet was developed by the Midwest Research Institute (MRI) and QuanTech, Inc., under a contract between MRI and the XRF manufacturer. HUD has determined that the information provided here is acceptable when used as guidance in conjunction with Chapter 7, Lead-Based Paint Inspection, of HUD's *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*.

# How to set up the Rotameter to the Rotameter Calibrator & High Volume Pump with plastic tube



# Rotameter Calibration

**ACC Environmental Consultants**
7977 Capwell Drive
Oakland, CA 94621

| | |
|---|---|
| Rotameter ID: | |
| Calibrated By: | |
| Date Calibrated: | |

| Actual Calibration Data | |
|---|---|
| Rotameter Reading (LPM) | Actual Flow (LPM) |
| 3.00 | 3.23 |
| 5.00 | 5.03 |
| 8.00 | 8.25 |
| 10.00 | 10.33 |
| 15.00 | 15.88 |

| slope | y- intercept |
|---|---|
| 1.060322581 | -0.150645161 |

| min rotameter value |
|---|
| 3 |

| max rotameter value |
|---|
| 15 |

| | |
|---|---|
| Temperature: | 76.5 |
| Relative Humidity: | 52.60% |
| Barometric Pressure | 29.95 |

| Regression of Data | |
|---|---|
| Rotameter Reading (LPM) | Actual Flow (LPM) |
| 1.00 | |
| 1.50 | |
| 2.00 | |
| 2.50 | |
| 3.00 | 3.03 |
| 3.50 | 3.56 |
| 4.00 | 4.09 |
| 4.50 | 4.62 |
| 5.00 | 5.15 |
| 5.50 | 5.68 |
| 6.00 | 6.21 |
| 6.50 | 6.74 |
| 7.00 | 7.27 |
| 7.50 | 7.80 |
| 8.00 | 8.33 |
| 8.50 | 8.86 |
| 9.00 | 9.39 |
| 9.50 | 9.92 |
| 10.00 | 10.45 |
| 10.50 | 10.98 |
| 11.00 | 11.51 |
| 11.50 | 12.04 |
| 12.00 | 12.57 |
| 12.50 | 13.10 |
| 13.00 | 13.63 |
| 13.50 | 14.16 |
| 14.00 | 14.69 |
| 14.50 | 15.22 |
| 15.00 | 15.75 |
| 15.50 | 16.28 |
| 16.00 | 16.81 |
| 16.50 | 17.34 |
| 17.00 | 17.87 |
| 17.50 | 18.41 |
| 18.00 | 18.94 |
| 18.50 | 19.47 |
| 19.00 | 20.00 |
| 19.50 | 20.53 |
| 20.00 | 21.06 |

**ACC Environmental Consultants**
7977 Capwell Drive
Oakland, CA 94621

**Rotameter No:**
**Calibrated By:**
**Date Calibrated:**

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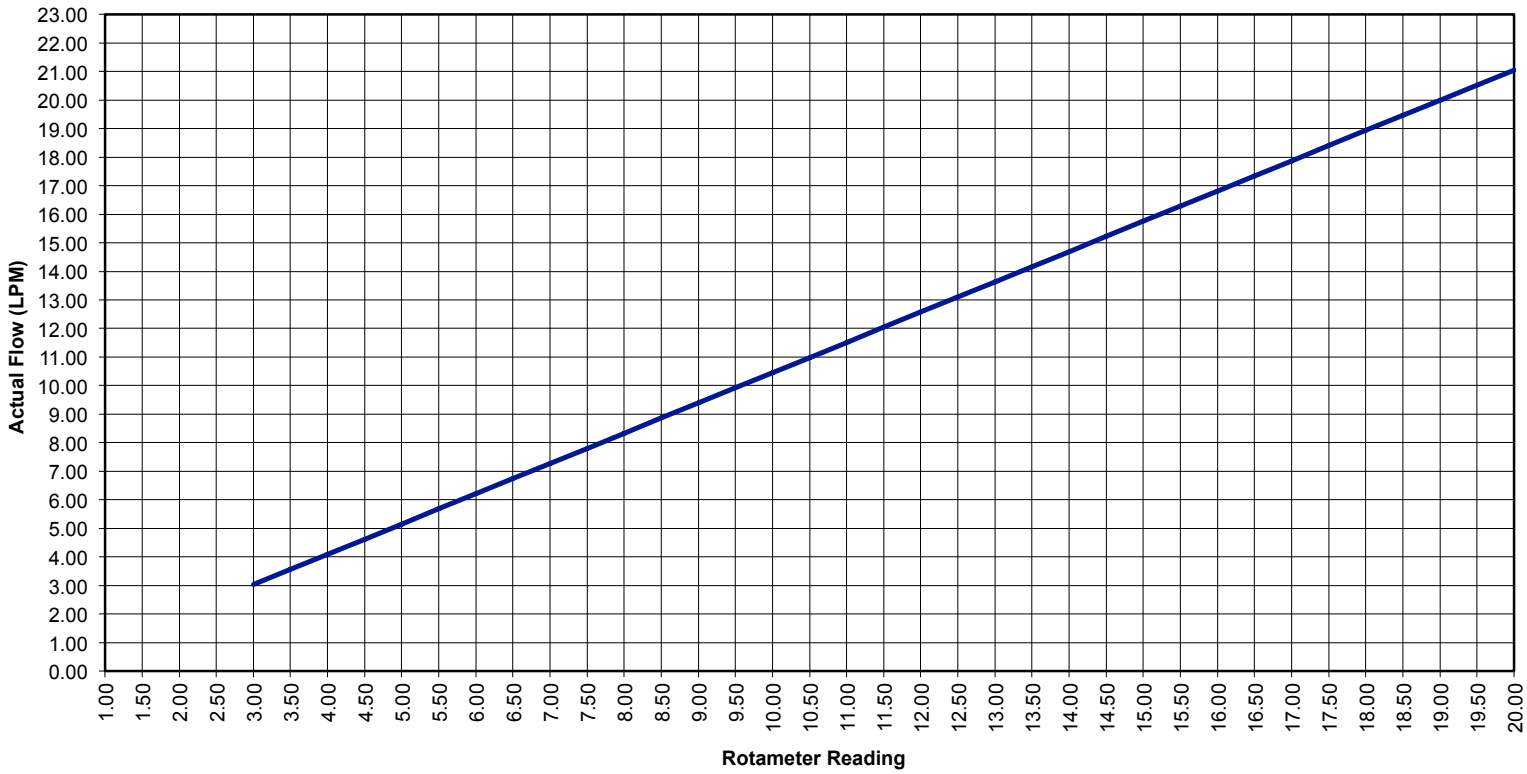
**Linear Regression of Rotameter Calibration**