Appendix A
Health and Safety Plan

Lower Duwamish Waterway Group

Port of Seattle / City of Seattle / King County / The Boeing Company

FISH, CRAB, CLAM, AND SURFACE WATER PERIODIC MONITORING QUALITY ASSURANCE PROJECT PLAN FOR THE LOWER DUWAMISH WATERWAY APPENDIX A: HEALTH AND SAFETY PLAN

Prepared for

Lower Duwamish Waterway Group

For submittal to

US Environmental Protection Agency

Prepared by:

ANCHOR QEA

1201 3rd Avenue • Suite 2600
Seattle, Washington • 98101

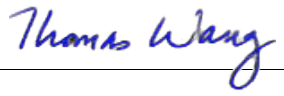
in association with

Windward environmental LLC

200 First Avenue West • Suite 500
Seattle, Washington • 98119

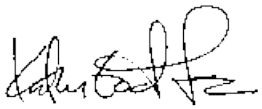
Title and Approval Page: Periodic Monitoring Health and Safety Plan

By their signature, the undersigned certify that this health and safety plan is approved and that it will be used to govern health and safety aspects of fieldwork described in the quality assurance project plan to which it is attached.



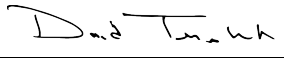
April 12, 2023
Date

Tom Wang
Anchor QEA Project Manager



April 12, 2023
Date

Kathy Godtfredsen
Windward Project Manager

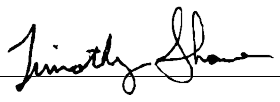


April 12, 2023
Date

David Templeton
Corporate Health and Safety Manager

April 12, 2023
Date

Thai Do
Windward Field Coordinator/Health and Safety Officer



April 12, 2023
Date

Tim Shaner
Anchor QEA Field Coordinator/Health and Safety Officer

HEALTH AND SAFETY PLAN ACKNOWLEDGEMENT FORM

Project Number: 210007-01.01

Project Name: Periodic Monitoring

My signature below certifies that I have read and understand the policies and procedures specified in this Health and Safety Plan (HSP). For non-Anchor QEA and Windward employees, this HSP may include company-specific appendices to this plan developed by entities other than Anchor QEA and Windward. A copy of this HSP must be always maintained, kept on-site, and available for employee review.

Date	Name (print)	Signature	Company

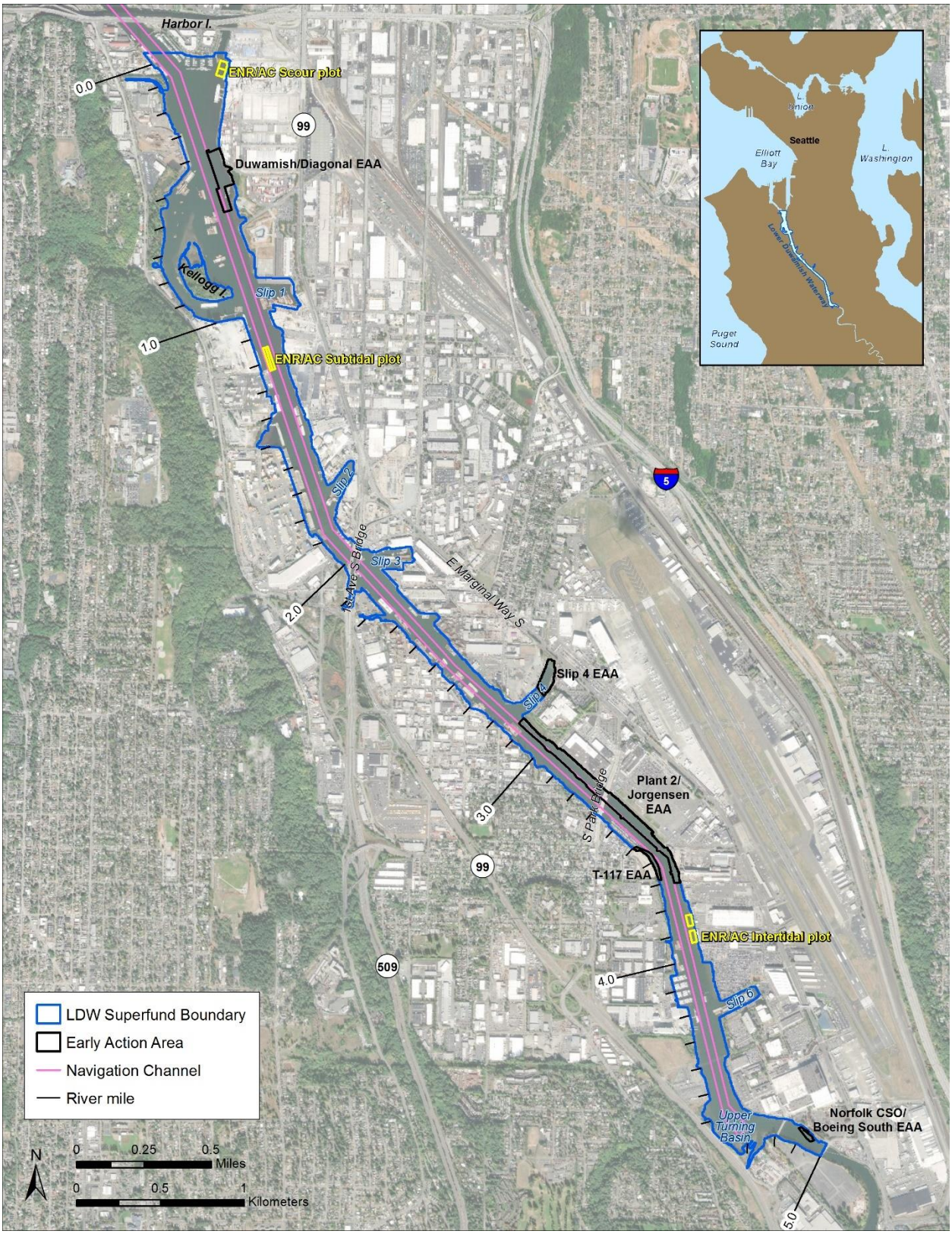


Figure A-i. General site location overview

EMERGENCY CONTACT INFORMATION

Table A-i. Site emergency form and emergency phone numbers

Category	Information	
Possible Chemicals of Concern	Metals, PCBs, PAHs, dioxin/furans, hydrogen sulfide	
Minimum Level of Protection	Modified Level D PPE	
Site(s) Location Address	Lower Duwamish Waterway, Seattle, Washington	
Emergency Phone Numbers		
Ambulance	911	
Fire	911	
Police	911	
Poison Control	(800) 222-1222	
PM	Tom Wang	Office: 206.903.3314 Cell: 206.465.0900
Windward FC/Health and Safety Coordinator	Thai Do	Office: 206.812.5407 Cell: to be provided
Anchor QEA FC/ Health and Safety Coordinator	Matt Woltman	Office: 206.903.3327 Cell: to be provided
Anchor QEA CHSM	David Templeton	Office: 206.287.9130 Cell: 206.910.4279
Anchor QEA Health and Safety Program Lead	Tim Shaner	Office: 251.375.5282 Cell: 251.281.3386
USCG	Emergency: 206.286.5400 General Information: 206.442.5295 VHF Channel 16	
Ecology NW Region Spill Response (24-hr emergency line)	206.649.7000	

In the event of any emergency, contact the PM and FC.

For local resources, please visit: http://www2.epa.gov/emergency-response/emergency-response-my-community.
The National Response Center hotline is 1-800-424-8802.

Anchor QEA – Anchor QEA, LLC
CHSM – corporate health and safety manager
Ecology – Washington State Department of Ecology
FC – field coordinator
PCB – polychlorinated biphenyls
PAH – polycyclic aromatic hydrocarbons
PM – project manager
PPE – personal protective equipment
USCG – US Coast Guard
Windward – Windward Environmental LLC

Table A-ii. Hospital information

Category	Information
Hospital Name	Harborview Medical Center
Address	325 9th Avenue
City, Province	Seattle, Washington 98104
Phone	206.323.3074
Emergency Phone	911

HOSPITAL ROUTE MAP AND DRIVING DIRECTIONS

The name, address, and telephone number of the hospital that will be used to provide medical care is as follows:

Harborview Medical Center
325 - 9th Avenue
Seattle, WA
206.323.3074

Directions from the vicinity of the Lower Duwamish Waterway (LDW) to Harborview Medical Center are as follows (Figure A-ii):

From the Duwamish River boat ramp (at South River Street, beneath the 1st Avenue South bridge):

- Drive east on South River Street.
- Turn left on Occidental Avenue South.
- Turn left on East Marginal Way South.
- Turn right on South Michigan Street.
- Look for entrance ramps to I-5 North.
- Drive north on I-5.
- Take the James Street exit.
- Drive east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

From the Harbor Island Marina (1001 Southwest Klickitat Way):

- From marina parking lot, turn sharp right onto Klickitat Way Southwest.
- Turn slight right onto Southwest Spokane Street
- Turn slight left to take the ramp toward WA-99 N/I-5/Columbian Way.
- Keep left at the fork in the ramp.
- Stay straight to go onto West Seattle Bridge.
- Merge onto I-5 North via the ramp on the left.
- Take the James Street exit.
- Head east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

From South Park Marina (8604 Dallas Avenue South):

- From marina parking lot, turn right onto Dallas Avenue South.
- Turn right onto 16th Avenue South.
- Turn left on East Marginal Way South.
- Look for entrance ramps to I-5 North.
- Drive north on I-5.
- Take the James Street exit.
- Drive east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

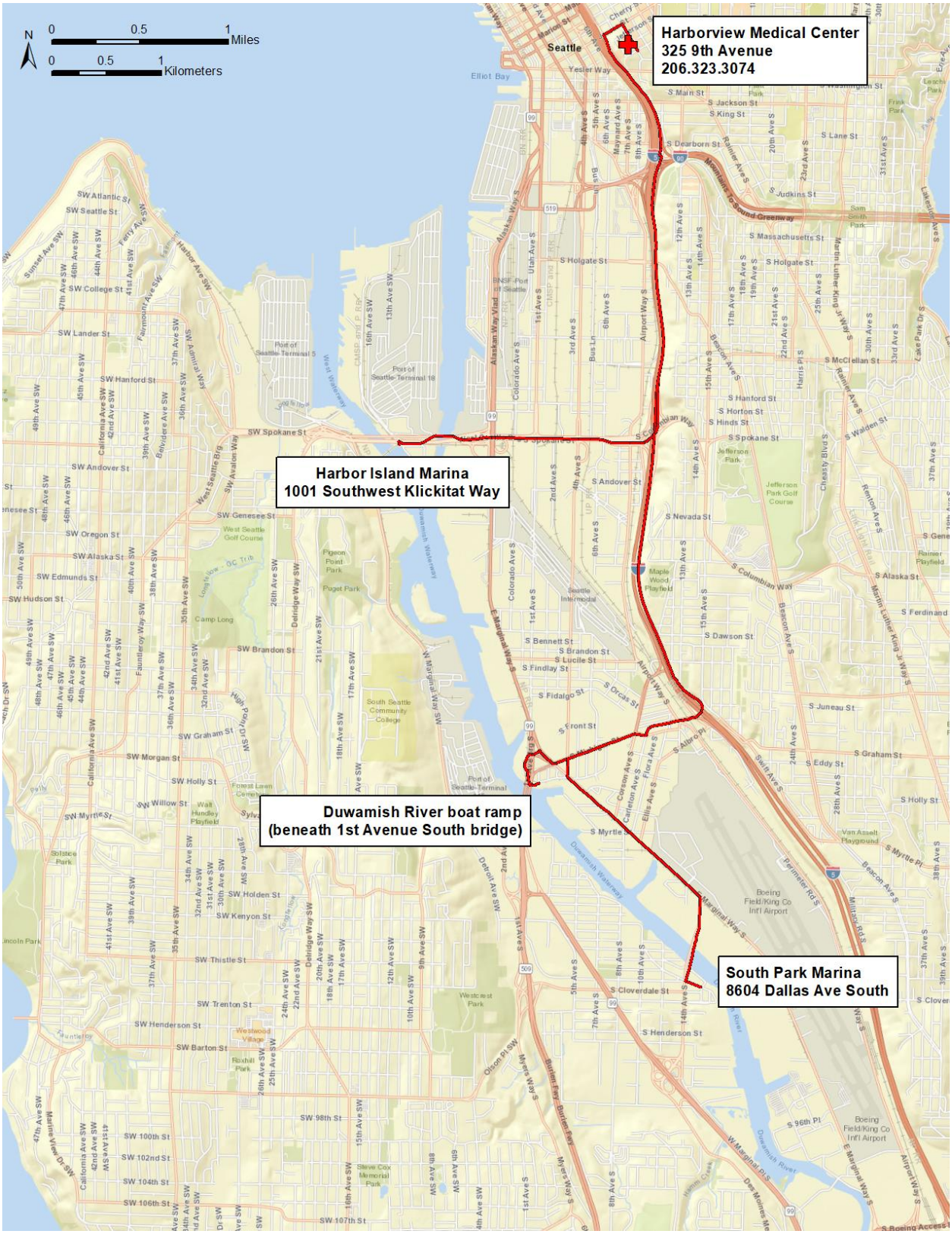


Figure A-ii. Hospital route map

PERSONAL INCIDENT RESPONSE PROCEDURES

In the event of an emergency, immediate action must be taken by the first person to recognize the event. Use the following steps as a guideline and refer to Figure A-iii:

- Survey the situation to verify that it is safe for you and the victim. Do not endanger your own life. Do not enter an area to rescue someone who has been overcome unless properly equipped and trained. Verify that all protocols are followed.
- Call the appropriate emergency number (911, if available) or direct someone else to do this immediately (see Table A-i). Explain the physical injury, chemical exposure, fire, or release and location of the incident.
- Have someone retrieve the nearest first aid kit (containing appropriate items for the particular work scope) and automated external defibrillator, if available. Note: Only use an automated external defibrillator if you have been properly trained and are currently certified to do so.
- Decontaminate the victim without delaying life-saving procedures.
- Administer first aid and cardiopulmonary resuscitation (CPR), if properly trained, until emergency responders arrive.
- In the event that evacuation is required, the field lead must perform a head count to verify that all personnel are accounted for.
- Notify the field coordinator (FC) and project manager (PM); the PM will notify the client contact. The PM will also contact the corporate health and safety manager (CHSM), who will facilitate the incident investigation. Adhere to all client requirements pertinent to personal incident reporting.
- Complete the appropriate incident investigation reports.

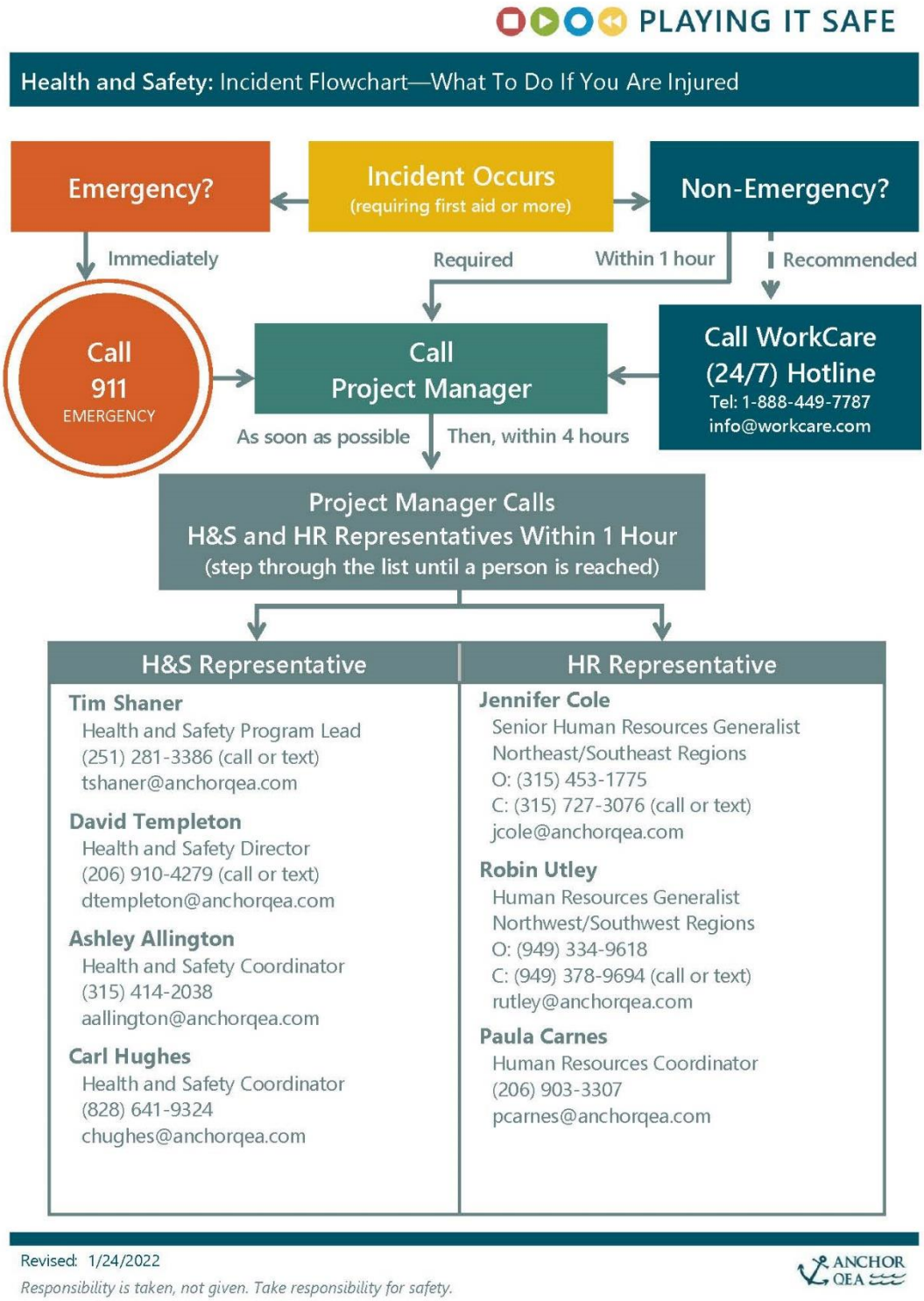


Figure A-iii. Incident flowchart

NON-PERSONAL INCIDENT RESPONSE PROCEDURES

All incidents including, but not limited to, fire, explosion, property damage, or environmental release will be responded to in accordance with the site-specific health and safety plan (HSP). In general, this will include securing the site as appropriate to the incident, turning control over to the emergency responders, or securing the site and summoning appropriate remedial personnel or equipment. Anchor QEA, LLC (Anchor QEA) will immediately notify the client of any major incident, fire, equipment or property damage, or environmental incident with a preliminary report. A full report will be provided within 72 hours.

Spills and Releases of Hazardous Materials

- When required, notify the National Response Center Hotline (800-424-8802) and US Coast Guard (USCG) (206.286.5400; VHF Channel 16). The following information should be provided:
  - Name and telephone number
  - Name and address of incident location
  - Time and type of incident
  - Name and quantity of materials involved, if known
  - Extent of injuries
  - Possible hazards to human health or the environment outside of the facility

If hazardous waste is released or produced through control of the incident, verify the following:

- Waste is collected and contained.
- Containers of waste are removed or isolated from the immediate site of the emergency.
- Treatment or storage of the recovered waste, contaminated soil or surface water, or any other material that results from the incident or its control is provided.
- No waste that is incompatible with released material is treated or stored in the facility until cleanup procedures are completed.

Verify that all emergency equipment used is decontaminated, recharged, and fit for its intended use before operations are resumed.

NEAR-MISS REPORTING

All near-miss incidents (i.e., those that could have reasonably led to an injury, environmental release, or other incident) must be reported to the field lead and PM immediately, so action can be taken to verify that the conditions that led to the near-miss incident are readily corrected to prevent future occurrences.

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Exhibits

Acronyms

AED	Automated External defibrillator
Anchor QEA	Anchor QEA, LLC
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CHSM	corporate health and safety manager
CPR	cardiopulmonary resuscitation
FC	field coordinator
HSM	health and safety manager
HSO	health and safety officer
HSP	health and safety plan
IDLH	immediately dangerous to life or health
JSA	job safety analysis
LDW	Lower Duwamish Waterway
L&I	Washington State Department of Labor and Industries
LEL	lower explosive limit
LFC	lowest feasible concentration
OEL	occupational exposure limit
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PFD	personal flotation device
PM	project manager
PPE	personal protective equipment
ppm	parts per million
QAPP	quality assurance project plan
STEL	short-term exposure limit
SVOC	semivolatile organic compound
TCDD	tetrachlorodibenzo-*p*-dioxin

TWA_8	8-hour time-weighted average
USCG	US Coast Guard
WAC	Washington Administrative Code
Windward	Windward Environmental LLC

1 Introduction

This site-specific health and safety plan (HSP) describes safe working practices for conducting field activities at potentially hazardous sites and for handling potentially hazardous materials/waste products. This HSP covers elements specified in 29 Code of Federal Regulations (CFR) 1910§120 and Washington Administrative Code (WAC) Chapter 296-843. The procedures and guidelines contained herein are based on generally recognized health and safety practices. Any changes or revisions to this HSP will be made by a written amendment that will become a permanent part of this document. The goal of this HSP is to establish procedures for safe working practices for all field personnel and visitors.

This HSP addresses all field activities associated with the periodic monitoring of fish, crab, clam, and surface water in the Lower Duwamish Waterway (LDW). During site work, this HSP is to be implemented by the field coordinator (FC), who is also the designated site health and safety officer (HSO), in cooperation with the Anchor QEA, LLC (Anchor QEA) health and safety manager (HSM) and the Anchor QEA and Windward Environmental LLC (Windward) project managers (PMs). All personnel involved in fieldwork on this project are required to comply with this HSP. The contents of this HSP reflect anticipation of the types of activities to be performed, knowledge of the physical characteristics of the site, and consideration of preliminary chemical data from previous investigations at the site. This HSP may be revised based on new information and/or changed conditions during site activities. Revisions will be documented in the project records.

This HSP will be modified by amendment, if necessary, to address changing field conditions or additional work tasks not already described in this document. Modifications will be reviewed by the HSM or authorized representative and approved by the PMs.

2 Site Description and Project Scope

2.1 SITE DESCRIPTION

The sampling area is in the LDW (see Maps 2-1 through 2-4 in the quality assurance project plan [QAPP] to which this document is an appendix). The QAPP provides complete details of the sampling program. This section summarizes the types of work that will be performed during field activities.

2.2 SCOPE OF WORK

Specific field activities included in the QAPP are as follows:

- Collection of clam samples in intertidal areas by foot with access via boat
- Collection of biological specimens from a boat using a high-rise otter trawl
- Collection of biological specimens from a boat using crab traps
- Deployment and retrieval of passive samplers
- Sample handling, processing, and shipping

Additional details on sampling design and methods are provided in Section 4 of the QAPP.

3 Health and Safety Personnel

Key health and safety personnel and their responsibilities are described below. These individuals are responsible for the implementation of this HSP; they will be responsible for informing all individuals who are assigned to work on the site, or who visit the site, of the contents of this plan, and for ensuring that each person signs the Health and Safety Plan Acknowledgment Form (see front matter). By signing the Health and Safety Plan Acknowledgment Form, individuals recognize the site health and safety hazards, known or suspected, and agree to adhere to the protocols required to minimize exposure to such hazards.

Project Managers: The Anchor QEA PM will have overall responsibility for the successful outcome of the project. The Anchor QEA PM will ensure that adequate resources and budget are provided for the health and safety staff to carry out their responsibilities during fieldwork. In consultation with the corporate health and safety manager (CHSM), the Anchor QEA PM will make final decisions concerning implementation of the HSP and resolution of site health and safety issues. The Anchor QEA PM will report directly to LDWG. The Windward PM will ensure proper implementation of the QAPP.

Field Coordinator/Health and Safety Officer: The FC/HSO will direct field sampling activities, coordinate the technical components of the field program with health and safety components, and ensure that work is performed according to the QAPP.

The FC/HSO will implement this HSP at the work location, and will be responsible for all health and safety activities and the delegation of duties to a health and safety technician in the field, if appropriate. The FC/HSO will also have stop-work authority, to be used if there is an imminent safety hazard or potentially dangerous situation. The FC/HSO or their designee will be present during sampling and operations.

Corporate Health and Safety Manager and Health and Safety Program Lead: The CHSM and health and safety program lead will have overall responsibility for preparation, approval, and revisions of this HSP. These individuals will not necessarily be present during fieldwork, but they will be readily available, if required, for consultation regarding health and safety issues during fieldwork.

Field Crew: All field crew members must be familiar and comply with the information in this HSP. They will also have the responsibility to report any potentially unsafe or hazardous conditions to the FC/HSO immediately. All field crew members will also have stop-work authority, to be used if there is an imminent safety hazard or potentially dangerous situation.

Site Visitors: Authorized visitors may come to the site to observe the sample collection/inspection activities. Visitors may be from the city, state, and federal regulatory and resource agencies that have a specific interest in the project, or visitors may be invited by the client, site contractors, or regulatory agencies. Visitors will be briefed on the hazards of the site, contents of the site-specific HSP, site safety rules,

hazard control measures, and required personal protective equipment (PPE). They will be escorted at all times by the field coordinator or a designated representative when entering work areas to observe the operations. Visitors will be expected to comply with all of the site health and safety requirements.

4 Hazard Evaluation and Control Measures

This section covers potential physical, biological (i.e., viral), and chemical hazards that may be associated with the proposed project activities, and presents control measures for addressing these hazards. An activity hazard analysis table, summarizing the potential hazards associated with each site activity and the recommended site controls for minimizing each potential hazard, is presented in Section 4.4.

Confined space entry will not be necessary for this project. Therefore, hazards associated with this activity are not discussed in this HSP.

4.1 PHYSICAL HAZARDS

For this project, it is anticipated that physical and biological hazards will present a greater risk of injury than will chemical hazards. Physical hazards are identified and discussed below.

4.1.1 Slips, trips, and falls

As with all field work, caution should be exercised to prevent slips on slick surfaces. In particular, sampling from a boat or other floating platform requires careful attention to minimize the risk of falling down or overboard. Extra care should be used in rainy conditions or on the shoreline where slick rocks or debris can be found. Slips can be minimized by wearing boots with good tread, made of material that does not become overly slippery when wet.

Trips are always a hazard on the uneven deck of a boat, in a cluttered work area, or in the intertidal zone where uneven substrate is common. Personnel will keep work areas as free as possible from items that interfere with walking.

Falls may be avoided by working as far from exposed edges as possible, by erecting railings, and by using fall protection when working on elevated platforms. For this project, open hatches may present a fall hazard, so hatches will remain closed when not being accessed for storage. Personnel should be aware of the area around any open hatches and use extra caution when accessing them.

4.1.2 Sampling equipment deployment

No hazards from using the sampling equipment during this project are anticipated. Clams will primarily hand collected by digging with a shovel or trowel; a high-rise otter trawl and crab traps will be used to collect tissue samples; passive samplers (affixed to a frame) will be manually deployed into and retrieved from the water. Sampling methods are described in further detail in Section 4 of the QAPP. Before sampling activities begin, there will be a training session for all field personnel for the equipment that will be onboard the sampling vessel.

All field personnel will wear modified Level D PPE when working around equipment. Corrective actions may involve installing guards over exposed, rotating parts; isolating or de-energizing equipment; establishing exclusion zones around high-hazard areas; and constructing guardrails around mechanical equipment to prevent inadvertent contact. Until such time as these hazards can be controlled or eliminated, project team members will avoid working in any areas where the hazard exists.

4.1.3 Falling overboard

Most sampling activities will be conducted from a boat. As with any work from a floating platform, there is a chance of falling overboard. US Coast Guard (USCG)-approved Type II or III personal flotation devices (PFDs) will be worn while working on the deck of the boat. If a person falls overboard into the water, a life ring will be thrown to the person immediately. One onboard person (a spotter) will keep an eye on the victim and shout the distance (boat lengths) and direction (o'clock) of the victim from the vessel. All work will stop work and the vessel will be used to retrieve the person in the water; the person in the water will be approached from downstream.

4.1.4 Manual lifting

Equipment and samples must be lifted and carried. Back strain can result if lifting is done improperly. During any manual handling tasks, personnel should lift with the load supported by their legs and not their backs. For heavy loads, an adequate number of people will be used, or if possible, a mechanical lifting/handling device will be used.

4.1.5 Heat stress

Heat stress could be an issue during hot days. Heat-related problems include heat rash, heat cramps, heat exhaustion, and heat stroke. The causes, symptoms, and first aid recommended by the National Institute for Occupational Health and Safety for each type of heat stress category are summarized in Table A-1. Workers should be aware of the key differences between the signs and symptoms of heat stroke and those of heat exhaustion, such as the lack of sweating, the color of the skin (red), and the rise in body temperature associated with the former. Heat stroke is a medical emergency that requires immediate medical attention.

Table A-1. Heat stress symptoms and recommended first aid

Type of Heat Stress	Cause	Symptoms	First Aid
Heat rash	Heat rash is a skin irritation caused by excessive sweating during hot, humid weather.	formation of rash (red cluster of pimples or small blisters) usually on the neck and upper chest, in the groin, under the breasts, and/or in elbow creases	Try to work in a cooler, less humid environment when possible.
			Keep the affected area dry. Dusting powder may be used to increase comfort.

Type of Heat Stress	Cause	Symptoms	First Aid
Heat cramps	Heat cramps usually affect workers who sweat a lot during strenuous activity. This sweating depletes the body's salt and moisture levels. Low salt levels in muscles cause painful cramps. Heat cramps may also be a symptom of heat exhaustion.	muscle pain or spasms, usually in the arms, legs, and abdomen	Have the person stop working and move him or her to a cool, shady area.
			Have the person drink clear juice or a sports beverage. Do not let person return to work until a few hours after cramps subside.
			Seek medical attention immediately if: 1) the person has heart problems, 2) the person is on a low-sodium diet, or 3) the cramps do not subside within 1 hour.
Heat exhaustion	Heat exhaustion is the body's response to an excessive loss of water and salt, usually through excessive sweating. Workers most prone to heat exhaustion are those who are elderly or have high blood pressure, and those working in a hot environment.	heavy sweating, extreme weakness or fatigue, dizziness or confusion, nausea, clammy moist skin, pale or flushed complexion, muscle cramps, slightly elevated body temperature, and fast and shallow breathing	Have the person stop working and move him or her to a cool, shady area.
			Give the person plenty of water, juice, or other cool, nonalcoholic beverages to drink.
			Have the person take a cool shower, bath, or sponge bath.
Heat stroke	Heat stroke is the most serious heat-related disorder. It occurs when the body becomes unable to control its temperature: The body's temperature rises rapidly, the sweating mechanism fails, and the body is unable to cool down. When heat stroke occurs, the body temperature can rise to 106°F or higher within 10 to 15 minutes. Heat stroke can cause death or permanent disability if emergency treatment is not given.	hot dry skin (no sweating), hallucinations, chills, throbbing headache, high body temperature, confusion/dizziness, and slurred speech.	Call 911 immediately.
			Have the person stop working and move him or her to a cool, shady area.
			Cool the person using methods such as 1) soaking person's clothes with water, 2) spraying, sponging, or showering person with room temperature water, and/or 3) fanning person's body. Ice or cold packs may also be used.

Source: CDC (2018), as modified in Amec et al. (2015).

4.1.6 Cold stress

Hypothermia occurs when the body's core temperature falls below 95°F. There is a risk of hypothermia if a crew member fails to dress warmly in cold weather, gets wet from rain or splashes, or falls into the water. To prevent hypothermia, all personnel will wear protective clothing appropriate for the weather conditions and physical activity. The FC/HSO will monitor all crew members for early symptoms of hypothermia (e.g., shivering, muscle incoordination, mild confusion). If such symptoms are observed, the FC/HSO will take immediate steps to reduce heat loss by providing extra layers of clothing, or by temporarily moving the affected crew member to a warmer environment. Other immediate steps that can be taken to reduce the symptoms of

hypothermia include minimizing exposure to cold and wet conditions, limiting sitting or standing still for long periods, rehydration with warm fluids, and the removal of any wet outer layers of clothing to permit sweat evaporation during rest periods in a warm environment.

Sampling operations and conditions that might result in the occurrence of frostbite are not anticipated. The sampling will take place during the time of year when extreme weather conditions are not expected to occur.

4.1.7 Weather

In general, field team members will be equipped for the normal range of weather conditions. The FC/HSO will be aware of current weather conditions, and of the potential for those conditions to pose a hazard to the field crew. Some conditions that might force work stoppage are electrical storms, strong winds, or high waves resulting from winds.

4.1.8 Small-boat operations

Boat operations are associated with various risks, such as: 1) passengers or crew members falling overboard/drowning, 2) coming in contact with other vessels or being contacted by other vessels operating in the area, 3) losing power or steering capability and drifting into hazardous areas (i.e., shores, bridges, industrial facilities, etc.), and 4) encountering severe weather and dangerous water conditions. The risk of a boating accident can be reduced by ensuring that the boat operators are experienced, operating the vessel in compliance with USCG rules and regulations, maintaining the vessel in good mechanical order, avoiding bad weather and dangerous water conditions, and ensuring that required emergency equipment is available onboard.

Safety precautions that will be implemented as part of boat operations for this project include the following:

- The vessels must have required USCG safely equipment onboard in good conditions, including a life jacket for each project team member, a first aid kit, fire extinguishers, distress flares, a throw-able life ring, navigation charts for the work area, running lights and a horn.
- Smoking is not permitted onboard the vessels.
- All crew members must be trained so that they know the locations and uses of onboard safety equipment.
- For vessels less than 25 ft long, at least one fire extinguisher must be onboard. For vessels greater than 26 ft in length but less than 40 ft, at least two fire extinguishers must be onboard.
- A life jacket must be worn by project team members at all times while working on boats, piers, docks that are not equipped with guardrails, and vessels when not tied to shore.

- The VHF radio must be turned on and monitored.
- Crew members should not untie mooring lines until instructed to do so by the vessel operator.
- Crew members should never jump between the vessel and the dock or other vessels.
- Docks, piers, and shoreline areas should be approached slowly. The boat should never be fended off by placing your body between the boat and any object.
- All crew members should watch for hazards such as approaching vessels or wakes. It should never be assumed that other crew members see such hazards; therefore, they should be alerted to any potential risks that are observed.
- Crew members should be aware of overhead power lines and underwater utility corridors.
- If lightning or thunder occurs before the crew can get safely off the water, the 30/30 rule should be used: If the time between seeing lightning and hearing thunder is 30 seconds or less, the boat should be moved near a tall structure such as a bridge and remain there until 30 minutes after the last thunder is heard.
- If refueling is necessary, the engine should be turned off and allowed to cool before fueling is attempted.

4.1.9 Vessel traffic

Because of the high volume of vessel traffic on the LDW, precautions and safe boating practices will be implemented to ensure that the field boat does not interrupt such traffic. As practical, the field boat will stay out of the navigation channel. Vessel traffic will be monitored on VHF channel 16.

4.1.10 Homeless encampment

Field staff will have contact information for the project field lead and PMs while performing all field investigation activities. If a homeless encampment is encountered during implementation of the planned work, the encampment residents will not be disturbed, and the field crew will leave the area. From a safe location, the field lead will notify the PMs, who in turn will notify LDWG representatives to evaluate next steps.

Workers who come across discarded needles or other sharp items should never pick them up with bare hands or toss them into general garbage. Using pliers or tongs to pick up such items is safer; they should then be placed in a Sharps disposal container or other strong container with a lid. Waste containers containing blood or other potentially infectious materials, or equipment contaminated with blood/other potentially infectious materials, must have the orange or red label with the biohazard symbol. The label must be securely attached so it cannot become lost or accidentally removed. Red

bags or red containers can be substituted for labels. Appropriate PPE, including latex or nitrile gloves, should also be worn.

4.2 BIOLOGICAL HAZARDS

COVID-19 is a contagious respiratory disease caused by the SARS-CoV-2 virus (CDC 2022). Infection with SARS-CoV-2 can cause mild to severe illness and can be fatal. Symptoms may include fever (>100.4°F), dry cough, shortness of breath, aches and pains, headache, sore throat, diarrhea, conjunctivitis, chest pain or pressure, a rash on the skin, discoloration of fingers or toes, loss of speech or movement, and a loss of taste or smell—while still others have been asymptomatic and experienced no symptoms at all. According to the US Department of Health and Human Services Centers for Disease Control and Prevention (CDC), symptoms of COVID-19 may appear in as few as 2 days or as many as 14 days after exposure (CDC 2022). The virus is primarily spread via inhalation and from person-to-person, including:

- Between people within close contact distance of one another (within approximately 6 ft for 15 minutes or more in a 24-hour period)
- Through respiratory droplets, which may land in or be inhaled via the mouths or noses of people who are nearby when an infected person coughs or sneezes

The virus may also be transmitted by touching a surface or object that has SARS-CoV-2 on it and then one's own mouth, nose, or eyes, but this method is not believed to be the primary route by which the virus spreads. It is believed that people are most contagious when they are most symptomatic (i.e., experiencing fever, cough, and/or shortness of breath), but transmission may be possible before symptoms are evident (CDC 2022).

As appropriate, site workers will implement good hygiene and infection control practices, including:

- Receiving the COVID-19 vaccine (required for all workers)
- Staying home when sick or showing symptoms[1]
    - If employees are showing symptoms, it is recommended that they contact their health care provider for medical advice. Further steps taken may include an examination and testing as recommended by their health care provider.
    - If employees are showing any symptoms, they will be asked to leave the site and not return for a minimum of 14 days or until released by a health care professional.
- Limiting field personnel to the minimum individuals required to safely complete the work

[1] If an employee has traveled to an affected country outside the United States or has had close contact (within 6 ft for 15 minutes or more in a 24-hour period) with infected individuals within the United States, self-quarantine from the project site is required until cleared by a health care professional to return.

- Limiting time spent in groups in enclosed spaces to the extent possible
- Monitoring workers for symptoms
- Following respiratory etiquette, including covering coughs and sneezes
- Washing hands frequently and thoroughly. If soap and running water are not immediately available, alcohol-based sanitizer containing at least 60% alcohol will be used.
- Wearing disposable PPE during sampling and properly disposing of PPE items as often as necessary
- Increasing physical distance among and between employees and others (i.e., use of social distancing strategies)
- Providing additional barriers to exposure, such as face coverings, face shields, and protective eyewear, as necessary
- Avoiding sharing personal items and using other workers' phones, pens, work tools, and equipment, when possible, or wiping down between uses
- Maintaining regular housekeeping practices, including routine cleaning and disinfecting of surfaces, equipment, and other elements of the work environment utilizing chemicals listed on the US Environmental Protection Agency list N as being suitable for COVID-19
- Considering alternative strategies to reduce exposure, such as staggering work shifts and breaks and covering common touch areas with cleanable materials
- Following the same prevention guidelines while off-site including while traveling, at a hotel, and participating in other activities in order to address potential exposures outside the workplace

4.3 CHEMICAL HAZARDS

Previous investigations have shown that some chemicals are present at higher-than-background concentrations in the sampling area. For the purposes of discussing the potential exposure of individuals to chemicals in sediments, the chemicals of concern are metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), semivolatile organic compounds (SVOCs), dioxins/furans, and hydrogen sulfides. Detailed information on exposure routes and chemical hazards is included in Table A-2.

Table A-2. Chemicals of concern

Chemical	Exposure Routes	Symptoms	Target Organs	OEL (STEL)[a]	Odor Threshold (ppm)	LEL (%)	Ionization Potential (eV)
PCBs (Chlorodiphenyls) (42% Cl/53469-21-9) (54% Cl/11097-69-1)	inhalation, skin absorption, ingestion, skin and/or eye contact	• irritated eyes, chloracne; liver damage; reproductive effects • potential occupational carcinogen	skin, eyes, liver, reproductive system	0.001 mg/m^3 TWA_8 skin IDLH/ceiling limit – 5 mg/m^3	na	na	?
PAHs – as coal tar pitch volatiles. (Includes benzo(a)pyrene, chrysene, phenanthrene, fluoranthene, pyrene, acenaphthene, methylnaphthalenes, and anthracene)	skin, eye, inhalation, and ingestion hazard	• Direct contact or exposure to the vapors may be irritating to the eyes. Direct contact can be highly irritating to the skin and can cause dermatitis. Exposure to high vapor concentrations may cause headaches, nausea, vomiting, and other symptoms. Chemical includes human carcinogens. Exposure to all routes should be carefully controlled to levels as low as possible. • confirmed animal carcinogen.	respiratory system, skin, bladder, kidneys	0.2 mg/m^3 TWA_8 0.1 mg/m^3 TWA_8 (cyclohexane-extractable fraction) IDLH/carcinogen – 80 mg/m^3	Varies	na	?
Dioxins/furans (as 2,3,7,8-TCDD)	inhalation, skin absorption, ingestion, skin and/or eye contact	• irritated eyes; allergic dermatitis, chloracne; porphyria; gastrointestinal disturbance; possible reproductive, teratogenic effects • in animals: liver, kidney damage; hemorrhage • potential occupational carcinogen	eyes, skin, liver, kidneys, reproductive system	LFC proposed OEL of 0.2 ng/m^3 skin IDLH/carcinogen – LFC	na	?	?
Hydrogen sulfide (7783-06-04) 1 ppm = 1.40 mg/m^3	inhalation, skin and/or eye contact	• irritated eyes, respiratory system; apnea, coma, convulsions; conjunctivitis, eye pain, lacrimation (discharge of tears), photophobia (abnormal visual intolerance to light), corneal vesiculation; dizziness, headache, lassitude (weakness, exhaustion), irritability, insomnia; gastrointestinal disturbance • liquid form: frostbite	eyes, respiratory system, central nervous system	1 ppm TWA_8 (5 ppm) ceiling limit – 10 ppm (10 minutes over an 8-hr shift) IDLH – 100 ppm	0.03 ppm	4.0	10.46
Arsenic and inorganic compounds (7440-38-2)	inhalation, skin absorption, skin and/or eye contact, ingestion	• ulceration of nasal septum, dermatitis, gastrointestinal disturbances, peripheral neuropathy, resp irritation, hyperpigmentation of skin • potential occupational carcinogen	liver, kidneys, skin, lungs, lymphatic system	ceiling limit – 0.002 mg/m^3 (15-minute) IDLH/carcinogen – 5 mg/m^3	na	na	na
Barium and soluble compound, including Barium chloride (7440-39-3) (10361-37-2)	inhalation, skin and/or eye contact	irritated eyes, skin, upper respiratory system; skin burns; gastroenteritis; muscle spasm; slow pulse, extrasystoles (heart contractions); hypokalemia (deficiency of potassium in the bloodstream)	eyes, skin, respiratory system, heart, central nervous system	0.5 mg/m^3 TWA_8 IDLH – 50 mg/m^3	na	na	na
Cadmium and compounds (7440-43-9)	inhalation, ingestion	• pulmonary edema, dyspnea (breathing difficulty), cough, chest tightness, substernal (occurring beneath the sternum) pain; headache; chills, muscle aches; nausea, vomiting, diarrhea; anosmia (loss of the sense of smell), emphysema, proteinuria, mild anemia • potential occupational carcinogen	respiratory system, kidneys, prostate, blood, prostatic & lung cancer	0.005 mg/m^3 TWA_8 IDLH/carcinogen – 9 mg/m^3	na	na	na
Chromium (II) inorganic compounds	inhalation, ingestion, skin and/or eye contact	irritated eyes; sensitization dermatitis	eyes, skin	0.5 mg/m^3 TWA_8 IDLH – 250 mg/m^3	na	na	na
Chromium (III) inorganic compounds (7440-47-3)	inhalation, ingestion, skin and/or eye contact	irritated eyes; sensitization dermatitis	eyes, skin	0.5 mg/m^3 TWA_8 (total dust) 0.003 mg/m^3 TWA_8 (inhalable fraction) IDLH – 25 mg/m^3	na	na	na
Chromium (VI) inorganic compounds (18540-29-9) (1333-82-0 as CrO_3)	inhalation, ingestion, skin and/or eye contact	• irritated respiratory system; nasal septum perforation; liver, kidney damage; leukocytosis (increased blood leukocytes), leukopenia (reduced blood leukocytes), eosinophilia; eye injury, conjunctivitis; skin ulcer, sensitization dermatitis • potential occupational carcinogen	blood, respiratory system, liver, kidneys, eyes, skin, lung cancer	0.0002 mg/m^3 TWA_8 IDLH/carcinogen – 15 mg/m^3	na	na	na

Chemical	Exposure Routes	Symptoms	Target Organs	OEL (STEL)[a]	Odor Threshold (ppm)	LEL (%)	Ionization Potential (eV)
Lead and inorganic compounds (7439-92-1)	inhalation, ingestion, skin and/or eye contact	lassitude (weakness, exhaustion), insomnia; facial pallor; anorexia, weight loss, malnutrition; constipation, abdominal pain, colic; anemia; gingival lead line; tremor; paralysis wrist, ankles; encephalopathy; kidney disease; irritation eyes; hypertension	eyes, gastrointestinal tract, central nervous system, kidneys, blood, gingival (gum) tissue	0.05 mg/m^3 TWA_8 IDLH – 100 mg/m^3	na	na	na
Mercury, elemental and inorganic compounds (7439-97-6)	inhalation, skin absorption, ingestion, skin and/or eye contact	irritated eyes, skin; cough, chest pain, dyspnea (breathing difficulty), bronchitis, pneumonitis; tremor, insomnia, irritability, indecision, headache, lassitude (weakness, exhaustion); stomatitis (inflammation of mucous membranes of the mouth), salivation; gastrointestinal disturbance, anorexia, weight loss; proteinuria (abnormal quantities of protein in the urine)	eyes, skin, respiratory system, central nervous system, kidneys	0.025 mg/m^3 TWA_8 ceiling limit – 0.1 mg/m^3 skin IDLH – 10 mg/m^3	na	na	na
Selenium compounds (7782-49-2)	inhalation, ingestion, skin and/or eye contact	irritated eyes, skin, nose, throat; visual disturbance; headache; chills, fever; dyspnea (breathing difficulty), bronchitis; metallic taste, garlic breath, gastrointestinal disturbance; dermatitis; eye, skin burns; In Animals: anemia; liver necrosis, cirrhosis; kidney, spleen damage	eyes, skin, respiratory system, liver, kidneys, blood, spleen	0.2 mg/m^3 TWA_8 IDLH – 1 mg/m^3	na	na	na
Silver metal, and soluble compounds (7440-22-4)	inhalation, ingestion, skin and/or eye contact	blue-gray eyes, nasal septum, throat, skin; irritation, ulceration skin; gastrointestinal disturbance	nasal septum, skin, eyes	0.01 mg/m^3 TWA_8 IDLH – 10 mg/m^3	na	na	na

[a] "Carcinogen" denotes a potential or confirmed human carcinogen; "skin" denotes an OEL based primarily on a skin exposure hazard.

IDLH – immediately dangerous to life or health
LEL – lower explosive limit
LFC – lowest feasible concentration
na – not applicable
OEL – occupational exposure limit
PAH – polycyclic aromatic hydrocarbon
PCB – polychlorinated biphenyl
ppm – parts per million
STEL – short-term exposure limit
TCDD – tetrachlorodibenzo-*p*-dioxin
TWA_8 – 8-hour time-weighted average

4.3.1 Exposure routes

Potential routes of chemical exposure include inhalation, dermal contact, and ingestion. Exposure will be minimized by using safe work practices (Section 6) and by wearing the appropriate PPE. Further discussion of PPE requirements is presented in Section 7.

4.3.1.1 Inhalation

Inhalation is not expected to be an important route of exposure, because sampling will be conducted outside on a boat, in the field, or in a well-ventilated area.

4.3.1.2 Dermal exposure

Dermal exposure to hazardous substances associated with sediments, surface water, or equipment decontamination will be controlled by the use of PPE, and by adherence to detailed sampling and decontamination procedures.

4.3.1.3 Ingestion

Incidental ingestion of sediment or surface water is not considered a major route of exposure for this project. Accidental ingestion of surface water is possible. However, careful handling of equipment and containers while onboard the boat should prevent water from splashing or spilling during sample collection and handling activities.

4.3.2 Description of chemical hazards

4.3.2.1 Metals

Exposure to metals at this site may occur via ingestion or skin contact. As mentioned, neither is a likely exposure route. Metal fumes or metal-contaminated dust will not be encountered during field and sample handling activities. Large amounts of sediment would need to be ingested for any detrimental effects to occur. Momentary skin contact allows little, if any, opportunity for metals to pass into the body. Field procedures require immediate washing of sediments from exposed skin.

4.3.2.2 Polycyclic aromatic hydrocarbons and semivolatile organic compounds

Exposure to PAHs or SVOCs at this site may occur via ingestion or skin contact. Inhalation—the most important human health exposure pathway for this group of chemicals—is not expected to occur at this site. Large amounts of sediment would need to be ingested for any detrimental effects to occur. Some PAHs may be carcinogenic after long periods of skin contact. However, momentary skin contact allows little, if any, opportunity for compounds to pass into the body. Field procedures require immediate washing of sediments from exposed skin.

4.3.2.3 Polychlorinated biphenyls

Exposure to PCBs at this site may occur via ingestion or skin contact. Acute and chronic exposure can damage the liver and cause symptoms of edema, jaundice, anorexia, nausea, abdominal pains, and fatigue. PCBs are a suspected human carcinogen,

although large amounts of sediment would need to be ingested for any detrimental effects to occur. Prolonged skin contact with PCBs may cause acne-like symptoms known as chloracne. Irritation to eyes, nose, and throat may also occur. However, momentary skin contact allows little, if any, opportunity for compounds to pass into the body. Field procedures require immediate washing of sediments from exposed skin.

4.3.2.4 Dioxins/furans

Exposure to dioxins/furans at this site may occur via ingestion or skin contact. Acute and chronic exposure can damage the liver, increase the risk of diabetes and abnormal glucose tolerance, and possibly increase the risk for reproductive and developmental effects. 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) is a possible human carcinogen, and a mixture of dioxins/furans with six chlorine atoms (four of the six chlorine atoms at the 2-, 3-, 7-, and 8-positions) is a probable human carcinogen. However, large amounts of sediment would need to be ingested for any detrimental effects to occur. Prolonged skin contact with dioxins/furans may cause chloracne. Other effects on the skin, such as red skin rashes, have been reported to occur in people following exposure to high concentrations of 2,3,7,8-TCDD. Momentary skin contact allows little, if any, opportunity for the passage of any of the compounds into the body. Field procedures require the immediate washing of sediments from exposed skin.

4.3.2.5 Hydrogen sulfides

Exposure to hydrogen sulfides at this site may occur primarily via inhalation or eye and skin contact. At lower concentrations typically found in sediments, hydrogen sulfides emit a rotten egg odor. Acute and chronic exposure to this odor can irritate the respiratory tract and eyes and cause symptoms of headaches, dizziness, nausea, and abdominal pains. Exposure to high concentrations may result in bronchitis, bronchial pneumonia, coma, unconsciousness, or respiratory arrest. However, momentary skin contact allows little, if any, opportunity for compounds to pass into the body. Field procedures require adequate ventilation and immediate washing of sediments from exposed skin.

4.4 ACTIVITY HAZARD ANALYSIS

The activity hazard analysis summarizes the hazards associated with periodic monitoring activities and the controls that can reduce or eliminate the risk of these hazards occurring (Table A-3). Job safety analysis (JSA) sheets, included in Exhibit 1, also describe potential hazards that may be encountered during implementation of the periodic monitoring activities (including clam, fish, and crab tissue sample collection, and deployment and retrieval of passive samplers).

Table A-3. Activity hazard analysis

Hazard[a]	Control
Slips and trips	Use extra care when walking on uneven and unstable surfaces along the shoreline, and under wet/slippery conditions. Wear boots with good tread.
Falling overboard	Use care in boarding/departing from the vessel. Wear a PFD. Provide and make readily accessible on each boat a life ring to throw to the person in the water. Have one onboard person (a spotter) keep an eye on the victim and shout the distance (boat lengths) and direction (o'clock) of the victim from the vessel. Stop work and use the vessel to retrieve the person in the water. Approach the person from downstream.
Skin or eye contact with contaminated sediments or liquids	Wear modified Level D PPE. Immediately wash sediments from exposed skin. Use an eyewash, if necessary, for contaminated or foreign debris in the eyes.
Back strain	Use appropriate technique for lifting equipment and samples, or seek help.
Overhead hazards	Use caution and be aware of overhead and gear hazards such as the high-rise otter trawl. Wear a hard hat and modified Level D PPE when working around this equipment.
Open hatches	Keep hatches closed when not being accessed. Be aware around hatch area and use caution when entering/exiting hatches.
Heat stress	Monitor crew members for signs/symptoms of heat stress. Remove person to cool area and remove extra layers of clothing. Promote evaporative cooling and rehydrate with electrolytic fluids.
Cold stress	Monitor crew members for signs/symptoms of hypothermia. Minimize prolonged exposure to wet and cold conditions. Remove person to warm area and remove wet clothing. Rehydrate with warm fluids.
Weather	Monitor weather forecast and local conditions. Stop work if conditions pose a hazard (e.g., electrical storms, high winds) and resume work when safe to do so.
Fatigue	Take regular breaks, and limit repeated excessively long work days.
Pinch points and cuts	Be aware of pinch points and potential for cuts during sample collection, handling, and processing. Handle equipment and tools with care. Use safety knives if necessary, and follow instruction manuals for any power tools.
Working at night	Make sure all lights are functional (navigation lights, flashlights, PDF lights, contractor-supplied lighting, etc.). Routinely inspect work area for unsafe conditions.
SARS-CoV-2 virus (COVID-19)	Follow all basic L&I requirements and guidance for preventing COVID-19. • Keep workers known or suspected to have COVID-19 from working around others by following appropriate isolation or quarantine guidance, as outlined by the Washington State Department of Health. • Provide hand-washing facilities and supplies, and regularly clean and sanitize surfaces. • Educate workers about COVID-19 prevention in the language they understand best. • Provide written notice of potential COVID-19 exposure within one business day to all workers, as well as the employers of any subcontracted workers who were at the same work site as the person who tested positive (without disclosing the person's identity). • Report COVID-19 outbreaks to L&I within one business day if they involve 10 or more workers at a workplace or job site with more than 50 workers. • Allow workers to voluntarily wear masks (respirators, medical procedure masks, or cloth face coverings) as long as it does not create a safety or security issue.

[a] Responses to boat emergencies are addressed in Table A-5.

L&I – Washington State Department of Labor and Industries

PFD – personal flotation device

PPE – personal protective equipment

5 Work Zones and Shipboard Access Control

During sampling and sample handling activities, work zones will be established to identify where sample collection and processing are actively occurring. The intent of the zones is to limit the migration of sample material out of its zone, and to restrict unauthorized access to active work areas by defining work zone boundaries. The work zones are described below.

5.1 SAMPLING ZONE

A sampling work zone will encompass the exclusion area where sample collection and handling activities are being performed. The FC/HSO will delineate the work zone as a particular area onboard the collection vessel or at each intertidal shoreline sampling location. Only persons with appropriate training, PPE, and authorization from the FC/HSO will be allowed to enter this zone while work is in progress.

5.2 DECONTAMINATION ZONE

A decontamination zone where personnel will clean soiled boots and/or PPE prior to leaving the work zone will be set up. The zone will have the buckets, brushes, soapy water, rinse water, or wipes necessary to clean boots, PPE, or other equipment leaving the work zone. Plastic bags will be provided for expendable and disposable materials. If the sampling location does not allow for the establishment of a decontamination zone, the FC/HSO will provide alternatives to prevent the spread of contamination.

Decontamination of the boat will be completed at the end of each work day. Cockpit and crew areas will be rinsed down with water to minimize the accumulation of sediment.

5.3 SUPPORT ZONE

The support zone is any work area beyond the sampling work zone and decontamination zone boundaries where sample collection and processing do not occur. Activities in the support zone include boat operations (e.g., piloting the boat and remotely controlling sampling equipment), administrative work (e.g., observing the field effort, taking notes, filling out paperwork, communicating with project managers, and directing field staff), and rest breaks. Prior to entering the support zone, personnel are required to decontaminate or dispose of soiled PPE or equipment to limit the spread of contamination into the clean area.

5.4 ACCESS CONTROL

Security and control of access to the boat will be the responsibility of the FC/HSO and boat captain. Boat access will be granted to necessary project personnel and authorized visitors only. Any security or access control problems will be reported to the client or appropriate authorities.

6 Communications and Safe Work Practices

Communications at the job site will occur by verbal direction, use of hand signals, radio, or a combination of all three. Site personnel will carry cellular telephones and a list of emergency telephone numbers included in this HSP. These telephone numbers are listed and in the front matter and in Section 14.3 of this HSP. Boat operators will have VHF radios capable of communicating with USCG emergency services and with other vessels operating in the immediate work area. An air horn will be staged at each work area to initiate an evacuation of the site in an emergency, should other means of communication (i.e., radio, telephone, etc.) fail. Site personnel will be informed of site emergency procedures and communication protocols during their initial site orientation.

Following common sense will minimize the risk of exposure or accidents at this work site. The following general safety rules will be adhered to on-site:

- Do not climb over or under obstacles of questionable stability (e.g., docks, piers).
- Do not eat, drink, smoke, or perform other hand-to-mouth transfers in the work zone.
- Work only in well-lighted spaces.
- Never enter a confined space without the proper training, permits, and equipment.
- Make eye contact with vessel/sampling equipment operators when moving within the range of their equipment.
- Be aware of the movements of shipboard equipment when not in the operator's range of vision.
- Get immediate first aid for all cuts, scratches, abrasions, or other minor injuries.
- Use the established sampling and decontamination procedures.
- Always use the buddy system.
- Be alert to your own and other workers' physical conditions.
- Report all accidents, no matter how minor, to the FC/HSO.
- Do not do anything dangerous or unwise even if ordered by a supervisor.

7 Personal Protective Equipment and Safety Equipment

Appropriate PPE will be worn as protection against potential hazards. In addition, a PFD will be required when working onboard the boat. Prior to donning PPE, the field crew will inspect their PPE for any defects that might render the equipment ineffective.

Fieldwork will be conducted in Level D or modified Level D PPE, as discussed in Sections 7.1 and 7.2. Situations requiring PPE beyond modified Level D are not anticipated. Should the FC/HSO determine that PPE beyond modified Level D is necessary, the HSM will be notified and an alternative selected. This HSP also acknowledges that site conditions may change during implementation of the work, possibly leading to a change in exposure pathways or contaminants of concern. If changes are observed, evaluation of potential changes in PPE needs will be completed.

New personnel or visitors will be informed of PPE requirements during their initial site briefing (see Section 3).

7.1 LEVEL D PERSONAL PROTECTIVE EQUIPMENT

Workers performing general activities during which skin contact with contaminated materials is unlikely will wear Level D PPE. Level D PPE includes the following:

- Protective clothing
- Chemical-resistant steel-toed boots
- Chemical-resistant gloves
- Safety glasses
- High-visibility vests
- American National Standards Institute/American Society for Testing and Materials-compliant hard hats (e.g., on trawling vessel)

7.2 MODIFIED LEVEL D PERSONAL PROTECTIVE EQUIPMENT

Workers performing activities during which skin contact with contaminated materials is possible, and during which inhalation risks are not expected, will be required to wear an impermeable outer suit. The type of outerwear will be chosen according to the types of chemical contaminants that might be encountered. Modified Level D PPE includes the following:

- Impermeable outer garb such as rain gear
- Waterproof and chemical-resistant steel-toed boots
- Waders and wader boots
- Chemical-resistant outer gloves
- Heavy-duty waterproof gloves

- High-visibility vests
- Hard hats (e.g., on trawling vessel)
- Safety glasses
- Protective face covering (as needed based on location and COVID-19 community transmission level)

When the ability to remain socially distant (i.e., minimum 6 ft apart) is limited (on boats), workers will be expected to comply with CDC recommendations for reducing exposure to COVID-19 in public spaces. As necessary, workers will be provided with safety glasses and disposable medical face masks designed to reduce the transfer of saliva and respiratory droplets to others, and to help block potentially infectious materials from reaching the skin, eyes, mouth, or nose of the wearer during daily activities. When used, workers will be expected to change disposable masks at least halfway through each work day and as frequently as necessary (i.e., when soiled or damaged). Face shields will also be available as an additional option for protection from COVID-19 exposure.

7.3 SAFETY EQUIPMENT

In addition to PPE that will be worn by shipboard personnel, basic emergency and first aid equipment will be provided. Equipment for the field team will include:

- A copy of this HSP
- A first aid kit adequate for the number of personnel
- Emergency eyewash
- Sunscreen
- Fire extinguisher

The FC/HSO will ensure that the safety equipment is onboard. Equipment will be checked daily to ensure its readiness for use.

8 Monitoring Procedures for Site Activities

A monitoring program that addresses the potential site hazards will be maintained. For this project, air, dust, and noise monitoring will not be necessary. The sampled media will be wet and will not pose a dust hazard, and none of the equipment will emit high-amplitude (> 85 dBA) sound. For this project, the monitoring program will consist of all workers monitoring themselves and their co-workers for signs that might indicate physical stress or illness.

All personnel will be instructed to look for and inform each other of any deleterious changes in their physical or mental condition during the performance of all field activities. Examples of such changes are as follows:

- Headaches
- Dizziness
- Nausea
- Fever
- Coughing
- Shortness of breath (difficulty breathing)
- Muscle pain
- Sore throat
- Loss of sense of taste or smell
- Symptoms of heat stress
- Blurred vision
- Cramps
- Irritation of eyes, skin, or respiratory system
- Changes in complexion or skin color
- Changes in apparent motor coordination
- Increased frequency of minor mistakes
- Excessive salivation or changes in papillary response
- Changes in speech ability or speech pattern
- Shivering
- Blue lips or fingernails

If personnel develop any of these conditions, work will be halted immediately and the affected person(s) evaluated. If further assistance is needed, personnel at the local hospital will be notified, and an ambulance will be summoned if the condition is thought to be serious. If the condition is the direct result of sample collection or handling activities, procedures will be modified to address the problem.

9 Decontamination

Decontamination is necessary to prevent the migration of contaminants from the work zone(s) into the surrounding environment, and to minimize the risk of exposure of personnel to contaminated materials that might adhere to PPE. The following sections discuss personnel and equipment decontamination. The following supplies will be available to perform decontamination activities:

- Wash buckets
- Rinse buckets
- Scrub brushes
- Clean water sprayers
- Paper towels
- Plastic garbage bags
- Alconox® or similar decontamination solution

9.1 MINIMIZATION OF CONTAMINATION

The first step in addressing contamination is to prevent or minimize exposure to biological hazards and existing contaminated materials and the spread of those materials. During field activities, the FC/HSO will enforce the following measures:

Personnel:

- Limit field staff to minimum number required to safely complete the work.
- Wash hands frequently and thoroughly. Use alcohol-based sanitizer with at least 60% alcohol if soap and running water are not readily available.
- Follow proper coughing and sneezing etiquette.
- Avoid sharing personal items.
- Avoid group gatherings in enclosed spaces.
- Maintain proper social distance (i.e., minimum 6 ft) to extent possible.
- Follow the same prevention guidelines off site including while traveling, at a hotel, and participating in other activities in order to address potential exposures outside the workplace.
- Do not walk through areas of obvious or known contamination, if avoidable.
- Do not handle, touch, or smell contaminated materials directly.
- Make sure PPE has no cuts or tears prior to use.
- Fasten all closures on outer clothing, covering with tape if necessary.
- Protect and cover any skin injuries.

- Stay upwind of airborne dusts and vapors.
- Do not eat, drink, chew tobacco, or smoke in the work zones.

Sampling equipment and boat:

- Avoid or minimize handling of equipment, tools, and supplies by multiple people.
- Clean or disinfect touch surfaces, handheld equipment, tools, and supplies frequently.
- Place clean equipment on a plastic sheet or aluminum foil to avoid direct contact with contaminated media.
- Keep contaminated equipment and tools separate from clean equipment and tools.
- Clean boots before entering the boat.

9.2 PERSONNEL DECONTAMINATION

The FC/HSO will ensure that all site personnel are familiar with personnel decontamination procedures. Personnel will perform the following decontamination procedures, as appropriate, before eating lunch, taking a break, or leaving the work location:

1. If outer suit is heavily soiled, rinse it off.
2. Remove outer suit.
3. Wash and rinse outer gloves and boots with soapy water.
4. Remove outer gloves; inspect and discard if damaged.
5. Remove inner gloves and discard.
6. Wash hands.

Before returning to work, personnel will re-don all necessary PPE. If leaving for the day, personnel will dispose of soiled, expendable PPE.

9.3 SAMPLING EQUIPMENT DECONTAMINATION

Sampling equipment will be decontaminated, as described in the QAPP, to minimize sample contamination and worker exposure to contamination from samples and potential exposure to COVID-19. The following practices will be followed:

- Shared equipment or supplies and workspaces will be disinfected frequently or between uses, as appropriate.
- Safety glasses and face shields will be assigned to a single user; will be disinfected frequently, at the end of the day or between uses; and will be stored in a clean, sealable bag.

- Sample processing surfaces will be cleaned and lined with aluminum foil to prevent direct contact with samples.
- Ice chests will be scrubbed with Alconox® detergent and rinsed with deionized water prior to any sampling activities. Lids and handles will be cleaned frequently throughout each day.
- Wet ice used for sample storage during field activities will be contained in separate plastic bags, and samples will be placed in resealable, waterproof plastic bags to avoid contamination from melting ice.
- Sampling equipment will be free from contaminants such as oils, grease, and fuels.

10 Disposal of Contaminated Materials

Contaminated materials that may be generated during field activities include PPE and excess sample material. These contaminated materials will be disposed of as an integral part of the project.

10.1 Personal Protective Equipment

All disposable sampling materials and PPE—such as disposable coveralls, gloves, and paper towels used in sample processing—will be placed in heavyweight garbage bags. Filled garbage bags will be placed in a normal refuse container for disposal as solid waste.

10.2 Excess Sample Materials

At each sampling location, excess or unwanted specimens collected for tissue samples will be returned to the river.

11 Training Requirements

Individuals performing work at locations where potentially hazardous materials and conditions may be encountered must meet specific training requirements. It is not anticipated that hazardous concentrations of contaminants will be encountered in sampled material, so training will consist of site-specific instruction for all personnel and oversight of inexperienced personnel by an experienced person for one working day. The following sections describe the training requirements for this fieldwork.

11.1 PROJECT-SPECIFIC TRAINING

In addition to HAZWOPER training, as described in Section 3.5 of the QAPP, field personnel will undergo training specifically for this project. All personnel and visitors must read this HSP and be familiar with its contents before beginning work or providing oversight. They must acknowledge reading the HSP by signing the Health and Safety Plan Acknowledgement Form (see front matter). The signed form will be kept in the project files.

The boat captain and FC/HSO will also be required to have USCG Auxiliary Boating Safety certification. The boat captain or a designee will provide project-specific training prior to the first day of fieldwork and whenever new workers arrive. Field personnel will not be allowed to begin work until project-specific training has been completed and documented by the FC/HSO. Training will address the HSP and all health and safety issues and procedures pertinent to field operations. Training will include, but not be limited to, the following topics:

- Activities with the potential for exposure to chemicals
- Activities that pose physical hazards, and actions to control the hazards
- Vessel access control and procedures
- Use and limitations of PPE
- Decontamination procedures
- Emergency procedures
- Use and hazards of sampling equipment
- Location of emergency equipment on the vessel
- Vessel safety practices
- Vessel evacuation and emergency procedures

11.2 DAILY SAFETY BRIEFINGS

The FC/HSO or a designee and the boat captain will present safety briefings before the start of each day's activities. These safety briefings will outline the activities expected for the day, update work practices and hazards, verify that medical screening has been

completed (if applicable), explain protective measures, address any specific concerns associated with the work location, and review emergency procedures and routes. Social distancing will be maintained during safety briefings, and COVID-19 safety requirements will be visibly posted at the work site (Inslee 2020; L&I 2020).

The FC/HSO or designee will document all safety briefings using the daily safety briefing form included in Exhibit 1.

11.3 FIRST AID AND CPR

At least two members of the field team must have first aid and cardiopulmonary resuscitation (CPR) training. Documentation of which individuals possess first aid and CPR training will be kept in the project health and safety files.

12 Medical Surveillance

A medical surveillance program conforming to the provisions of 29 CFR 1910§120(f) is not necessary for field team members on this project, because they do not meet any of the following four criteria outlined in the regulations for implementation of a medical surveillance program:

- Employees who are or may be exposed to hazardous substances or health hazards at or above permissible exposure levels for 30 days or more per year (1910.120(f)(2)(I))
- Employees who must wear a respirator for 30 days or more per year (1910.120(f)(2)(ii))
- Employees who are injured or become ill as a result of possible overexposures involving hazardous substances or health hazards from an emergency response or hazardous waste operation (1910.120(f)(2)(iii))
- Employees who are members of HAZMAT teams (1910.120(f)(2)(iv))

Specific attention will be given to the requirement to screen all workers at the beginning of their shifts by taking their temperatures and asking them if they have a fever, cough, shortness of breath, fatigue, muscle aches, or new loss of taste or smell. If used, thermometers used shall be 'no touch' or 'no contact' models to the greatest extent possible. If a 'no touch' or 'no contact' thermometer is not available, the thermometer will be properly sanitized between each use. Any worker with a temperature of 100.4°F or higher will be considered to have a fever and will be sent home.

As described in Section 8, employees will monitor themselves and each other for any deleterious changes in their physical or mental conditions during the performance of all field activities.

Regarding monitoring exposures to COVID-19, there are three possible scenarios:[2]

- Primary exposure: when an employee has tested positive for the virus
- Secondary exposure: when an employee has had close contact with someone diagnosed with or presumed to have COVID-19 within two days of the onset of symptoms or positive test, whichever comes first
- Tertiary exposure: when an employee has had close contact with a secondary exposure or was in the same general work area with a confirmed or presumed case but there was no close contact.

The FC/HSO (or designee) will also act as the on-site COVID-19 Supervisor, and shall monitor the health of employees and enforce the measures established to minimize exposure to COVID-19. Workers are expected to inform the FC/HSO if they develop symptoms of or have been exposed to anyone with COVID-19.

[2] Adapted from Anchor (2020).

12.1 COVID-19 PRIMARY EXPOSURE

If an employee has tested positive for COVID-19, the FC/HSO will immediately take the following actions:

- The employee will be immediately sent away for isolation (until cleared by the third party healthcare provider) if they are at the site.
- The employee's steps will be traced to identify work areas with which the individual may have been in close contact in the two days prior to symptoms or a positive test.
- All identified areas will be quarantined and marked as off limits to all site personnel, until a decontamination/disinfection process following CDC guidelines has been implemented.
- Employees who have been in close contact (within 6 ft for 15 minutes or more during a 24-hour period) with the infected individual will be asked to quarantine for 14 days or until released by the third party healthcare provider.

12.2 COVID-19 SECONDARY EXPOSURE

If an employee has had close contact with someone who has been diagnosed with COVID-19 within the two days prior to symptoms or a positive test, whichever comes first, the FC/HSO will immediately take the following actions:

- Immediately send the employee home until released by the third party healthcare provider.
- Consult with the Washington State Department of Health for additional guidance if the employee is diagnosed with COVID-19 and has been instructed to self-quarantine.
- Inform the CHSMs and PMs immediately.
- Continue cleaning common touch areas with recommended disinfectants.
- Follow primary exposure scenario (Section 12.1) if an employee is confirmed as positive for COVID-19.

12.3 COVID-19 TERTIARY EXPOSURE

It is more difficult to manage tertiary exposure because there is innately less control in a situation wherein an employee may have had close contact with a secondary exposure, or has been in the general area with a confirmed or presumed case with no close contact. The FC/HSO will request that all site workers provide any relevant exposure information. If an employee is believed to have been subject to tertiary exposure, take the following actions:

- Consult with the Washington State Department of Health for additional guidance if the acquaintance who is diagnosed with or screened for COVID-19 has been instructed to self-quarantine.
- Inform the CHSMs and PMs immediately.
- Follow up with the field team after test results for the potentially exposed employee have been received.
- Continue cleaning common touch areas with recommended disinfectants.
- Follow secondary exposure scenario (Section 12.2) if the acquaintance is confirmed as positive for COVID-19.

13 Reporting and Record Keeping

Each member of the field crew will sign the Health and Safety Plan Acknowledgement Form (see front matter). If necessary, accident/incident report forms and Occupational Safety and Health Administration (OSHA) Form 200s will be completed by the FC/HSO.

The FC/HSO or a designee will maintain a health and safety field logbook with daily records of health- and safety-related details for the project. The logbook will utilize daily safety briefing forms (Exhibit 1) and must be bound, and the pages must be numbered consecutively. Entries will be made with indelible ink. At a minimum, each day's entries must include the following information:

- Project name or location
- Names of all personnel onboard
- Weather conditions
- Type of fieldwork being performed

The person maintaining the entries will initial and date the bottom of each completed page. Blank space at the bottom of an incompletely filled page will be lined out. Each day's entries will begin on the first blank page after the previous workday's entries.

Additionally, for COVID-19 tracking purposes, a record of all site workers and visitors and their contact information (i.e., phone numbers and e-mail addresses) will be kept on file for a minimum for four weeks from the last day they were on site.

14 Emergency Response Plan

As a result of the hazards onboard the sampling vessels and the conditions under which operations will be conducted, the potential exists for an emergency situation to occur. Emergencies may include personal injury, exposure to hazardous substances, fire, explosion, or release of toxic or non-toxic substances (spills). OSHA regulations require that an emergency response plan be available for use onboard to guide actions in emergency situations.

Hazards may also be encountered with shore-based activities and sampling. Emergencies may include personal injury, exposure to hazardous substances, fire, explosion, or release of toxic or non-toxic substances (spills). OSHA regulations require that an emergency response plan be available for use on site to guide actions in emergency situations.

Onshore organizations will be relied upon to respond to emergency situations. Given the location of the site, the local fire department and ambulance service can provide timely response. Field personnel will be responsible for identifying an emergency situation, providing first aid if applicable, notifying the appropriate personnel or agency, and evacuating any hazardous area. Shipboard personnel will attempt to control only very minor hazards that could present an emergency situation, such as a small fire; otherwise, all personnel will rely on outside emergency response resources.

The following sections identify the onboard individual(s) who should be notified in case of emergency, provide a list of emergency telephone numbers, offer guidance for particular types of emergencies, and provide directions and a map for getting from any sampling location to a hospital.

14.1 PRE-EMERGENCY PREPARATION

Before the start of field activities, the FC/HSO will ensure that preparation has been made in anticipation of emergencies. Preparatory actions include the following:

- Meeting between the FC/HSO and equipment handlers concerning emergency procedures in the event that a person is injured
- A training session given by the FC/HSO informing all field personnel of emergency procedures, locations of emergency equipment and its uses, and proper evacuation procedures
- A training session given by senior staff on operating field equipment to apprise field personnel of operating procedures and specific risks associated with that equipment
- Ensuring that field personnel are aware of the existence of the emergency response plan in the HSP, and ensuring that a copy of the HSP including all attachments accompanies the field team

14.2 PROJECT EMERGENCY COORDINATOR

The FC/HSO will serve as the project emergency coordinator in the event of an emergency. They will designate their replacement during those times when they are not onboard the vessel, are not on site, or are not serving as the project emergency coordinator; the designation will be noted in the logbook. The project emergency coordinator will be notified immediately when an emergency is recognized. The project emergency coordinator will be responsible for evaluating the emergency situation, notifying the appropriate emergency response units, coordinating access with those units, and directing interim actions before the arrival of emergency response units. The project emergency coordinator will notify the HSM and the PMs as soon as possible after initiating an emergency response action. The PMs will have responsibility for notifying the client.

14.3 EMERGENCY RESPONSE CONTACTS

All personnel must know whom to notify in the event of an emergency situation, even though the FC/HSO has primary responsibility for notification. Table A-4 lists the names and phone numbers for emergency response services and individuals.

Table A-4. Emergency response contacts

Contact	Telephone Number
Emergency Numbers	
Ambulance	911
Police	911
Fire	911
Harborview Medical Center	206.323.3074
Emergency Responders	
US Coast Guard Emergency General information	 206.286.5400 206.442.5295 VHF Channel 16
National Response Center	800.424.8802
US Environmental Protection Agency	800.424.8802
Washington State Department of Ecology – Northwest Region Spill Response (24-hour emergency line)	206.649.7000
Emergency Contacts	
Anchor QEA Project Manager	
Tom Wang	206.903.3314
Windward Project Manager	
Kathy Godtfredsen	206.812.5413
Corporate Health and Safety Manager	
David Templeton	206.910.4279

Contact	Telephone Number
Health and Safety Program Lead	
Tim Shaner	251.281.3386
Field Coordinator/ Field Health and Safety Officer	
Thai Do (Windward)	206.812.5407
Matt Woltman (Anchor QEA)	206.903.3327

14.4 RECOGNITION OF EMERGENCY SITUATIONS

Emergency situations will generally be recognizable by observation. An injury or illness will be considered an emergency if it requires treatment by a medical professional and cannot be treated with simple first aid techniques.

14.5 DECONTAMINATION

In the case of evacuation, decontamination procedures will be performed only if doing so does not further jeopardize the welfare of site workers. If an injured individual is also heavily contaminated and must be transported by emergency vehicle, the emergency response team will be informed of the type of contamination. To the extent possible, contaminated PPE will be removed from the injured individual, but only if doing so does not exacerbate the injury. Plastic sheeting will be used to reduce the potential for spreading contamination to the inside of the emergency vehicle.

14.6 FIRE

Field personnel will attempt to control only small fires, should they occur. If an explosion appears likely, personnel will follow evacuation procedures specified during the training session. If a fire cannot be controlled with the available fire extinguisher that is part of the required safety equipment, personnel will either withdraw from the vicinity of the fire or evacuate the boat or area as specified in the training session.

14.7 PERSONAL INJURY

In the event of serious personal injury, including unconsciousness, possibility of broken bones, severe bleeding or blood loss, burns, shock, or trauma, the first responder will immediately do the following:

- Designate an individual to call 911 and administer first aid, if qualified.
- If not qualified, seek out an individual who is qualified to administer first aid, if time and conditions permit.
- Notify the project emergency coordinator of the incident, the name of the injured individual(s), the location of the individual, and the nature of the injury.

The FC/HSO or designee will immediately do the following:

- Notify the boat captain and the appropriate emergency response organization.
- Assist the injured individual(s).
- Follow the emergency procedures for retrieving or disposing of equipment reviewed in the training session, and leave the site en route to the predetermined land-based emergency pickup.
- Designate someone to accompany the injured individual to the hospital.
- If a life-threatening emergency occurs (i.e., injury where death is imminent without immediate treatment), the FC/HSO or boat captain will call 911 and arrange to meet the ambulance unit at the nearest accessible dock or other appropriate location.
- If a non-life-threatening emergency occurs (i.e., broken bones, minor lacerations, etc.), the project emergency coordinator will follow the procedures outlined above and proceed to the Harbor Island Marina, or to an alternative location of their choice if that would be more expedient.
- Notify the HSM and the PM.

If the project emergency coordinator determines that an emergency response is not necessary, they may direct someone to decontaminate and transport the individual by vehicle to the nearest hospital. Directions showing the route to the hospital are in Section 14.11.

If a worker leaves the boat or site to seek medical attention, another worker should accompany him to the hospital. When in doubt about the severity of an injury or exposure, always seek medical attention as a conservative approach, and notify the project emergency coordinator.

The project emergency coordinator will be responsible for completing all accident/incident field reports, OSHA Form 300s, and other required follow-up forms.

14.8 OVERT PERSONAL EXPOSURE OR INJURY

If an overt exposure to toxic materials occurs, the first responder to the victim will initiate actions to address the situation. The following actions should be taken, depending on the type of exposure.

14.8.1 Skin contact

- Wash/rinse the affected area thoroughly with copious amounts of soap and water.
- If eye contact has occurred, rinse the eyes for at least 15 minutes using the eyewash that is part of the onboard emergency equipment.

- After initial response actions have been taken, seek appropriate medical attention.

14.8.2 Inhalation

- Move victim to fresh air.
- Seek appropriate medical attention.

14.8.3 Ingestion

Seek appropriate medical attention.

14.8.4 Puncture wound or laceration

Seek appropriate medical attention.

14.9 Spills and Spill Containment

No bulk chemicals or other materials subject to spillage are expected to be used during this project. Accordingly, no spill containment procedure is required for this project. If crew members encounter a spill created by any others, they will immediately notify the contractor in charge of the spill areas so they can initiate a cleanup action.

14.10 Boating Emergency Hazards

Emergency responses to boating hazards are described in Table A-5. Boat operators will have VHF radios that are capable of communicating with US Coast Guard emergency services and with other boats operating in the immediate work area. USCG monitors channel 16.

Table A-5. Potential boat emergency hazards and responses

Potential Emergency Hazard	Response
Fire or explosion	If manageable, attempt to put out a small fire with a fire extinguisher. Otherwise, call the USCG or 911, evacuate the area (by life rafts, rescue boat, or swimming), and meet at a designated location. The HSO will take roll call to make sure everyone has evacuated safely. Emergency meeting locations will be determined in the field during the daily safety briefings.
Medical emergency/ personal injury	At least two people with current first aid and CPR training will be onboard the vessel at all times. This person will attempt to assess the nature and critical path of the injury, call 911 immediately, and apply first aid/CPR if necessary. Stop work and wait for medical personnel to arrive. Fill out a site accident report.
Falling into an open hatch	Stop work and rescue the person, if safe and necessary. Assess the nature of the injury, and follow the response for medical emergency/personal injury.
Person overboard	Immediately throw a life ring to the person in the water. Have one onboard person(a spotter) keep an eye on the victim and shout the distance (boat lengths) and direction (o'clock) of the victim from the vessel. Stop work and use the vessel to retrieve the person in the water. Approach the person from downstream.
Sinking vessel	Call the USCG immediately. If possible, wait for a rescue boat to arrive to evacuate vessel personnel. See fire/explosion section (above) for emergency evacuation procedures. The HSO will take roll call to make sure everyone has evacuated safely.
Hydraulic oil spill or leak	If the leak/spill is small, immediately apply absorbent pads to control the leak and continue work. If the leak/spill is uncontainable, stop work, call 911 immediately, and wait for assistance. The vessel operator will assess the personal safety hazard associated with the leak/spill and begin evacuation procedures if necessary.
Lack of visibility	If navigation visibility or personal safety is compromised because of smoke, fog, or other unanticipated hazards, stop work immediately. The vessel operator and HSO will assess the hazard and, if necessary, send out periodic horn blasts to notify other vessels potentially in the area of the sampling vessel's location. Move to a secure location (i.e., berth) and wait for visibility to clear.
Loss of power	Stop work and call the USCG for assistance. Vessel personnel should watch for potential collision hazards and notify vessel operator if hazards exist. Secure vessel to a berth, dock, or mooring as soon as possible.
Collision	Stop work and call the USCG for assistance. HSO and vessel operator will assess damage and potential hazards. If necessary, vessel will be evacuated and secured until repairs can be made.

CPR – cardiopulmonary resuscitation
HSO – health and safety officer
USCG – US Coast Guard

14.11 EMERGENCY ROUTES TO THE HOSPITAL

The name, address, and telephone number of the hospital that will be used to provide medical care is as follows:

Harborview Medical Center
325 - 9th Avenue
Seattle, WA
206.323.3074

Directions from the vicinity of the LDW to Harborview Medical Center are as follows (Figure B-ii):

From the Duwamish River boat ramp (at South River Street, beneath the 1st Avenue South bridge):

- Drive east on South River Street.
- Turn left on Occidental Avenue South.
- Turn left on East Marginal Way South.
- Turn right on South Michigan Street.
- Look for entrance ramps to I-5 Northbound.
- Drive north on I-5.
- Take the James Street exit.
- Drive east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

From the Harbor Island Marina (1001 Southwest Klickitat Way):

- From marina parking lot, turn sharp right onto Klickitat Way Southwest.
- Turn slight right onto Southwest Spokane Street
- Turn slight left to take the ramp toward WA-99 N/I-5/Columbian Way.
- Keep left at the fork in the ramp.
- Stay straight to go onto West Seattle Bridge.
- Merge onto I-5 North via the ramp on the left.
- Take the James Street exit.
- Head east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

From South Park Marina (8604 Dallas Ave South):

- From marina parking lot, turn right onto Dallas Avenue South.
- Turn right onto 16th Avenue South.
- Turn left on East Marginal Way South.
- Look for entrance ramps to I-5 Northbound.
- Drive north on I-5.
- Take the James Street exit.
- Drive east on James Street to 9th Avenue.
- Turn right on 9th Avenue.
- Emergency entrance will be two blocks south on the right.

15 References

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EXHIBIT 1.
DAILY SAFETY BRIEFING FORM AND JOB SAFETY ANALYSIS SHEETS

Daily Safety Briefing Form

Date: _____

Project No: _____

Project Name: _____

Person Conducting Meeting: _____ **Health & Safety Officer:** _____ **Project Manager:** _____

TOPICS COVERED: *Highlighted topics are required*

- ☐ Emergency Procedures and Evacuation Route
- ☐ Directions to Hospital
- ☐ HASP Review and Location
- ☐ Safety Equipment Location
- ☐ Proper Safety Equipment Use
- ☐ Employee Right-to-Know/ SDS Location
- ☐ Fire Extinguisher Location
- ☐ Eye Wash Station Location
- ☐ Buddy System
- ☐ Self and Coworker Monitoring
- ☐ Lines of Authority
- ☐ Communication
- ☐ Site Security
- ☐ Vessel Safety Protocols
- ☐ Work Zones
- ☐ Vehicle Safety and Driving/ Road Conditions
- ☐ Equipment Safety and Operation
- ☐ Proper Use of PPE
- ☐ Decontamination Procedures
- ☐ Near Miss Reporting Procedures
- ☐ Lifting Techniques
- ☐ Slips, Trips, and Falls
- ☐ Hazard Exposure Routes
- ☐ Heat and Cold Stress
- ☐ Overhead and Underfoot Hazards
- ☐ Chemical Hazards
- ☐ Flammable Hazards
- ☐ Biological Hazards
- ☐ Eating/Drinking/Smoking
- ☐ Reviewed Prior Lessons Learned
- ☐ Field Team Medical Conditions for Emergency Purposes (Confidential): _____
- ☐ Other: _____

Weather Conditions: _____

Daily Work Scope: _____

Site-specific Hazards: _____

Safety Comments: _____

Attendees

Printed Name	Signature

End of Day Wellness Check

PLAYING IT SAFE

Job Safety Analysis

Field Activities

Project Name: Lower Duwamish Waterway Remedial Design	**Project Number:** 180067-02.01	**JSA Number:** 001	**Issue Date:** May 29, 2020
Location: Seattle, Washington	**Contractor:** Anchor QEA, LLC and Windward Envronmental, LLC	**Analysis by:** K. Gross	**Analysis Date:** May 29, 2020
Work Operation: Field activities	**Superintendent/Competent Person:** M. Woltman	**Revised by:** NA	**Revised Date:** NA
Required Personal Protective Equipment (PPE): • Modified Level D—Long pants, long sleeves, and/or Tyvek coveralls if handling potentially contaminated media, and steel-toed footwear conforming to ASTM International (ASTM) F2412-05/ASTM F2413-05, safety glasses/splash goggles, nitrile gloves, high-visibility safety vest, and protective face mask • Depending on activity, the following PPE may also be required: hard hat, outer gloves, face shield, and, if boating, U.S. Coast Guard-approved personal flotation device (PFD; see cold stress section for cold-weather PFD information)		**Reviewed by:** M. Woltman	**Reviewed Date:** May 29, 2020
		Approved by: T. Shaner	**Approved Date:** May 29, 2020

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
General	COVID-19	• Refer to Health and Safety Plan (HSP).	Refer to HSP.
If boating		• Follow the Job Safety Analysis (JSA) for boating activities.	
Outdoor, physical activity	Slips, trips, and falls	• Avoid walking while writing or texting—maintain a heads-up posture. • Be aware of potentially slippery surfaces and tripping hazards. Use handrails where available. Wear footwear that has sufficient traction. • Maintain good housekeeping practices. Clean up all spills immediately. • Be aware of weather effects on the work area, including wet and/or frozen ground. • Jumping, running, and horseplay are prohibited. • Keep all areas clean and free of debris to prevent any trips and falls. • Be aware of and limit loose clothing or untied shoelaces that may contribute to slips, trip, and falls. • Notify the field team members of any unsafe conditions.	• Routinely inspect work area for unsafe conditions.

Job Safety Analysis

Field Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Outdoor, physical activity (continued)	Heat stress	• Adjust work schedules, as necessary, to avoid the hottest part of the day. • Take rest breaks as warranted. • Provide shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods. • Maintain body fluids at normal levels. • Train workers to recognize the symptoms of heat-related illness.	• Review weather forecast prior to field work. • Monitor workers' physical conditions. • Monitor outside temperature versus worker activity.
	Cold stress	• Provide shelter (enclosed, heated environment) to protect personnel during rest periods. • Educate workers to recognize the symptoms of frostbite and hypothermia. • Use appropriate cold-weather gear, up to and including Mustang-type bib coveralls or jacket/bib combinations. • Consider additional precautions if working near water in cold weather. • Have a dry change of clothing available. • Train workers to recognize the symptoms of cold-related illness.	• Review weather forecast prior to field work. • Monitor workers' physical conditions and PPE. • Monitor outside and water temperature versus worker activity and PPE.
	Rain or snow	• Wear appropriate PPE (rain gear). • Be aware of slip hazards, puddles, and electrical hazards when working in wet conditions. • If extremely cold conditions are forecast, consider additional precautions or postponing work activity.	• Review weather forecast prior to field work. • Inspect PPE daily prior to use. • Routinely inspect work area for deteriorating conditions.
	Sunshine	• Have sunscreen available for ultraviolet protection. • Have abundant water available to prevent dehydration. • Consider wearing wide-brimmed headwear and light-colored, lightweight, sun-blocking clothing.	• Ensure that sunscreen and water are available.
	Lightning	• Do not begin or continue work until lightning subsides for at least 30 minutes. Disconnect and do not use or touch electronic equipment. • Immediately head for shore if on the water and lightning is observed. If not able to get to shore, disconnect and do not use or touch the major electronic equipment, including the radio, throughout the duration of the storm.	• Obtain weather forecast and updates as needed.

Job Safety Analysis

Field Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Outdoor, physical activity (continued)	High winds	• Wear goggles or safety glasses if dust or debris are visible.	• Review weather forecast prior to field work. • Ensure that goggles or safety glasses are available.
	Biological hazards (flora [e.g., poison ivy and poison oak] and fauna [e.g., bees, spiders, and mosquitoes])	• Be aware of likely biological hazards in the work area. • Wear appropriate clothing (i.e., hat, long-sleeve shirt, long pants, leather gloves, boots, and Tyvek coveralls, as appropriate), and apply insect repellant. • Wear hand and arm protection when clearing plants or debris from the work area. • Be aware of potential wildlife and defensive behavior (e.g., nesting birds, or animals with young).	• Ensure that insect repellent is available. • Inspect clothing and skin for insects (e.g., ticks) after working in insect-prone areas.
	Noise exposure	• Wear hearing protection in high noise environments or when working around heavy machinery or equipment (action level of 85 decibels averaged over an 8-hour day).	• Ensure that hearing protection is available.

Training Requirements:

- All personnel working on hazardous waste sites must receive appropriate training as required by 29 Code of Federal Regulations (CFR) 1910.120(e), including but not limited to initial 40-hour, 8-hour supervisor, and annual 8-hour refresher trainings.
- Medical clearance must be received on an annual basis as required by 29 CFR 1910.120(f).
- If boating is involved, and a professional captained vessel is not in use, boat operators must take the appropriate state or provincial boater safety courses.
- All assigned employees are required to familiarize themselves with the contents of this JSA before starting a work activity and review it with their supervisor during their daily safety meeting.

Job Safety Analysis

General Boating Activities

Project Name: Lower Duwamish Waterway Remedial Design	**Project Number:** 180067-02.01	**JSA Number:** 002	**Issue Date:** May 29, 2020
Location: Seattle, Washington	**Contractor:** Anchor QEA, LLC and Windward Envronmental, LLC	**Analysis by:** K. Gross	**Analysis Date:** May 29, 2020
Work Operation: General boating activities	**Superintendent/Competent Person:** M. Woltman	**Revised by:** NA	**Revised Date:** NA
Required Personal Protective Equipment (PPE): • U.S. Coast Guard (USCG)-approved personal flotation device (PFD; see cold stress section for cold-weather PFD information)		**Reviewed by:** M. Woltman	**Reviewed Date:** May 29, 2020
		Approved by: T. Shaner	**Approved Date:** May 29, 2020

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
General	COVID-19	• Refer to Health and Safety Plan (HSP).	• Refer to HSP.
Walking on deck	Pinch points	• Secure any unsecured objects on deck; they may shift quickly in wave, current, or engine acceleration conditions. • Maintain a safe distance from closing mechanisms and moving parts, such as on sampling gear. • Avoid placing your hands or yourself between the boat and the dock or piles.	• Docks, piers and shoreline areas should be approached slowly.

Job Safety Analysis

General Boating Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
	Slips, trips, and falls	• Avoid walking while writing or texting—maintain a heads-up posture. • Be aware of potentially slippery surfaces, including boat decks, riprap, muddy or algae-covered rocks, shoreline plants or seaweed, thick mud, and tripping hazards. Use handrails where available. • Wear footwear that has sufficient traction. • Maintain good housekeeping practices. • Clean up all spills immediately. • Be aware of weather effects on the work area, including wet and/or frozen ground. • Jumping, running, and horseplay are prohibited. • Be cautious when entering or exiting the vessel, and load/unload items onto/off of the pier or shore once boarded. • Keep all areas clean and free of debris to prevent any trips and falls. • Notify the field team members of any unsafe conditions. • Keep rope lines neatly coiled and stowed. Avoid stepping on or over lines.	• Routinely inspect work area for unsafe conditions. • All crew members should watch for hazards such as approaching vessels or wakes. It should never be assumed that other crew members see such hazards.
	Exceeding boat capacity	• Keep the number of passengers and equipment as posted on boat placards within limits at all times. If conditions warrant, reduce capacity to maintain boat stability.	• Ensure that field team is aware of limits and adheres accordingly.
Walking on deck (continued)	Noise exposure	• Wear hearing protection in high noise environments or when working around heavy machinery or equipment (action level of 85 decibels averaged over an 8-hour day).	• Ensure that hearing protection is available.
Working outdoors	Heat stress	• Adjust work schedules, as necessary, to avoid the hottest part of the day. • Take rest breaks as warranted. • Provide shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods. • Maintain body fluids at normal levels. • Train workers to recognize the symptoms of heat-related illness.	• Review weather forecast prior to field work. • Monitor workers' physical conditions. • Monitor outside temperature versus worker activity.
	Cold stress	• Provide shelter (enclosed, heated environment) to protect personnel during rest periods. • Educate workers to recognize the symptoms of frostbite and hypothermia. • If the combined air and water temperature is below 90 degrees Fahrenheit (˚F), wear a USCG-approved float coat, Mustang-type bib coveralls, or one-piece survival suit. • Have a dry change of clothing available. • Train workers to recognize the symptoms of cold-related illness.	• Review weather forecast prior to field work. • Monitor workers' physical conditions and PPE. • Monitor outside and water temperature versus worker activity and PPE.

Job Safety Analysis

General Boating Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
	Rain or snow	• Wear appropriate PPE (rain gear). • Be aware of slip hazards, puddles, and electrical hazards when working in wet conditions. • If extremely cold conditions are forecast, consider additional precautions or postponing work activity.	• Review weather forecast prior to field work. • Inspect PPE daily prior to use. • Routinely inspect work area for deteriorating conditions.
	Sunshine	• Have sunscreen available for ultraviolet protection. • Have abundant water available to prevent dehydration. • Consider wearing wide-brimmed headwear and light-colored, lightweight, sun-blocking clothing.	• Ensure that sunscreen and water are onboard.
	Fog	• Wait for fog to lift for adequate visibility.	• Review weather forecast prior to field work.
Working outdoors (continued)	Lightning	• Do not begin or continue work until lightning subsides for at least 30 minutes. Disconnect and do not use or touch electronic equipment. • Immediately head for shore if on the water and lightning is observed. • If not able to get to shore, disconnect and do not use or touch the major electronic equipment, including the radio, throughout the duration of the storm. • If the time between seeing the lightening and hearing thunder is 3 seconds or less, the boat should be moved near a tall structure such as a bridge and remain there until 30 minutes after the last thunder is heard.	• Obtain weather forecast and updates as needed.
	High river flows or high waves	• Be aware of waves and forecasts and recent rainfall in your watershed.	• Have forecast available.
	High winds	• Wear goggles or safety glasses if dust or debris are visible. • Stow or secure loads or equipment that could be moved by wind, particularly when underway.	• Review weather forecast prior to field work. • Ensure that goggles or safety glasses are onboard.
	Biological hazards (e.g., mosquitoes)	• Wear appropriate clothing (i.e., hat, long-sleeve shirt, long pants, leather gloves, boots, and Tyvek coveralls, as appropriate), and apply insect repellent.	• Ensure that insect repellent is onboard.

Job Safety Analysis

General Boating Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Vessel emergencies	Person overboard	If you witness someone fall overboard: • Yell, "Person overboard!" • Throw a flotation device immediately. • If the engine is running, take it out of gear and swing the stern clear to keep from hitting the person. • Call 911 or USCG as appropriate. • Assign a spotter to keep the person in sight at all times. • Contact nearby vessels for assistance. • Recover the person from the water. If you fall overboard: • Hold your mouth and nose closed and protect your head. • When you reach the surface, look for movement, listen for sounds, and call for help. Use the whistle attached to the PFD and activate the beacon light. • It is only sensible to swim if there is reason to believe you have a chance of reaching your destination. Too much movement in cold water causes hypothermia.	• Ensure that flotation devices are available. • Ensure that team wears PFDs. • Inspect PFDs for integrity, particularly the cartridge charge on inflatable PFDs. • All crew members must be trained so that they know the location and use of onboard safety equipment.
Vessel emergencies (continued)	Fire, abandon ship	• Be prepared to abandon ship in case of major fire (too large to control with a fire extinguisher), or other emergency. • Only the boat captain can order abandon ship. • Communicate intent to abandon ship to all personnel onboard. • Notify USCG and nearby vessels of intent to abandon ship. • Call 911. • Notify the Project Manager and Field Lead, if time permits. • Be aware of the propeller position before abandoning ship. • Identify a rally point for all personnel. • Know the dangers of hypothermia. • Use the buddy system to support injured personnel.	• For vessels less than 25 feet long, at least one fire extinguisher must be onboard. For vessels greater than 26 feet in length but less than 40 feet, at least two fire extinguishers must be onboard. • Review abandon ship procedures with field team prior to work.
Navigation	Boat traffic	• Maintain a safe operating distance from shoreline and other vessels.	• Be aware of on-water surroundings. • The VHF radio must be turned on and monitored.

Job Safety Analysis

General Boating Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Motor vehicle operation and trailering	Boat not secured properly	• Ensure that latches, straps, antennas, and onboard gear are secure. Ensure that motor is up and lights are plugged in for driving. • Follow Job Safety Analysis (JSA) for motor vehicle operation. • Crew members should not untie mooring lines until instructed to do so by the vessel operator.	• Inspect around entire boat before driving.

Training Requirements:

- All personnel working on hazardous waste sites must receive appropriate training as required by 29 Code of Federal Regulations (CFR) 1910.120(e), including but not limited to initial 40-hour, 8-hour supervisor, and annual 8-hour refresher trainings.
- Medical clearance must be received on an annual basis as required by 29 CFR 1910.120(f).
- If professional captained vessel is not in use, boat operators must take appropriate state or provincial boater safety courses.
- All assigned employees are required to familiarize themselves with the contents of this JSA before starting a work activity and review it with their supervisor during their daily safety meeting.

Job Safety Analysis

Decontamination Activities

Project Name: Lower Duwamish Waterway Remedial Design	**Project Number:**	**JSA Number:** 003	**Issue Date:** May 29, 2020
Location: Seattle, Washington	**Contractor:** Anchor QEA, LLC and Windward Environmental, LLC	**Analysis by:** T. Do	**Analysis Date:** May 29, 2020
Work Operation: Decontamination activities	**Superintendent/Competent Person:** T. Do	**Revised by:** NA	**Revised Date:** NA
Required Personal Protective Equipment (PPE): • Modified Level D—Long pants, long sleeves, and/or Tyvek coveralls if handling potentially contaminated media, steel-toed footwear conforming to American Society for Testing and Materials International (ASTM) F2412-05/ASTM F2413-05, safety glasses/splash goggles, high-visibility safety vest, medical face mask, and nitrile gloves • Depending on activity, the following personal protective equipment (PPE) may also be required: hard hat, face shield, and, if boating, U.S. Coast Guard-approved personal flotation device (PFD).		**Reviewed by:** S. McGroddy	**Reviewed Date:** May 29, 2020
		Approved by: S. McGroddy	**Approved Date:** May 29, 2020

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
General	COVID-19	• Refer to Health and Safety Plan (HSP) (Attachment H)	Refer to HSP (Attachment H).
If boating		• Follow the Job Safety Analysis (JSA) for boating activities.	
Decontamination area set up	Vehicle, heavy equipment traffic, or boat traffic in work area	• Wear high-visibility safety vest and hard hat PPE. • Be alert when working around heavy equipment and/or other boats, especially if wearing hearing protection.	• Ensure that safety vests are available for staff and visitors.
	Muscle strain or injuries from improper lifting	• Use proper lifting techniques or ask for assistance with heavy objects. • If boating, avoid carrying objects directly onto or off of the boat; rather, load/unload objects while on the boat to/from the pier/shore.	• Evaluate weight and center of gravity of heavier items prior to lifting or moving.
	Biological hazards (fauna [e.g., ticks, bees, spiders, and mosquitoes])	• Be aware of likely biological hazards in the work area. • Wear appropriate clothing (i.e., hat, long-sleeve shirt, long pants, leather gloves, boots, and Tyvek coveralls, as appropriate), and apply insect repellent.	• Ensure that insect repellent is available. • Inspect clothing and skin for insects (e.g., ticks) after working in insect-prone areas.

Job Safety Analysis

Decontamination Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Decontamination activities	Injury from hand and power tool operation (e.g., spatula or drill)	• Be aware of sharp edges on hand tools (e.g., spatulas, knives, drill bits, and saw blades). • Be aware of electrical connections and water hazards when working with electric- or battery-operated tools. • Ensure that all tools are working properly; repair or replace defective tools. Repair any defective tools when unplugged and off. • Keep guards on power tools when not in use.	• Inspect tools to ensure that they are in good working order. • Inspect electrical connections (if applicable). • Inspect tools periodically to ensure dry and clean operation.
	Noise exposure	• Wear hearing protection in high noise environments or when working around heavy machinery or equipment (action level of 85 decibels averaged over an 8-hour day).	• Ensure that hearing protection is available.
	Slips, trips, and falls	• Avoid walking while writing or texting—maintain a heads-up posture. • Be aware of potentially slippery surfaces and tripping hazards. Use handrails where available. Wear footwear that has sufficient traction. • Maintain good housekeeping practices. Clean up all spills immediately. • Be aware of weather effects on the work area, including wet and/or frozen ground. • Jumping, running, and horseplay are prohibited. • Keep all areas clean and free of debris to prevent any trips and falls. • Notify the field team members of any unsafe conditions.	• Routinely inspect work area for unsafe conditions.
	Ingestion of contaminants or decontamination fluids, or skin or eye contact with contaminants or decontamination fluids	• Wear appropriate PPE to prevent/reduce exposure. • Contact 911, as necessary; perform CPR if breathing stops. • Move exposed person away from source of contamination and rinse mouth. If exposure to skin occurs, promptly wash contaminated skin using soap or mild detergent and water. Rinse eyes with large amounts of water. • Follow decontamination procedures as outlined in the HSP.	• Ensure that decontamination procedures are on hand and are reviewed. • Ensure that PPE and rinsing water are available.
Working outdoors	Heat stress	• Adjust work schedules, as necessary, to avoid the hottest part of the day. • Take rest breaks as warranted. • Provide shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods. • Maintain body fluids at normal levels. • Train workers to recognize the symptoms of heat-related illness.	• Review weather forecast prior to field work. • Monitor workers' physical conditions. • Monitor outside temperature versus worker activity.

Job Safety Analysis

Decontamination Activities

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Working outdoors (continued)	Cold stress	• Provide shelter (enclosed, heated environment) to protect personnel during rest periods. • Educate workers to recognize the symptoms of frostbite, hypothermia and other cold-related illness. • Use appropriate cold-weather gear, up to and including Mustang-type bib coveralls or jacket/bib combinations. • Consider additional precautions if working near water in cold weather. • Have a dry change of clothing available.	• Review weather forecast prior to field work. • Monitor workers' physical conditions and PPE. • Monitor outside and water temperature versus worker activity and PPE.
	Rain or snow	• Wear appropriate PPE (rain gear). • Be aware of slip hazards, puddles, and electrical hazards when working in wet conditions. • If extremely cold conditions are forecast, consider additional precautions or postponing work activity.	• Review weather forecast prior to field work. • Inspect PPE daily prior to use. • Routinely inspect work area for deteriorating conditions.
	Sunshine	• Have sunscreen available for ultraviolet protection. • Have abundant water available to prevent dehydration. • Consider wearing wide-brimmed headwear and light-colored, lightweight, sun-blocking clothing.	• Ensure that sunscreen and water are available.
	Lightning	• Do not begin or continue work until lightning has ceased for at least 30 minutes. Disconnect and do not use or touch electronic equipment.	• Obtain weather forecast and updates as needed.
	High winds	• Wear goggles or safety glasses if dust or debris are visible.	• Review weather forecast prior to field work. • Ensure that goggles or safety glasses are available.

Job Safety Analysis

Decontamination Activities

Training Requirements:

- All personnel working on hazardous waste sites must receive appropriate training as required by 29 Code of Federal Regulations (CFR) 1910.120(e), including but not limited to initial 40-hour, 8-hour supervisor, and annual 8-hour refresher trainings.
- If boating is involved, and a professional captained vessel is not in use, boat operators must take the appropriate state or provincial boater safety courses.
- All assigned employees are required to familiarize themselves with the contents of this JSA before starting a work activity, and to review it with their supervisor during their daily safety meeting.

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Project Name: Lower Duwamish Waterway Remedial Design	**Project Number:** 180067-02.01	**JSA Number:** 004	**Issue Date:** May 29, 2020
Location: Seattle, Washington	**Contractor:** Anchor QEA, LLC and Windward Envronmental, LLC	**Analysis by:** K. Gross	**Analysis Date:** May 29, 2020
Work Operation: Anchor QEA motor vehicle operation	**Superintendent/Competent Person:** Vehicle Driver	**Revised by:** NA	**Revised Date:** NA
Required Personal Protective Equipment (PPE): • Wear seat belt at all times • Make sure that clothing will not interfere with driving		**Reviewed by:** M. Woltman	**Reviewed Date:** May 29, 2020
		Approved by: T. Shaner	**Approved Date:** May 29, 2020

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Anchor QEA motor vehicle operation	Unfamiliar with the vehicle	• Allow yourself some time to get familiar with an Anchor QEA vehicle, a rental vehicle, or one not used often. • Test the lights, windshield wipers, hazard lights, horn, parking brake, and other important functions. • Review the dashboard controls, steering radius, and overhead and side clearances. • Allow extra side, front, and back space around the vehicle while driving or parking an unfamiliar vehicle. • Adjust mirrors and the seat while the vehicle is in park. • Drive slowly in confined locations, as in a parking garage, parking lots, or industrial settings. Confirm adequate clearances by sight before turning or backing up in tight or unfamiliar locations. • Use a second person to be a spotter outside the vehicle if needed in tight spaces.	• Inspect fluid levels and air pressure in tires, adjust mirrors and seat positions appropriately, monitor the fuel level, and fill up when the fuel level is low

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
	Speed and braking	• Fasten and properly adjust the seat belt. • Obey all posted and designated speed limits. • Radar detectors are prohibited in all company-owned, leased, or rented vehicles. • Reduce travel speed during hazardous conditions (e.g., rain, fog, or snow). • Identify whether your vehicle has Anti-Lock Brakes (ABS). If it does, DO NOT pump the brakes to stop when the vehicle has begun to skid. Apply steady pressure to the brakes. If the vehicle does not have ABS, pump the brakes to stop during slippery conditions.	• Seatbelt • Identify designated speed limits • Determine if vehicle has ABS
Anchor QEA motor vehicle operation (continued)	Distance spacing	• Continually check your rear and side view mirrors. • Use the 3-second rule to keep a safe distance between vehicles. • Increase the 3-second rule as necessary during hazardous travel conditions. • Regularly scan the area you will be entering in the next 10 to 12 seconds. • Always leave yourself an “out” during travel. • When stopping, make sure that you leave enough distance between you and the car in front of you. You should be able to see the rear tires of the vehicle in front when stopped. • Obey the speed limit and traffic regulations. • When at a red light and it turns green, use the “delayed start” technique, by counting to three before you take your foot off the brake. • DO NOT TAILGATE. • Keep headlights (and running lights, if available) on for maximum visibility.	• Seatbelt
	Skids	• If the vehicle has begun to skid out of control, turn the steering wheel in the direction of the skid and re-adjust the wheel, as necessary. • Reduce speed during hazardous travel conditions. • Use 4-wheel drive, if available, when driving vehicles off-road, on steep inclines, or in muddy conditions. • Do not take vehicles off-road if they cannot be operated safely in such conditions.	• Seatbelt
	Blind spots	• Become familiar with any blind spots associated with your vehicle. • Adjust mirrors to give the maximum viewing area. • Use your directional devices to signal all turns and when changing lanes; check rear and side view mirror and glance over your shoulder to check that the lane is clear. • Avoid other driver’s blind spots; slow down and let the other vehicle pass. • If parked for an extended period and staying in the vehicle, be sure to inspect the area for changed conditions (e.g., a car that moved in behind you) before leaving.	• Seatbelt • Mirrors

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
	Backing	• Back into parking spaces upon arrival whenever possible. • Perform a 360-degree walk around the vehicle before backing to identify any new conditions or obstructions. • Use a spotter when backing whenever possible. • Understand hand signals. • Sound the horn prior to backing. • Check the rear and side view mirrors prior to backing. • Back slowly in areas of obstructed vision. • Anticipate others who may be backing out into your pathway and adjust accordingly.	• Seatbelt • Mirrors
Anchor QEA motor vehicle operation (continued)	Distractions (e.g., cell phones, reading maps or directions, eating)	• Do not engage in distracted driving—focus on operating the vehicle, and on your surroundings (e.g., road conditions and other drivers). • Obey state or local laws regarding cell phone use, at a minimum. • Certain clients prohibit cell phone use regardless of the state you are operating in—know your client's policy. • Use hands-free devices (not hand-held cellular phones) while driving. • Pull over to the side of the road when making a call or checking directions.	• Seatbelt • Hands-free devices connected and ready for use
	Accidents	• In the event of an accident, use the following procedures: – Stop, call for medical assistance, notify police, and complete an accident report and submit it to your supervisor. – Notify the Project Manager (PM) and Field Lead (FL). – Complete the appropriate incident investigation reports. – Contact Sara Weiskotten, Operations Liaison, at (857) 445-4987. – Contact Diana Reynolds, Insurance Liaison, at (302) 236-8403.	• Seatbelt
	Influenced by drugs or alcohol	• NEVER DRIVE UNDER THE INFLUENCE OF DRUGS OR ALCOHOL. • Keep in mind that the person in another vehicle may be under the influence of controlled substances, and be prepared for erratic or sudden driving changes on their part.	• Seatbelt
	Driver attitude	• Do not operate any vehicle when abnormally tired, temporarily disabled (i.e., injured), or under the influence of drugs or alcohol. • Keep an even temper when driving. Do not let the actions of others affect your attitude. • Do not allow yourself to become frustrated, rushed, distracted, or drowsy.	• Seatbelt

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
	Fatigue	• Stop and rest if fatigued. Exit the road and enter a safe area. Rest until fully refreshed. • Be aware that certain medications (such as cold or allergy medicines) may make you drowsy when driving a vehicle.	• Seatbelt
	Vehicle loading	• DO NOT OVERLOAD the vehicle. • Secure all equipment and supplies within the body of the vehicle using proper tie-downs. • Do not block side view mirrors with the load. • Do not transport U.S. Department of Transportation (DOT)-manifested hazardous materials. • Dispatch all equipment and personnel with proper forms and identification.	• Seatbelt
Anchor QEA motor vehicle operation (continued)	Equipment failure	• Perform daily inspections of your vehicle. • Maintain vehicle safety equipment (e.g., mirrors, alarms, horns, wipers, lights, and brakes). • Maintain the vehicle (e.g., tire pressure and fluid levels). • Any vehicle with mechanical defects that may endanger the safety of the driver, passengers, or the public shall not be used. • Ensure that appropriate safety equipment is in the vehicle. Safety equipment should include a spare tire, jack, first-aid kit, fire extinguisher, and flashlight. Flares and/or reflective triangles should be available in larger trucks. • Ensure that the proper documentation is in the vehicle. Documentation should include an operations manual for the vehicle, insurance card, vehicle registration, and accident forms.	• Inspect and maintain the vehicle

Training Requirements:

- All drivers are required to have a valid driver's license, and all vehicles must have appropriate state vehicle registration and inspection stickers. The use of hand-held wireless devices is prohibited while driving any vehicle for business use at any time, for personal use during business hours, and as defined by law.
- **If operating a vehicle or vehicle and trailer with a capacity greater than 10,000 pounds, U.S. Department of Transportation regulations may apply. Contact the PM prior to any travel in this configuration.**

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

- All assigned employees are required to read, familiarize themselves with the contents of this Job Safety Analysis, and sign the signature page before the operation of an Anchor QEA vehicle, and review it with their supervisor during their daily safety meeting.
- All assigned employees are required to enroll and complete the Smith System Virtual Driving training programs (*Distracted Driving* and *Small Vehicle Forward - Five Keys to Safe Driving*) prior to driving an Anchor QEA vehicle.

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Vehicle Operation Job Safety Analysis Acknowledgement Form

The Anchor QEA Motor Vehicle Operation Job Safety Analysis must be read, understood, and signed before the operation of any Anchor QEA vehicle. My signature below certifies that I have read and understand the procedures presented in the Anchor QEA Motor Vehicle Operation Job Safety Analysis and have completed the Smith System Virtual Driving *Distracted Driving* and *Small Vehicle Forward - Five Keys to Safe Driving* training programs.

Date	Name (print)	Signature

Job Safety Analysis

Anchor QEA Motor Vehicle Operation

Date	Name (print)	Signature

Appendix B
Field Forms

CLAM COLLECTION FORM

Project Name: _____ **Task No.:** _____

Date: _____ **Start/Stop Time:** _____

Weather: _____ **Clam Area ID:** _____

Crew: _____ **Target number:** _____

Time	Clam ID	Width (mm)	Comment
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			

TARGET SPECIES TALLY FORM

Project Name:	**Location:**
Date:	**Field Crew Initials:**
Collection Method:	**Target Species:**

COLLECTION TIME	SPECIMEN ID NO.	LENGTH (mm)	WEIGHT (g)	COMMENTS

NON-TARGET SPECIES TALLY FORM

Project Name: **Location:**

Date: **Field Crew Initials:**

Collection Method:

COLLECTION TIME	SPECIES	COUNT	COMMENTS

SPECIMEN LABEL

Windward Environmental LLC 200 W. Mercer St., Suite 401, Seattle, WA 98119 Tel: 206.378.1364 Fax: 206.973.3048	
Project/Task #:	Sampler:
Collection date:	Collection time:
Location:	Method:
Species:	
Specimen ID:	
Length (mm):	Weight (g)

COMPOSITE SAMPLE FORM

Project Name:		**Task #:**
Date composited:	**Species:**	**Area/Subarea:**
Composite mass:	**Tissue type:** (circle one) whole-body fillet remainder edible meat hepatopancreas	
Composite sample ID:		**Number of individuals:**

SPECIMEN ID	LENGTH (mm)	WEIGHT (g)	COMMENTS
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			

_____ of _____

CHAIN-OF-CUSTODY/TEST REQUEST FORM

Project/Client Name: _____ Ship to: _____

Project Number: _____ Attn: _____ Shipping Date: _____

Contact Name: _____ Shipper: _____ Airbill Number: _____

Sampled By: _____ Form filled out by: _____ Turnaround requested: _____

Sample Collection Date (m/d/y)	Time	Sample Identification	Volume of Sample / # of Containers	Matrix	Test(s) Requested (check test(s) required)										Comments / Instructions [Jar tag number(s)]
Total Number of Containers				Purchase Order / Statement of Work #											

1) **Released by:**	2) **Released by:**	3) **Released by:**	4) **Released by:**	5) **Released by:**
Company:	Company:	Company:	Company:	Company:
Date/Time:	Date/Time:	Date/Time:	Date/Time:	Date/Time:
Rec'd by:	**Rec'd by:**	**Rec'd by:**	**Rec'd by:**	**Rec'd by:**
Company:	Company:	Company:	Company:	Company:
Date/Time:	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Wind Ward environmental LLC

200 First Ave W
Suite 500
Seattle, WA 98119
Tel: (206) 378-1364
Fax: (206) 217-9343

To be completed by Laboratory upon sample receipt:

Date of receipt::	Laboratory W.O. #:
Condition upon receipt:	Time of receipt:
Cooler temperature:	Received by:

PROTOCOL MODIFICATION FORM

Project Name/Task Number: _____

Material to be Sampled: _____

Measurement Parameter: _____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):

Reason for Change in Field Procedure or Analysis Variation: _____

Variation from Field or Analytical Procedure: _____

Special Equipment, Materials or Personnel Required: _____

Initiator's Name: _____ Date: _____

Project Manager: _____ Date: _____

QA Manager: _____ Date: _____

Appendix C. Analytical Data Quality Indicators

TABLES

Table C-1
Data Quality Indicators

Parameter	Units	Precision	Accuracy		Completeness
			CRM/LCS[1,2]	Spiked Samples[1]	
Fish, Crab, and Clam tissue					
PCB congeners	ng/kg ww	± 50%	40–145%	15–150%	90%
Inorganic Arsenic	mg/kg ww	± 25%	na	75–125%	90%
cPAHs[3]	µg/kg ww	± 35%	50-150%	50-150%	90%
Dioxins/furans congeners[4]	ng/kg ww	± 50%	70–130%	17–197%	90%
SVOCs[5]	µg/kg ww	± 35%	na	20–130%	90%
TBT	µg/kg ww	± 35%	na	20–130%	90%
Vanadium	mg/kg ww	± 30%	na	75–125%	90%
Organochlorine pesticides[6]	µg/kg ww	± 50%	10–150%	30–150%	90%
Lipids	% ww	± 30%	na	na	90%
Percent solids	% ww	± 20%	na	na	90%
Passive Sampler					
PCB congeners	pg/sample	± 20%	60–135%	15–145%	90%

Notes:
1. Values listed are performance-based limits provided by the laboratories.
2. An LCS may be used to assess accuracy when a CRM is unavailable.
3. cPAH components include benzo(a)anthracene, benzo(a)pyrene, total benzofluoranthenes, chrysene, dibenzo(a,h)anthracene, and indeno(1,2,3-cd)pyrene.
4. Dioxin/furan congeners include 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD, 1,2,3,4,7,8-HxCDD, 1,2,3,6,7,8-HxCDD, 1,2,3,7,8,9-HxCDD, 1,2,3,4,6,7,8-HpCDD, OCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDF, 2,3,4,7,8-PeCDF, 1,2,3,4,7,8-HxCDF, 1,2,3,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF, 2,3,4,6,7,8-HxCDF, 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, and OCDF.
5. Target SVOCs include bis(2-ethylhexyl) phthalate, PCP, carbazole, and hexachlorobenzene.
6. Target organochlorine pesticides include aldrin, alpha-BHC, beta-BHC, total chlordane, total DDTs, dieldrin, gamma-BHC, heptaochlor, and heptachlor epoxide.
BHC: benzene hexachloride
cPAH: carcinogenic polycyclic aromatic hydrocarbon
CRM/LCS: certified reference material/laboratory control sample
DDT: dichlorodiphenyltrichloroethane
HpCDD: heptachlorodibenzo-*p*-dioxin
HpCDF: heptachlorodibenzofuran
HxCDD: hexachlorodibenzo-*p*-dioxin
HxCDF: hexachlorodibenzofuran
na: not applicable
OCDD: octachlorodibenzo-*p*-dioxin
OCDF: octachlorodibenzofuran
RL: reporting limit
PCB: polychlorinated biphenyl
PCP: pentachlorophenol
PeCDD: pentachlorodibenzo-*p*-dioxin
PeCDF: pentachlorodibenzofuran

SVOC: semivolatile organic compound
TBD: to be determined
TBT: tributyltin
TCDD: tetrachlorodibenzo-*p*-dioxin
TCDF: tetrachlorodibenzofuran
ww: wet weight

The laboratory method detection limit (MDL) and reporting limit (RL) values for each analytical method are compared to their respective target tissue level (TTL) values in Table C-2. All of the analytical methods are sufficiently sensitive.

Table C-2
Summary of Fish, Crab, and Clam Tissue Analytes, Methods, and RL Goals for Each Analyte

Analyte	Method	Laboratory MDL	RL	TTL (ROD Table 21)
ROD Tissue COCs				
PCB congeners (sum) (µg/kg ww)	EPA 1668A	0.004[1]	0.012[2]	12 (benthic fish, fillet) 1.8 (pelagic fish, whole body) 1.1 (crab, edible meat) 9.1 (crab, whole body) 0.42 (clam, whole body)
cPAH (µg TEQ/kg ww)	GC/HRMS Isotope Dilution	0.1[3]	0.2[4]	1.5 (clam, whole body)[5]
Dioxins/furans (ng TEQ/kg ww)	EPA 1613B	0.17[6]	0.5[7]	0.35 (benthic fish, whole body) 0.53 (crab, edible meat) 2.0 (crab, whole body) 0.71 (clam, whole body)
Inorganic arsenic (mg/kg ww)	IC-ICP-CRC-MS	0.004	0.010	0.09 (clam, whole body)
Metals and Organometals				
TBT (µg/kg ww)	EPA 8270E-SIM	0.450	3.86	na
Vanadium (mg/kg ww)	EPA 6020B	na[8]	0.004	na
SVOCs				
BEHP (µg/kg ww)	EPA 8270E	na[8]	50.0[9]	na
Carbazole (µg/kg ww)	EPA 8270E	na[8]	20.0[9]	na
Hexachlorobenzene (µg/kg ww)	EPA 8270E	na[8]	20.0[9]	na
PCP (µg/kg ww)	EPA 8270E	na[8]	100[9]	na
Organochlorine Pesticides				
Aldrin (µg/kg ww)	EPA 8270D/ 1699 Mod	0.22[10]	1.0[9]	na
alpha-BHC (µg/kg ww)	EPA 8270D/ 1699 Mod	0.26[10]	1.0[9]	na

Analyte	Method	Laboratory MDL	RL	TTL (ROD Table 21)
beta-BHC (µg/kg ww)	EPA 8270D/ 1699 Mod	0.4[10]	1.0[9]	na
Dieldrin (µg/kg ww)	EPA 8270D/ 1699 Mod	0.22[10]	1.0[9]	na
gamma-BHC (µg/kg ww)	EPA 8270D/ 1699 Mod	0.17[10]	1.0[9]	na
Heptaclor (µg/kg ww)	EPA 8270D/ 1699 Mod	0.09[10]	1.0[9]	na
Heptachlor epoxide (µg/kg ww)	EPA 8270D/ 1699 Mod	0.061[10]	1.0[9]	na
Total chlordane[11] (µg/kg ww)	EPA 8270D/ 1699 Mod	0.77[10]	2.5[9]	na
Total DDTs[12] (µg/kg ww)	EPA 8270D/ 1699 Mod	0.46[10]	2.5[9]	na
Conventionals				
Lipids (%)	Bligh and Dyer (1959) (mod)	0.040	na	na
Percent solids (%)	PSEP (1997)	0.010	na	na

Notes:
1. The PCB congener MDL is based on the laboratory-estimated detection limit from Cape Fear and represents the value for an individual PCB congener. Individual congener EDLs are listed in Table C7. MDL is a sample-specific detection limit. The value provided is an estimate, and the sample-specific values will vary based on sample mass and the analytical conditions at the time of analysis.
2. The PCB congener LOQ is based on the laboratory minimum calibration level from Cape Fear and represents the value for an individual PCB congener. Individual congener LOQs are listed in Table C7. LOQ is Cape Fear's lowest calibration limit. Detected values below the LOQ will be J-qualified. The reported LOQ will be adjusted based on the sample mass of each sample.
3. The MDL cPAH TEQ value was calculated using one-half the MDL for each of the cPAH compounds and appropriate TEF values (California EPA 2009).
4. The RL cPAH TEQ value was calculated using one-half the RL for each of the cPAH compounds and appropriate TEF values (California EPA 2009).
5. Clam TTL for cPAHs based on the EPA ESD (EPA 2021).
6. The dioxin/furan MDL is based on the laboratory-estimated detection limit from Cape Fear for 2,3,7,8-TCDD and the mammal TEF value (Van den Berg et al. 2006) for this congener. Individual congener EDLs are listed in Table C6. The value provided is an estimate, and the sample-specific values will vary based on sample mass and the analytical conditions at the time of analysis.
7. The dioxin/furan LOQ is based on the laboratory minimum calibration level from Cape Fear for 2,3,7,8-TCDD and the mammal TEF value (Van den Berg et al. 2006) for this congener. Individual congener LOQs are listed in Table C6. LOQ is Cape Fear's lowest calibration limit. Detected values below the LOQ will be J-qualified. The reported LOQ will be adjusted based on the sample mass of each sample.
8. SW846 no longer requires MDL values. ARL does not report to the MDL for these analytes due to tissue matrix interferences at concentrations below the reporting limit.
9. RL values are consistent with the LLOQ values required under EPA SW846.
10. SW846 no longer requires MDL values. The laboratories have the option to use these values to assess sensitivity for EPA 8000 series methods. ALS-Kelso has continued to maintain MDL studies for OC pesticides.
11. The components of total chlordane include alpha-Chlordane, cis-Nonachlor, gamma-Chlordane, oxychlordane, and trans-Nonachlor. Individual component MDLs and RLs are listed in Table C5.
12.The components of total DDT include 2,4'-DDD, 2,4'-DDE, 2,4'-DDT, 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT. Individual component MDLs and RLs are listed in Table C5.
BEHP: bis(2-ethylhexyl) phthalate
BHC: benzene hexachloride
cPAH: carcinogenic polycyclic aromatic hydrocarbon
DDD: dichlorodiphenyldichloroethane
DDE: dichlorodiphenyldichloroethylene
DDT: dichlorodiphenyltrichloroethane
EPA: US Environmental Protection Agency
ESD: explanation of significant difference
LOQ: limit of quantitation

MDL: method detection limit
na: not available
PCB: polychlorinated biphenyl
PCP: pentachlorophenol
PSEP: Puget Sound Estuary Program
RL: reporting limit
ROD: Record of Decision
SIM: selected ion monitoring
SVOC: semivolatile organic compound
TBT: tributyltin
TCDD: tetrachlorodibenzo-p-dioxin
TEF: toxic equivalency factor
TEQ: toxic equivalent
TTL: target tissue level
ww: wet weight

Standard tissue mass requirements are specified to meet RLs for each particular analytical method. Table 4-9 summarizes the tissue mass needed for each sample type. The masses listed in the table include the mass required for quality control (QC) samples. Mass required for standard analyses is 120 g for crab and clam tissue and 80 g for fish tissue.

Table C-3
Tissue Mass Required per Sample Type

Analyte	Tissue Mass (g) for Crabs[1,2]	Tissue Mass (g) For Fish	Tissue Mass (g) For Clams
PCB congeners[3] and dioxins/furans	40	40	40
Inorganic arsenic	3.5	3.5	3.5
cPAHs	80	na	80
selected SVOCs[3]	37.5	37.5	37.5
TBT[3]	15	15	15
Vanadium[3]	7.5	7.5	7.5
Selected organochlorine pesticides[3]	20	20	20
Lipids and percent solids	Taken from SVOC extract[4]	Taken from SVOC extract[4]	Taken from SVOC extract[4]
Total Mass	203.5	123.5	203.5

Notes:
1. Separate tissue mass will be collected for edible meat and for hepatopancreas.
2. In the event that there is not sufficient hepatopancreas mass to perform all QC analyses on a single hepatopancreas composite, QC for some analytes may be performed on separate hepatopancreas composites.
3. Selected SVOCs, TBT, vanadium, and selected organochlorine pesticides will be analyzed in a subset of samples, as described in the QAPP.
4. Solvent extraction with acetone/DCM for SVOCs.
cPAH: carcinogenic polycyclic aromatic hydrocarbon
DCM: dichloromethane
na: not available
PCB: polychlorinated biphenyl
QC: quality control

SVOC: semivolatile organic compound
TBT: tributyltin

Table C-4
Laboratory Quality Control Sample Analysis Summary

Analysis Type	Initial Calibration	Initial Calibration Verification (second source)	Continuing Calibration Verification	CRM or LCS[1]	Laboratory Replicates	MSs	MSDs	Method Blanks	Surrogate Spikes
Fish, Crab, and Clam tissue									
PCB congeners	Prior to analysis	After initial calibration	Every 12 hours	1 per batch or SDG[2]	1 per batch or SDG	na	na	1 per prep batch	Each sample
Inorganic arsenic	Prior to analysis	After initial calibration	Every 10 samples	na	1 per 10 samples or SDG	1 per 10 samples or SDG	1 per 10 samples or SDG	1 per prep batch	na
cPAHs	Prior to analysis	After initial calibration	Every 10–20 analyses or 12 hours	1 per batch or SDG[3]	1 per batch or SDG	na	na	1 per prep batch	Each sample
Dioxins/furans congeners	Prior to analysis	After initial calibration	Every 12 hours	1 per batch or SDG[2]	1 per batch or SDG	na	na	1 per prep batch	Each sample
SVOCs	Prior to analysis	After initial calibration	Every 10–20 analyses or 12 hours	1 per batch or SDG[4]	na	1 per batch or SDG	1 per batch or SDG	1 per prep batch	Each sample
TBT	Prior to analysis	After initial calibration	Every 10 samples	1 per batch or SDG[4]	na	1 per batch or SDG	1 per batch or SDG	1 per prep batch	Each sample
Vanadium	Prior to analysis	After initial calibration	Every 10 samples	1 per batch or SDG[4]	1 per batch or SDG	1 per batch or SDG	na	1 per prep batch	na
Organochlorine pesticides	Prior to analysis	After initial calibration	Every 10–20 analyses or 12 hours	1 per batch or SDG[3]	na	1 per batch or SDG	1 per batch or SDG	1 per prep batch	Each sample
Percent solids	na	na	na	na	1 per batch or SDG	na	na	na	na

Analysis Type	Initial Calibration	Initial Calibration Verification (second source)	Continuing Calibration Verification	CRM or LCS[1]	Laboratory Replicates	MSs	MSDs	Method Blanks	Surrogate Spikes
Lipids	na	na	na	na	1 per batch or SDG	na	na	na	na
Passive Sampler									
PCB congeners	Prior to analysis	After initial calibration	Every 12 hours	1 per prep batch[2]	na	na	na	1 per prep batch	Each sample

Notes:
A batch is a group of samples of the same matrix analyzed or prepared at the same time, not to exceed 20 samples.
1. An LCS may be used to assess accuracy when an CRM is unavailable.
2. EDF_2526 will be used to assess accuracy for PCB congeners and dioxins/furans.
3. SRM 1974c will be used to assess accuracy for cPAHs and organochlorine pesticides.
4. There is no CRM available so an LCS will be used to assess accuracy.
cPAH: carcinogenic polycyclic aromatic hydrocarbon
CRM: certified reference material
LCS: laboratory control sample
MS: matrix spike
MSD: matrix spike duplicate
na: not applicable or not available
PCB: polychlorinated biphenyl
SDG: sample delivery group
SRM: standard reference material
SVOC: semivolatile organic compound
TBT: tributyltin

Table C-5
Methods and RL Goals for cPAHs, Metals, TBT, Organochlorine Pesticides, SVOCs, and Conventionals

Analyte	Method	Unit	MDL	RL
cPAHs				
Benzo(a)anthracene[1]	GC/HRMS isotope dilution	µg/kg ww	0.01	0.2
Benzo(a)pyrene[1]	GC/HRMS isotope dilution	µg/kg ww	0.03	0.2
Total benzofluoranthenes	GC/HRMS isotope dilution	µg/kg ww	0.04	0.2
Chrysene[1]	GC/HRMS isotope dilution	µg/kg ww	0.02	0.2
Dibenzo(a,h)anthracene[1]	GC/HRMS isotope dilution	µg/kg ww	0.07	0.2
Indeno(1,2,3-cd)pyrene[1]	GC/HRMS isotope dilution	µg/kg ww	0.05	0.2
Metals				
Inorganic arsenic	IC-ICP-CRC-MS	mg/kg ww	0.004	0.010
Vanadium	EPA 6020B	mg/kg ww	na[2]	0.004
TBT	EPA 8270D-SIM	µg/kg ww	0.450	3.86
Organochlorine Pesticides				
Aldrin	EPA 8270D/1699 Mod	µg/kg dw	0.22[3]	1.0[4]
alpha-BHC	EPA 8270D/1699 Mod	µg/kg dw	0.26[3]	1.0[4]
beta-BHC	EPA 8270D/1699 Mod	µg/kg dw	0.4[3]	1.0[4]
Carbazole	EPA 8270D/1699 Mod	µg/kg dw	7.37[3]	20.0[4]
Dieldrin	EPA 8270D/1699 Mod	µg/kg dw	0.22[3]	1.0[4]
gamma-BHC	EPA 8270D/1699 Mod	µg/kg dw	0.17[3]	1.0[4]
Heptachlor	EPA 8270D/1699 Mod	µg/kg dw	0.09[3]	1.0[4]
Heptachlor epoxide	EPA 8270D/1699 Mod	µg/kg dw	0.061[3]	1.0[4]
alpha-Chlordane[5]	EPA 8270D/1699 Mod	µg/kg dw	0.12[3]	1.0[4]

Analyte	Method	Unit	MDL	RL
cis-Nonachlor[5]	EPA 8270D/1699 Mod	µg/kg dw	0.13[3]	1.0[4]
gamma-Chlordane[5]	EPA 8270D/1699 Mod	µg/kg dw	0.13[3]	1.0[4]
Oxychlordane[5]	EPA 8270D/1699 Mod	µg/kg dw	0.77[3]	2.5[4]
trans-Nonachlor[5]	EPA 8270D/1699 Mod	µg/kg dw	0.094[3]	1.0[4]
2,4'-DDD[6]	EPA 8270D/1699 Mod	µg/kg dw	0.31[3]	2.5[4]
2,4'-DDE[6]	EPA 8270D/1699 Mod	µg/kg dw	0.42[3]	2.5[4]
2,4'-DDT[6]	EPA 8270D/1699 Mod	µg/kg dw	0.46[3]	1.0[4]
4,4'-DDD[6]	EPA 8270D/1699 Mod	µg/kg dw	0.13[3]	1.0[4]
4,4'-DDE[6]	EPA 8270D/1699 Mod	µg/kg dw	0.7[3]	2.5[4]
4,4'-DDT[6]	EPA 8270D/1699 Mod	µg/kg dw	0.35[3]	1.0[4]
SVOCs				
BEHP	EPA 8270E	µg/kg ww	na[2]	50.0[4]
Carbazole	EPA 8270E	µg/kg ww	na[2]	20.0[4]
PCP	EPA 8270E	µg/kg ww	na[2]	100[4]
Hexachlorobenzene	EPA 8270E	µg/kg ww	na[2]	20.0[4]
Conventionals				
Total solids	PSEP 1986	% dw	na	0.040
Lipids	Bligh and Dyer (mod)	% ww	na	0.010

Notes:
1. Components of cPAH sum.
2. SW846 no longer requires MDL values. ARL does not report to the MDL for these analytes due to tissue matrix interferences at concentrations below the reporting limit.
3 . SW846 no longer requires MDL values. The laboratories have the option to use these values to assess sensitivity for EPA 8000 series methods. ALS-Kelso has continued to maintain MDL studies for OC pesticides.
4. RL values are consistent with the LLOQ values required under EPA SW846.
5. Components chlordane sum.
6. Components of total DDx sum.
BEHP: bis(2-ethylhexyl) phthalate
BHC: benzene hexachloride

cPAH: carcinogenic polycyclic aromatic hydrocarbon
DDD: dichlorodiphenyldichloroethane
DDE: dichlorodiphenyldichloroethylene
DDT: dichlorodiphenyltrichloroethane
EPA: US Environmental Protection Agency
MDL: method detection limit
na: not available
PCB: polychlorinated biphenyl
PCP: pentachlorophenol
PSEP - Puget Sound Estuary Program
RL: reporting limit
SIM: selective ion monitoring
SVOC: semivolatile organic compounds
TBT: tributyltin
total DDx: DDT isomers (2,4′-DDD, 4,4′-DDD, 2,4′-DDE, 4,4′-DDE, 2,4′-DDT and 4,4′-DDT)
ww: wet weight

Table C-6
RL Goals for Dioxins/Furan Congeners

Analyte	EPA Method 1613B	
	Tissue (ng/kg ww) Based on 10-g sample	
	MDL[1]	LOQ[2]
2,3,7,8-TCDD	0.333	1.00
1,2,3,7,8-PeCDD	1.67	5.00
1,2,3,4,7,8-HxCDD	1.67	5.00
1,2,3,6,7,8-HxCDD	1.67	5.00
1,2,3,7,8,9-HxCDD	1.67	5.00
1,2,3,4,6,7,8-HpCDD	1.67	5.00
OCDD	3.33	10.0
2,3,7,8-TCDF	0.333	1.00
1,2,3,7,8-PeCDF	1.67	5.00
2,3,4,7,8-PeCDF	1.67	5.00
1,2,3,4,7,8-HxCDF	1.67	5.00
1,2,3,6,7,8-HxCDF	1.67	5.00
1,2,3,7,8,9-HxCDF	1.67	5.00
2,3,4,6,7,8-HxCDF	1.67	5.00
1,2,3,4,6,7,8-HpCDF	1.67	5.00
1,2,3,4,7,8,9-HpCDF	1.67	5.00
OCDF	3.33	10.0

Notes:
1. MDL is a sample-specific DL. The value provided here is an estimate, and the sample-specific values will vary based on sample mass and the analytical conditions at the time of analysis
2. LOQ is Cape Fear's lowest calibration limit. Detected values below the LOQ are J-qualified. The reported LOQ will be adjusted based on the sample mass of each sample.
DL: detection limit
EPA: US Environmental Protection Agency
HpCDD: heptachlorodibenzo-p-dioxin
HpCDF: heptachlorodibenzofuran
HxCDD: hexachlorodibenzo-p-dioxin
HxCDF: hexachlorodibenzofuran
J: estimated concentration
MDL: method detection limit
LOQ: limit of quantitation
OCDD: octachlorodibenzo-p-dioxin
OCDF: octachlorodibenzofuran
PeCDD: pentachlorodibenzo-p-dioxin
PeCDF: pentachlorodibenzofuran
RL: reporting limit
TCDD: tetrachlorodibenzo-p-dioxin

TCDF: tetrachlorodibenzofuran
ww: wet weight

Table C-7
RL Goals for PCB Congeners in Tissues

Analyte	EPA Method 1668A	
	Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-1	1.00	2.00
PCB-2	1.00	2.00
PCB-3	1.00	2.00
PCB-4	1.00	2.00
PCB-5	1.00	2.00
PCB-6	1.00	2.00
PCB-7	1.00	2.00
PCB-8	1.00	2.00
PCB-9	1.00	2.00
PCB-10	1.00	2.00
PCB-11	2.00	10.0
PCB-12/13	1.00	4.00
PCB-14	1.00	2.00
PCB-15	1.00	2.00
PCB-16	1.00	2.00
PCB-17	0.667	2.00
PCB-18/30	0.667	4.00
PCB-19	1.33	2.00
PCB-20/28	1.33	4.00
PCB-21/33	0.667	4.00
PCB-22	1.33	2.00
PCB-23	1.33	2.00
PCB-24	0.667	2.00
PCB-25	0.667	2.00
PCB-26/29	0.667	4.00
PCB-27	0.667	2.00
PCB-31	0.667	2.00

Analyte	EPA Method 1668A	
	Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-32	0.667	2.00
PCB-34	0.667	2.00
PCB-35	0.667	2.00
PCB-36	0.667	2.00
PCB-37	0.667	2.00
PCB-38	0.667	2.00
PCB-39	0.667	2.00
PCB-40/41/71	1.33	4.00
PCB-42	1.00	2.00
PCB-43	1.33	4.00
PCB-44/47/65	3.33	6.00
PCB-45/51	1.33	4.00
PCB-46	0.667	2.00
PCB-48	0.667	2.00
PCB-49/69	1.33	4.00
PCB-50/53	1.33	4.00
PCB-52	1.33	4.00
PCB-54	0.667	2.00
PCB-55	1.00	2.00
PCB-56	0.667	2.00
PCB-57	1.00	2.00
PCB-58	1.00	2.00
PCB-59/62/75	2.00	6.00
PCB-60	0.667	2.00
PCB-61/70/74/76	2.67	8.00
PCB-63	0.667	2.00
PCB-64	1.00	2.00
PCB-66	1.00	2.00
PCB-67	1.00	2.00
PCB-68	0.667	2.00
PCB-72	0.667	2.00
PCB-73	1.00	2.00

Analyte	EPA Method 1668A	
	Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-77	0.667	2.00
PCB-78	0.667	2.00
PCB-79	0.667	2.00
PCB-80	0.667	2.00
PCB-81	0.667	2.00
PCB-82	1.00	2.00
PCB-83	1.00	2.00
PCB-84	1.00	2.00
PCB-85/116/117	2.00	6.00
PCB-86/87/97/109/119/125	4.00	12.00
PCB-88/91	133	4.00
PCB-89	0.667	2.00
PCB-90/101/113	2.00	6.00
PCB-92	1.00	2.00
PCB-93/100	2.00	4.00
PCB-94	0.667	2.00
PCB-95	1.33	4.00
PCB-96	0.667	2.00
PCB-98/102	1.33	4.00
PCB 99	1.33	4.00
PCB-103	0.667	2.00
PCB-104	0.667	2.00
PCB-105	1.00	2.00
PCB-106	1.00	2.00
PCB-108/124	1.33	4.00
PCB-107	0.667	2.00
PCB-108/124	1.33	4.00
PCB-110/115	2.67	8.00
PCB-111	0.667	2.00
PCB-112	1.33	4.00
PCB-114	0.667	2.00
PCB-118	1.33	4.0

Analyte	EPA Method 1668A	
	Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-120	0.667	2.00
PCB-121	0.667	2.00
PCB-122	0.667	2.00
PCB-123	0.667	2.00
PCB-126	0.667	2.00
PCB-127	0.667	2.00
PCB-128/166	1.33	4.00
PCB-129/138/160/163	2.67	6.00
PCB-130	1.00	2.00
PCB-131	0.667	4.00
PCB-132	1.00	2.00
PCB-133	0.667	2.00
PCB-134/143	1.33	4.00
PCB-135/151	1.33	4.00
PCB-136	0.667	2.00
PCB-137	1.33	4.00
PCB-139/140	1.33	4.00
PCB-141	1.33	4.00
PCB-142	0.667	2.00
PCB-143	1.33	4.00
PCB-144	0.667	2.00
PCB-145	0.667	2.00
PCB-146	1.00	2.00
PCB-147/149	2.00	4.00
PCB-148	0.667	2.00
PCB-150	1.00	2.00
PCB-152	0.667	2.00
PCB-153/168	2.00	4.00
PCB-155	0.667	2.00
PCB-156/157	1.33	4.00
PCB-158	0.667	2.00
PCB-159	0.667	2.00

Analyte	EPA Method 1668A	
	Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-160	0.667	2.00
PCB-161	1.00	2.00
PCB-162	1.00	2.00
PCB-164	0.667	2.00
PCB-165	0.667	2.00
PCB-167	0.667	2.00
PCB-169	1.00	2.00
PCB-170	0.667	2.00
PCB-171/173	1.33	4.00
PCB-172	1.00	2.00
PCB-174	1.33	4.00
PCB-175	1.00	2.00
PCB-176	0.667	2.00
PCB-177	1.00	2.00
PCB-178	1.33	4.00
PCB-179	0.667	2.00
PCB-180/193	2.00	4.00
PCB-181	1.00	2.00
PCB-182	0.667	2.00
PCB-183/185	1.33	4.00
PCB-184	0.667	2.00
PCB-186	0.667	2.00
PCB-187	1.00	2.00
PCB-188	0.667	2.00
PCB-189	0.667	2.00
PCB-190	1.00	2.00
PCB-191	0.667	2.00
PCB-192	0.667	2.00
PCB-194	0.667	2.00
PCB-195	1.00	2.00
PCB-196	0.667	2.00
PCB-197/200	1.33	4.00

Analyte	EPA Method 1668A Tissue (ng/kg ww) Based on 10-g Sample	
	MDL[1]	LOQ[2]
PCB-198/199	1.33	4.00
PCB-201	0.667	2.00
PCB-202	1.00	2.00
PCB-203	1.33	4.00
PCB-204	1.00	2.00
PCB-205	0.667	2.00
PCB-206	1.00	2.00
PCB-207	0.667	2.00
PCB-208	0.667	2.00
PCB-209	0.667	2.00

Notes:
1. MDL is a sample-specific DL. The value provided here is an estimate, and the sample-specific values will vary based on sample mass and the analytical conditions at the time of analysis.
2. LOQ is Cape Fear's lowest calibration limit. Detected values below the lowest calibration limit are J-qualified. The reported lowest calibration limit will be adjusted based on the sample mass of each sample.
DL: detection limit
EPA: US Environmental Protection Agency
J: estimated concentration
LOQ: limit of quantification
PCB: polychlorinated biphenyl
MDL: method detection limit
ww: wet weight

Table C-8
RL Goals for PCB Congeners – Passive Samplers

Analyte	EPA Method 1668C Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-1	1.0	4.0
PCB-2	1.0	4.0
PCB-3	1.0	4.0
PCB-4	2.0	4.0
PCB-5	2.0	4.0
PCB-6	2.0	4.0
PCB-7	2.0	4.0
PCB-8	2.0	4.0
PCB-9	2.0	4.0

Analyte	EPA Method 1668C	
	Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-10	2.0	4.0
PCB-11	2.0	4.0
PCB-12/13	2.0	4.0
PCB-14	2.0	4.0
PCB-15	2.0	4.0
PCB-16	1.0	4.0
PCB-17	1.0	4.0
PCB-19	1.0	4.0
PCB-21/33	1.0	4.0
PCB-22	1.0	4.0
PCB-23	1.0	4.0
PCB-24	1.0	4.0
PCB-25	1.0	4.0
PCB-26/29	1.0	4.0
PCB-27	1.0	4.0
PCB-28/20	1.0	4.0
PCB-30/18	1.0	4.0
PCB-31	1.0	4.0
PCB-32	1.0	4.0
PCB-34	1.0	4.0
PCB-35	1.0	4.0
PCB-36	1.0	4.0
PCB-37	1.0	4.0
PCB-38	1.0	4.0
PCB-39	1.0	4.0
PCB-41/40/71	1.0	4.0
PCB-42	1.0	4.0
PCB-43	1.0	4.0
PCB-44/47/65	1.0	4.0
PCB-45/51	1.0	4.0
PCB-46	1.0	4.0
PCB-48	1.0	4.0
PCB-50/53	1.0	4.0

Analyte	EPA Method 1668C	
	Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-52	1.0	4.0
PCB-54	1.0	4.0
PCB-55	1.0	4.0
PCB-56	1.0	4.0
PCB-57	1.0	4.0
PCB-58	1.0	4.0
PCB-59/62/75	1.0	4.0
PCB-60	1.0	4.0
PCB-61/70/74/76	1.0	4.0
PCB-63	1.0	4.0
PCB-64	1.0	4.0
PCB-66	1.0	4.0
PCB-67	1.0	4.0
PCB-68	1.0	4.0
PCB-69/49	1.0	4.0
PCB-72	1.0	4.0
PCB-73	1.0	4.0
PCB-77	1.0	4.0
PCB-78	1.0	4.0
PCB-79	1.0	4.0
PCB-80	1.0	4.0
PCB-81	1.0	4.0
PCB-82	1.0	4.0
PCB-83/99	1.0	4.0
PCB-84	1.0	4.0
PCB-88/91	1.0	4.0
PCB-89	1.0	4.0
PCB-92	1.0	4.0
PCB-94	1.0	4.0
PCB-95/100/93/102/98	1.0	4.0
PCB-96	1.0	4.0
PCB-103	1.0	4.0
PCB-104	1.0	4.0

Analyte	EPA Method 1668C	
	Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-105	1.0	4.0
PCB-106	1.0	4.0
PCB-108/124	1.0	4.0
PCB-109/119/86/97/125/87	1.0	4.0
PCB-107	1.0	4.0
PCB-110/115	1.0	4.0
PCB-111	1.0	4.0
PCB-112	1.0	4.0
PCB-113/90/101	1.0	4.0
PCB-114	1.0	4.0
PCB-117/116/85	1.0	4.0
PCB-118	1.0	4.0
PCB-120	1.0	4.0
PCB-121	1.0	4.0
PCB-122	1.0	4.0
PCB-123	1.0	4.0
PCB-126	1.0	4.0
PCB-127	1.0	4.0
PCB-128/166	1.0	4.0
PCB-130	1.0	4.0
PCB-131	1.0	4.0
PCB-132	1.0	4.0
PCB-133	1.0	4.0
PCB-134/143	1.0	4.0
PCB-136	1.0	4.0
PCB-137	1.0	4.0
PCB-138/163/129/160	1.0	4.0
PCB-139/140	1.0	4.0
PCB-141	1.0	4.0
PCB-142	1.0	4.0
PCB-144	1.0	4.0
PCB-145	1.0	4.0
PCB-146	1.0	4.0

Analyte	EPA Method 1668C	
	Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-147/149	1.0	4.0
PCB-148	1.0	4.0
PCB-150	1.0	4.0
PCB-151/135/154	1.0	4.0
PCB-152	1.0	4.0
PCB-153/168	1.0	4.0
PCB-155	1.0	4.0
PCB-156/157	1.0	8.0
PCB-158	1.0	4.0
PCB-159	1.0	4.0
PCB-161	1.0	4.0
PCB-162	1.0	4.0
PCB-164	1.0	4.0
PCB-165	1.0	4.0
PCB-167	1.0	4.0
PCB-169	1.0	4.0
PCB-170	1.0	4.0
PCB-171/173	1.0	4.0
PCB-172	1.0	4.0
PCB-174	1.0	4.0
PCB-175	1.0	4.0
PCB-176	1.0	4.0
PCB-177	1.0	4.0
PCB-178	1.0	4.0
PCB-179	1.0	4.0
PCB-180/193	1.0	4.0
PCB-181	1.0	4.0
PCB-182	1.0	4.0
PCB-183/185	1.0	4.0
PCB-184	1.0	4.0
PCB-186	1.0	4.0
PCB-187	1.0	4.0
PCB-188	1.0	4.0

Analyte	EPA Method 1668C	
	Passive Sampling (pg/sample) Based on 1 PE Strip	
	EDL[1]	LMCL[2]
PCB-189	1.0	4.0
PCB-190	1.0	4.0
PCB-191	1.0	4.0
PCB-192	1.0	4.0
PCB-194	1.0	4.0
PCB-195	1.0	4.0
PCB-196	1.0	4.0
PCB-197/200	1.0	4.0
PCB-198/199	1.0	4.0
PCB-201	1.0	4.0
PCB-202	1.0	4.0
PCB-203	1.0	4.0
PCB-204	1.0	4.0
PCB-205	1.0	4.0
PCB-206	1.0	4.0
PCB-207	1.0	4.0
PCB-208	1.0	4.0
PCB-209	1.0	4.0

Notes:
1. EDL is a sample-specific DL. The value provided here is an estimate, and the sample-specific values will vary based on sample mass and the analytical conditions at the time of analysis.
2. LMCL is Axys's lowest calibration limit. Detected values below the LMCL are J-qualified. The reported LMCL will be adjusted based on the volume or mass of each sample.
Axys: Axys Analytical Services Ltd.
DL: detection limit
EDL: estimated detection limit
EPA: US Environmental Protection Agency
J: estimated concentration
LMCL: lower method calibration limit
PCB: polychlorinated biphenyl
PE: polyethylene

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Appendix D
Standard Operating Procedures

Appendix D.1
Passive Sampler Standard Operating Procedure

GUIDANCE DOCUMENT

Passive PE Sampling in Support of In Situ Remediation of Contaminated Sediments: Standard Operating Procedure for PED Preparation

ESTCP Project ER-200915

December 2012

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14. ABSTRACT

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used in the deployment and retrieval of polyethylene devices (PEDs) in sediment-pore water environments for the sampling of hydrophobic organic compounds (HOCs) such as polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs). The overall objective of PED sampling is to determine the horizontal and vertical distributions of HOCs in pore waters of bed sediments. This SOP does not discuss deployment of PEDs in atmospheric or aqueous systems. This SOP should be used in conjunction with companion SOPs for Preparation of Polyethylene (PE) Media and Extraction Procedures for PE Media.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
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Reset

Standard Operating Procedure for the Preparation of Polyethylene (PE) and Polyethylene Devices (PEDs) Used for Passive Sampling

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for preparing and handling polyethylene (PE) films that will be cut into strips and used in polyethylene devices (PEDs) to passively sample hydrophobic organic compounds (HOCs) in environmental media.

1.2 This method generates PE that can be deployed within PEDs for passive sampling of HOCs in atmospheric, aqueous, or sediment-porewater systems.

1.3 PE that is prepared by this method is suitable for laboratory or *in situ* field deployment.

2.0 SUMMARY OF METHOD

2.1 A known mass of low density polyethylene (LDPE) sheet, usually gram quantities, is cleaned by sequentially extracting with methylene chloride, methanol, and ultrapure water in a closed glass vessel.

2.2 Clean PE is equilibrated with performance reference compounds (PRCs) dissolved in water or methanol-water (see Appendix 1 for possible PRCs).

2.3 Prepared PE is stored in contaminant-free, sealed, glass vessels.

2.4 Shortly before deployment, the PE is cut into strips and either placed in aluminum mesh bags for water sampling water or aluminum frames for sediment sampling. PEDs are transported to the field wrapped in clean aluminum foil.

2.5 In the field, the PE is exposed to the environmental medium of concern. HOCs in the medium diffuse into the PE, while PRCs diffuse out.

3.0 INTERFERENCES

3.1 PE is susceptible to contamination from atmospheric vapors and contact with surfaces (e.g., worker hands), so it must remain in clean sealed vessels until deployment.

4.0 APPARATUS AND MATERIALS

4.1 Extraction vessels: 1-L glass bottles or screw capped jars (foil-lined lids).

4.2 Storage vessels: bottles with glass stoppers or amber jars (foil-lined lids).

4.3 Bottle/jar tumbler, shaker table, bottle roller, or equivalent.

4.4 Low density polyethylene (LDPE): commercial grade, large sheet at 25μm (1 mil) or 51μm (2 mil) thickness. The thickness is chosen to be strong enough to withstand stresses during deployment (e.g., insertion into sediment), but thin enough to exchange a significant fraction (e.g., >20%) of its PRCs during the deployment time to be used.

4.5 Food grade aluminum foil (solvent cleaned and/or combusted to remove any organic residue from foil production)

4.6 Stainless steel forceps

4.7 Teflon (or similar non-contaminating material) cutting board

5.0 REAGENTS

5.1 Methylene chloride, CH_2Cl_2, pesticide grade or equivalent

5.2 Methanol, CH_3OH, pesticide grade or equivalent

5.3 Organic-free reagent water (as defined in SW-846 Chapter 1)

5.4 Research grade PRCs certified >98+% pure.

Note: Specific standard materials, concentrations, solvents, and solvent purity requirements will be determined based upon that target HOCs of concern for the particular application

6.0 PRESERVATION AND HANDLING

6.1 Clean PE should be stored in clean sealed glass vessels.

6.2 Until deployment, prepared PE (PE loaded with PRCs) is stored in sealed glass containers with a few mL of organic-free reagent water added to maintain 100% relative humidity within the storage vessels (minimizing sorptive losses of PRCs to glass vessel walls).

6.3 Laboratory and field personnel should wear nitrile or latex gloves whenever handling clean PE.

6.4 Methylene chloride-rinsed, stainless steel forceps and scissors are used when manipulation of clean PE is required.

6.5 Methylene chloride-rinsed, aluminum foil is used to cover any surface that clean PE may encounter.

7.0 PROCEDURE

7.1 Polyethylene Cleaning Procedure: LDPE is purchased from hardware/painting stores in large sheets ('dropcloth or plastic tarp' material) with thickness of 25μm (1 mil) or 51μm (2 mil), depending on the user's need for strength (choose thicker) and desire to use short deployment times (used thinner). The sheet is cut into strips sized for environment and frames to be used. An organic solvent cleaning sequence is then used to prepare the PE. This process ensures that extractable oligomers, plasticizers, and contaminating organic chemicals are removed from the PE prior to use. All extractions are performed sequentially in the same container.

7.1.1 Methylene chloride is placed into the extraction vessel, and the PE strips are immersed in the container for 24 hours to enable time for diffusive transfers out of the PE. The initial methylene chloride extract is discarded and a second methylene chloride extraction is performed for 24 hours. The second methylene chloride extract is discarded and replaced by methanol in order to remove methylene chloride from the PE. Methanol immersion is also done for 24 hours. The initial methanol extract is discarded and followed by a second methanol soak for 24 hours. Finally, the second methanol extract is discarded and the PE undergoes three 24-hour soaks with organic-free reagent water (within the same

extraction vessel) to remove residual methanol from the PE.

7.1.2 The cleaned PE is stored in organic-free reagent water in the extraction vessel until further processing.

7.2 Polyethylene Preparation with Performance Recovery Compounds (PRCs)***:*** PRCs are loaded into the clean PE, prior to its field deployment, by utilizing either aqueous (Fernandez et al. 2009) or 80:20 methanol:water equilibrations (Booij et al., 2002). Depending on the hydrophobic organic compounds of interest, PRCs should be chosen which mimic mass transfer phenomena governing exchanges during field deployments. It is important to avoid adding PRCs that the analytical laboratory already uses as surrogate or injection standards. PRC loading is performed by placed the PE in pre-cleaned glass vessels containing known PRC solutions made up in organic-free reagent water with or without pesticide-grade methanol. The PE user should estimate the expected accumulation of target compounds in the passive sampler and seek to load with similar levels of PRCs to facilitate the eventual chemical analyses. Sufficient PRC equilibration time during this PE preparation step is necessary to ensure uniform PE loading across the entire PE thickness; hence thicker PE sheet is more robust for field use, but takes longer to load with PRCs.

7.2.1 Isotopically labeled compounds are useful internal standards when Gas Chromatography-Mass Spectrometry (GCMS) is the method of separation and detection. For example, deuterated polycyclic aromatic hydrocarbons (PAHs) and C13-labeled PCBs are effective methodological standards for PE passive sampling. One subset of compounds, distributed across the range of PAHs to be assessed (e.g., d10-phenanthrene, d10-pyrene, and d12-chrysene), should be used as PRCs, while another set (e.g., d10-anthracene, d10-fluoranthene, and d12-benz(a)anthracene) is used as surrogate (recovery) compounds during later analysis of field-deployed PE. Finally, compounds such as d10-acenaphthene, d14-*m*-terphenyl, and d12-perylene can be used as injection standards. Similar sets of labeled compounds should be used for other compound classes (see Appendix 1). Note: if PE samples are eventually to be analyzed at a contract laboratory, PRC choices must be made so as not to conflict with recovery and injection standards used by that laboratory.

7.2.2 As subsequent analysis (e.g., GCMS) is best achieved with both PRCs and target HOCs present at like concentrations in the PE extracts, the optimal concentration level of the PRC loaded into the PE is dependent on the environment in which the PE is to be deployed. For example, if a target HOC is expected to occur in the water or pore water near 1 ng/L levels, one can use that compound's LDPE-water partition coefficient (e.g., Fernandez et al., 2009; Lohmann, 2012) to estimate the expected levels in the PE after deployment:

$$\text{Concentration in PE (ng/kg)} \sim K_{LDPE\text{-}water} * \text{concentration in (pore)water (ng/L)}$$

So if the $K_{LDPE\text{-}water}$ for the target HOC of interest is 10^5 (L/kg), then the concentration of the target HOC in the PE will approach 100 ug/kg. Based on this estimate, the PRCs are loaded into the PE at similar concentrations. Appendix 2 shows a typical calculation used to design a PRC-containing MeOH:H_2O solution of PCBs suited for causing an 0.82 g strip of PE to acquire about 100 ug of each PRC per kg of PE (equivalent to 100 ng/g PE).

7.2.3 Aqueous PRC Loading: A solvent-cleaned and dried glass container is filled with ultrapure water that has been spiked with known concentrations of PRCs (e.g., using calculations like those shown in Appendix 2). A known mass of pre-cleaned PE is then added and weighted to insure complete PE submersion. The vessel is agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-water phase ratio. For PAHs and PCBs, use at least 30 days to insure homogeneous distributions of the PRCs throughout the entire thickness of the PE film unless faster equilibration has been confirmed. Confirmation can be done by time course measures of PRC concentrations in the PE or by showing that concentrations of PRCs are the same for films of different thicknesses, but the same masses. Generally, PE is stored in the PRC solution until it is to be deployed.

7.2.4 Methanol-Aided PRC Loading: A solvent-cleaned and dried glass container is filled with an 80:20 mixture of pesticide grade methanol and ultrapure water that has been spiked with known concentrations of PRCs (e.g., see calculations in Appendix 2). A known mass of pre-cleaned PE is then added and weighted to insure complete submersion. The vessel should be agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-solvent phase ratio, but typically this step is completed within 7 days since methanol swells the PE and thereby speeds PRC diffusion into the polymer sheet (Booij et al., 2002). Generally, the PE is stored in the PRC solution until shortly before it is to be deployed. Before deployment, the PRC-loaded PE is rinsed with ultrapure water, and then it is soaked in ultrapure water for 24 h to remove methanol from the PE. This methanol leaching step is repeated twice to insure complete methanol removal.

7.3 PED Assembly

7.3.1 PEDs can be pre-assembled with prepared PE strips up to a few days prior to deployment depending on the target compounds of interest.

7.3.2 FOR WATER SAMPLING WITH PE IN A STAINLESS STEEL MESH BAG. Since PE that is openly exposed in the water column has been observed to be eaten by aquatic organisms, the PE must be protected by deploying it in a mesh bag.

7.3.2.1 Cut rectangles from the mesh that are larger than the piece of PE to be deployed. Clean the mesh with methylene chloride, methanol, and water.

7.3.2.2 Wearing nitrile gloves, and using solvent-rinsed stainless steel forceps, lay a piece of the mesh on a clean surface such as an aluminum-foil covered lab bench. Remove the PE strip from its container and lay it on top of a stainless steel mesh. Place the second mesh on top. The two meshes are sealed together by folding the edges over on one another, and then sewing them together with nylon fishing line. Grommets can be added to the upper corners to facilitate mesh labeling and attachments in the field.

7.3.3 FOR SEDIMENT BED SAMPLING WITH PE IN AN ALUMINUM SHEET METAL FRAME. In order to insert the PE strips into a sediment bed, the PE must be carried by an aluminum frame (Figure 1).

7.3.3.1. Aluminum sheet metal is cut into two complementary pieces that can be bolted together such that a piece of PE sheet is held in place. After cutting, these pieces of aluminum must be washed with organic solvents (e.g., methylene chloride and methanol) and then rinsed with water.

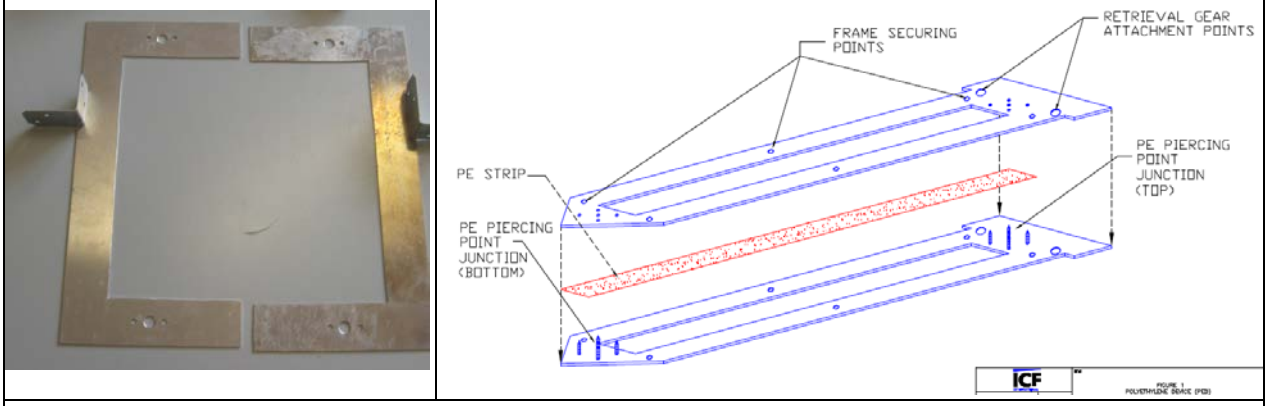

Figure 1.
(left panel) Aluminum sheet cut into two "C-shaped" pieces allowing the investigator to mount and hold ~25 cm strips of PE an open window when the two pieces are overlapped and bolted together.
(right panel) Drawing of two aluminum sheet pieces cut so as to sandwich a strip of PE and expose about 50 cm of length.

7.3.3.2 Wearing nitrile gloves, lay a piece of the aluminum frame containing the PE piercing points (sheet metal screws, see Figure 1), sharp side up, on a sheet of solvent-rinsed aluminum foil.

7.3.3.3 Using solvent-rinsed stainless steel forceps, remove the PE strip from its container and lay the strip lengthwise across both sets of PE piercing point junctions. PE strips should have been sized to fit the frame with a little extra length, allowing the investigator to cut a small strip of PE from one end to serve as sample for PRC concentration measures before the sampler is deployed. At one end of the PED frame, gently push the remainder of the PE strip onto the PE piercing points so all points penetrate the PE strip. Gently pull the other end of the PE strip over the adjacent PE piercing points, keeping the PE strip taut, and push that end of the PE strip into the PE piercing points. The tautness of the PE strip should have as minimal deflection as possible between the two PE piercing point junctions, but not too tight so that movement of the PE causes it to rip or tear. Place the other PED frame over the PED frame containing the PE strip so that each of the PE piercing point junctions meet and both PED frames are flush against each other. Secure the two frames together using the appropriate hardware (stainless steel machine screws, locking washers, and cap nuts).

7.3.3.4 Wrap the entire PED frame in solvent-rinsed aluminum foil to prevent exposure during transport and field preparation activities.

7.4 PE and PED Storage and Shipment*:*

7.4.1 Prepared PEDs in their foil envelops may be stored a few days at ambient temperature prior to deployment. Freezing or excessive heat should be avoided to minimize the likelihood of changing the polymer crystallinity. It is recommended that PEDs be hand carried or shipped in a timely fashion (Overnight or Next Day if possible) to minimize chances sampler contamination or damage.

7.4.2 If PE is to be shipped to another location for PED assembly, it is recommended that the PE strips are individually sealed in pre-cleaned glass vials that contain a little water. Freeze shipping should be avoided, but cold (refrigeration temperature) packing may be necessary depending on time of season and individual laboratory handling/quality control procedures.

8.0 QUALITY CONTROL

8.1 PRC Loading Validation: At least six representative samples of prepared PE should be collected (e.g., 6 x 10 mg pieces), extracted, and analyzed prior to field deployment to validate that the PRC concentrations are consistent with their intended loadings and these standards have uniform concentrations in a batch of PE.

8.2 Target HOC Blanks: Subsamples of prepared PE, commensurate in size with the planned environmental PE samples (e.g., 10 cm wide by 5 cm long by 25 um thick and therefore weighing about 120 mg), should be be collected, extracted, and analyzed prior to field deployment to demonstrate that other substances have not contaminated the PE which would contribute to interfering background for the target HOCs.

9.0 METHOD PERFORMANCE

9.1 PRC data, obtained from PE samples collected from >six parts of the prepared PE, should be consistent within about 10% (i.e., 100 x standard deviation / mean).

9.2 Target HOC concentrations should be undetectable in the prepared PE (e.g., < 1 ng/g PE assuming 100 mg PE subsamples).

10.0 REFERENCES

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Appendix 1 Suggested Performance Reference Compounds (PRCs), Surrogate Compounds (Recovery Standards), and Injection Standards.

A. PRCs, suitable for polycyclic aromatic hydrocarbon (PAH) determinations when Gas Chromatography-Mass Spectrometry (GCMS) is the preferred method of detection, include, but are not restricted to, deuterated PAH compounds. One subset should be used as PRCs, while reserving others for use as surrogate (recovery) compounds. Still other compounds such as terphenyl can be used as injection standards.

Targets: PAHs	**Method: GCMS**	**Detection Limit ~ 100 pg / 100 mg PE**	
PRCs	d10-phenanthrene	d10-pyrene	d12-chrysene
Surrogates	d10-anthracene	d10-fluoranthene	d12-benz(a)anthracene
Injection Standards	d10-acenaphthene	d14-*m*-terphenyl	d12-perylene

B. PRCs and surrogate compounds suitable for polychlorinated biphenyl (PCB) determinations when GCMS is the preferred method of detection include, but are not restricted to, ^{13}C-labeled or deuterated PCB congeners. One subset, for example including a tri-, tetra-, penta-, hexa-, and heptachloro-biphenyl, can be used as PRCs while reserving different tri-, tetra-, penta-, hexa-, and heptachloro-biphenyl congeners to serve as surrogate compounds. Still other compounds such as deuterated PAHs or rare PCBs (not contained in Aroclor/Clophan mixtures such as: PCB-39, PCB-55, PCB-104, PCB-150 and PCB-188) can be used as injection standards.

Targets: PCBs	**Method: GCMS**		**Detection Limit ~ 100 pg / 100 mg PE**			
PRCs	^{13}C PCB-28	^{13}C PCB-52	^{13}C PCB-101	^{13}C PCB-153	^{13}C PCB-180	
Surrogates	^{13}C PCB-19	d_6 PCB-77	^{13}C PCB-105	^{13}C PCB-167	^{13}C PCB-170	^{13}C PCB-194
Injection Standards	d17-39	d22-104	d34-55	d40-150	d52-188	

C. When analyzing for organochlorine pesticides such as DDT using GCMS, ^{13}C labeled compounds can serve as PRCs and surrogate standards. Since DDT has been seen to degrade to form DDE or DDD in certain situations, one should use the 4,4'- isomer of DDT and the 2,4'-isomers of DDE and DDD as PRCs to allow appearance of ^{13}C-labelled 4,4'-DDE of 4,4'-DDD to be interpreted as arising from reaction of the DDT PRC during the deployment. Deuterated or ^{13}C labeled PCBs can be used as surrogate (recovery) and injection standards.

Targets: DDTs	**Method: GCMS**	**Detection Limit ~ 200 pg / 100 mg PE**	
PRCs	^{13}C 2,4'-DDE	^{13}C 2,4'-DDD	^{13}C 4,4'-DDT
Surrogates	^{13}C-PCB111	^{13}C-PCB153	^{13}C 2,4'-DDT
Injection Standards	d6 PCB 77	^{13}C PCB 105	^{13}C PCB 167

Appendix 2. Example of spreadsheet used to design solution needed to impregnate PE with Performance Reference Compounds (PRCs) for PCB sampling. The leftmost section uses data from Booij et al. (2002) to establish a correlation between log K(polyethylene-80:20 MeOH:H2O) values and log K_{ow} values from Hawker and Connell (1988). With this relationship, the second section shows is use to estimate the PE-MeOH:H2O partition coefficients for PRCs of interest. Using these partition coefficients and a user-chosen mass of PE to prepare (here 0.82 g), the third section allows the user to find the PRC spiking solution concentration needed to obtain any desired initial PE concentration (here set to be 100 ng each PRC per g PE); for example, for congener 52, one needs to have 11 ng/mL of the initial 80:20 MeOH:H_2O solution to end up with about 100 ng/g PE. Finally, the right-most section uses the polyethylene-water partition coefficients (from Lohmann 2012) to check the importance of PRC losses from the PE when the MeOH is leached out in three successive steps after PRC loading. Successive calculations are described in the text below.

Example spreadsheet calculation for spiking PCBs into LDPE with 80:20 methanol-water solutions.

	Training data for estimation of Kpe-meoh:H2O		13C-labelled PRCs		use correlation to estimate	fraction in PE for PE mass (g)	0.82042	Solution concentration needed in ng/mL in order to get 100 ng/g PE			fraction in PE after each water soak to remove MeOH		
PCB congener	log Kpe-meoh:water (ref 1)	log Kow (ref 2)	congener	log Kow (ref 2)	log Kpe-meoh:water(80:20)	with VMeOH:water (mL)	125	ng/mL MeOH:H2O	congener	estim log Kpe-w log Kpew = 1.14*log Kow-1.14 (ref 3)	1st leach using	2nd leach 1000	3rd leach mL water
4	0.20	4.65	**52**	5.84	0.97	0.058		11.29	**52**	5.52	0.9966	0.9932	0.9898
29	1.05	5.6	**101**	6.38	1.26	0.107		6.15	**101**	6.13	0.9990	0.9980	0.9971
155	1.29	6.41	**153**	6.92	1.55	0.188		3.49	**153**	6.75	0.9997	0.9994	0.9991
204	1.67	7.3	**180**	7.36	1.78	0.284		2.31	**180**	7.25	0.9999	0.9998	0.9997
			28	5.67	0.88	0.048		13.75	**28**	5.32	0.9950	0.9900	0.9850
			47	5.85	0.98	0.059		11.16	**47**	5.53	0.9967	0.9934	0.9901
			111	6.76	1.46	0.160		4.10	**111**	6.57	0.9996	0.9992	0.9988
			153	6.92	1.55	0.188		3.49	**153**	6.75	0.9997	0.9994	0.9991
			178	7.14	1.66	0.233		2.82	**178**	7.00	0.9998	0.9997	0.9995

use to find following correlaltion:

log Kpe-mw(80:20) = 0.532 (+/- 0.094) * log Kow(Hawker) - 2.133 (+/- 0.572)

N = 4, R2 = 0.94, S.E. 0.18

PE mass

number of strips	1
PE density (g/cm3)	0.95
PE thickenss (cm)	0.00254 for 1 mil sheet
PE length (cm)	68
PE width (cm)	5
length*width*thickness *number of strips*density	
mass of PE (g)	**0.82**

references

1. Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.
2. Hawker DW and Connell DW. 1988. Environ. Sci. Technol. 22: 382-387.
3. Lohmann, R. Environ. Sci. & Technol.; 2012, 46, 606–618.

Step 1: find/estimate PE-spiking solvent partition coefficients for PRCs in solvents of interest. Here 80:20 MeOH:H_2O values from Booij et al. (2002) are used to develop a LFER with K_{ow} values from the literature (Hawker and Connell, 1988); this relation is then used to estimate $K_{pe\text{-}meoh:h2o}$ values for other PCB congeners.

Step 2: choose the size of PE needed for the sampling exercise (here a single 1 mil-thick strip of 5 cm width and 68 cm length) and solve for the PE mass (here 0.82 g). Also choose a vessel which is large enough in volume to fit the PE inside without extensive PE-PE surface contact, but small enough so that unacceptably expensive masses of the labeled PRCs are not used (here 125 mL ground glass stopped flask). For this PE mass and solution volume, use the PE-solution partition coefficients from step 1 to solve for the fractions of each PRC that will be in the PE at equilibrium using:

$$\text{fraction in PE} = 1 - (1 / (1+\text{Mass}_{pe}*K_{pe\text{-}solution}/\text{Volume}_{solution}))$$

(e.g., 5.8% for congener #52)

Step 3. solve for spiking solution concentrations of PRCs that result in desired PRC loadings in the PE (here 100 ng/g_{PE}) using:

$$C_{initial\ spiking\ solution} = C_{desired\ in\ PE} * \text{Mass}_{pe} / \text{fraction in PE} / \text{Volume}_{solution}$$

(e.g., here find need about 11.3 ng congener #52 per mL to achieve 100 ng/g PE; this is concentration of the spiking solution that the investigator must make up to prepare PE for subsequent sampling at sites where it is expected that the (pore)water will cause the PE to accumulate about 10 to 100 ng of target PCBs/g_{PE}).

Step 4. PE is stored in the PRC loading solution until shortly before passive sampling use.

Step 5. if spiking solutions that contain organic cosolvents like MeOH were used, this MeOH must be leached out of the PE before it can be used for passive sampling. To insure that MeOH leaching will not substantially change PRC loading, calculate whether substantial fractions of the PRCs will be lost in subsequent steps required to leach the co-solvent from the PE. Since the leaching steps involve use of H_2O, use the PE-water partition coefficients; for PCBs, these are derived from a LFER found in the review by Lohmann (2012). With these values, we can solve for the fractional losses of individual PRCs to the leach water (assumes negligible MeOH builds up in the leach water) contained in 1000 mL ground glass stoppered flasks, using:

$$\text{fraction remaining in PE after a single leach step} = 1 - (1 / (1+K_{pe\text{-}H2O}*\text{Mass}_{pe} / \text{Volume}_{H2O}))$$

(e.g., in this case for congener #52, one finds 99.66% of the PRC remains in the PE after the first leach. Two additional leaches lower this to 99.32% and 98.98%, respectively. More hydrophobic congeners are leached even less.)

Appendix D.2
Alpha Analytical Laboratory Tissue Homogenization Standard Operating Procedure

Tissue Preparation and Homogenization

References: This standard operating procedure (SOP) is a performance-based method. This SOP describes the procedure as developed by Alpha Analytical.

1. Scope and Application

Matrices: This method is applicable to the preparation and homogenization of plant and animal tissue including: mammals (mice or shrew etc.), fish (whole body and fillets), mollusks (mussels or clams, etc.), crustaceans (lobster or shrimp, etc.), reptiles and amphibians (frogs or turtles, etc.) macro invertebrates (benthic worms, eels, insects and other biota), and vegetation (coastal and wetland grasses)

Definitions: Refer to Alpha Analytical Quality Manual.

This preparation and homogenization procedure may be used prior to the extraction or digestion of the matrices listed above, for the ultimate detection of organic and inorganic analytes. Because this procedure is performance based, it should only be used for compounds where studies have assessed the precision, accuracy, and sensitivity of the technique relative to the project specific goals.

This method is intended to describe the preparation and homogenization procedures to be followed prior to the extraction, digestion and/or clean up of sample extracts or digestates. This procedure uses a variety of cutting and grinding equipment for size reduction, compositing and homogenization. See Section 7 for Equipment and Materials. This method is applicable to the matrices described above. The final determinative analytical methods contain the lists of potential target compounds. Applicable extraction, digestion, and cleanup methods include:

- *Microwave Assisted Acid Digestion of Sediments, Soils, Tissues and Waters* (2150),
- *Gel Permeation Chromatography* (2167),
- *Sulfuric Acid Cleanup - Method 3665A* (2169),
- *Microscale Solvent Extraction (2172),*
- *Tissue Extraction (2264),*
- *Silica Cleanup (2170),*
- *Alumina Column Cleanup (2260).*
- *Solid Phase Extractions (23528)*

Data derived from the analysis of tissue samples is generally used to determine if human health, and/or ecological risk criteria have been exceeded.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts.

2. Summary of Method

This method describes the tissue processing and homogenization procedures to be used prior to the extraction/digestion and analysis of the sample. Samples are best processed when partially frozen. Samples may be re-frozen after processing pending extraction or digestion.

Fish tissue samples (whole bodies, carcass or fillets) are weighed and the weights are recorded following project specifications. Measurements may be taken as needed depending upon the project specifications. The fish may be processed with the skin on or off, depending upon the project specifications. If fillets are to be removed and processed separately, this is generally done after the removal of the skin, however fillets can be processed with the skin on if requested. If compositing is required, the identified samples for composite are filleted or skinned prior to compositing homogenization. The carcass of the fish (after removal of the fillet) may be maintained for separate homogenization and analysis if requested.

Mammals such as mice, shrew or other rodents, should be prepared in a glove box if one is available, due to the potential health hazards associated with mammal tissue. All project specific sample preparation (weighing, skinning, compositing and homogenization) should be performed in the glove box. Waste from the processing must be containerized and treated with bleach before disposal. The outside surfaces of the sample containers must be disinfected before removal from the glove box, or any other work area.

Mollusks, crustaceans and other like invertebrates are measured and weighed prior to processing. Mollusks must be removed from their shells before processing. Due to the low weight of a single mollusk, crustacean or invertebrate, these sample types are generally composited with others of the same species and/or sampling area prior to homogenization. Gender determination may need to be performed with larger crustaceans such as lobsters. This is done prior to any processing and recorded. Additionally, lobsters are usually dissected, and the edible meat (tail and claw) is removed for homogenization. Certain internal organs such as the hepatopancreas may need to be processed separately. If crabs are being processed, the legs, claws and body cavity are generally homogenized together.

Reptiles and amphibians are generally processed as whole body samples. Depending upon the size, the specimen may need to be cut into small pieces and processed in part, then re-combined as a single sample. Due to the thickness of the skin of most reptiles, such as frogs, it is recommended that these be processed without the skin. If the skin must be processed, ensure that the grinder or processor blades are sharpened before use. The blades may need to be re-sharpened between every few samples as needed. Turtles must be removed from the shell prior to processing by digging out the head and legs, and as much of the body as feasible.

Macro invertebrates such as worms, eels, insects or benthic biota are generally processed as whole body samples. Depending upon the size, the specimen may need to be cut into small pieces and processed in part, then re-combined as a single sample. Due to the low weight of a single invertebrate, these sample types are generally composited with others of the same species and/or sampling area prior to homogenization.

Plants are rinsed prior to processing to remove soil, silt, small insects or other debris. Depending upon the size of the plant and the leaves, the sample may be processed mechanically, or may have to be cut into small pieces by hand. Plants can be processed either wet or dry, depending upon project specifications.

After tissue processing, organic samples will be extracted and the extracts cleaned if needed, then analyzed by the determinative analytical procedure. Inorganic digestates do not require further clean up and will only undergo analysis by the determinative analytical procedure.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Not applicable to this method. Refer to the analytical method SOPs.

4. Interferences

Solvents, reagents, processing equipment and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the Alpha Analytical SOP *Reagent, Solvent and Standard Control* (1816) and *Laboratory Glassware Cleaning* (1753), for additional details.

Field Blanks are recommended to ensure that the field sample packing materials are not a potential source of contamination. This can be done by pouring contaminate free water over the sample collection material and collecting the water in an appropriate container with preservative as needed (*i.e.,* 1L glass amber bottle for organic and a 500mL polyethylene bottle with 1:1 HNO_3 preservative for metals).

Equipment used to process samples for *organic* analyses should be made of stainless steel, Teflon, ceramic, or PTFE. Tissue should be removed with clean, high-quality, corrosion-resistant stainless steel, ceramic or titanium instruments, knives and blades. Homogenates must be stored in borosilicate glass, quartz, or PTFE containers with PTFE-lined lids.

Many interferences can be removed by sample cleanup. The organic cleanup methods performed by Alpha Analytical include those listed in Section 1. Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest. For example, sulfuric acid cleanup is not applicable to samples requiring pesticide analysis because this rigorous cleanup will destroy the majority of pesticides.

Soapy residue may result in basic conditions on glassware and may cause degradation of the pesticides Aldrin and Heptachlor, some organophosphorous pesticides, and can cause metals instrument interferences. All glassware must be rinsed thoroughly with deionized water and solvents/nitric acid to remove soapy residue. See the Alpha Analytical SOP (1753) *Laboratory Glassware Cleaning*, for additional details.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

As guidance, a minimum of 50 grams of sample must be collected for organic analyses, and 5 grams for metals analyses, in a glass jar with a Teflon or PTFE-lined screw cap. The amount of sample needed, will depend upon the project DQOs, such as reporting limits and the need for MS/MSD and/or duplicate analyses. Extra sample must be collected, if possible, to allow the laboratory adequate sample volume in case re-preparation and re-analysis is needed. Large whole individual fish, fillets, or vegetation may be wrapped in plastic or aluminum foil depending upon the requested analyses. (See Section 4 or additional details about allowable materials). Large crustaceans, reptiles or amphibians may be individually packed in well-labeled Styrofoam coolers. When sampling for PFAS, protocols which exclude products known to contain PFAS will be followed to avoid cross-contamination of samples.

6.2 Sample Preservation

It is recommended that samples are preserved by freezing them with dry ice at $\leq$ -20°C. If samples are not shipped frozen, they will be stored in freezers at Alpha Analytical upon arrival, and until processing. The samples must remain frozen and maintained at $\leq$ -20°C $\pm$ 10°C until processing. After processing, individual sample homogenates must also be stored at $\leq$ -20°C $\pm$10°C until extraction/digestion and analysis.

6.3 Sample Shipping

Refer to Section 6.2.

6.4 Sample Handling

Sample processing and extraction/digestion hold times are suspended by freezing the sample. Hold time monitoring is resumed when samples are removed from freezers for processing and then returned to freezers pending extraction/digestion. Movement of samples into and out of freezers is tracked through LIMS. The organic hold time is 14 days from sample collection to extraction, and 40 days from extraction to analysis. The metals hold time is six months from sample collection to digestion and analysis. If mercury is to be determined, the hold time is 28 days from sample collection to digestion and analysis.

7. Equipment and Supplies

7.1 Cutting board: Made of either glass, Teflon or polypropylene

7.2 Food processor: Black & Decker with titanium cutting blade (small).

7.3 Dissection Tools: Tools may include the following utensils: knives/blades (ceramic, stainless steel, or titanium), stainless steel picks, spatulas (stainless steel or Teflon-coated stainless steel), stainless steel scissors/snips, stainless steel tweezers. (Refer to Section 4.0 for interferences and/or contamination associated with different materials.)

7.4 Pliers: Stainless Steel

7.5 Balances: Analytical Balance with precision to 0.0001g; Top loading balance with precision to 0.01g; Top loading balance with precision to 0.2g.

7.6 Grinding unit: Omni-GLH, electric, custom fitted with stainless steel or titanium interior saw tooth probes (10mm, 20mm, 45mm), or equivalent.

7.7 Tissuemizer: *Janke & Kunkel* IKA - Labortechnik Ultra Turrax T25, stainless steel

7.8 Grinder: LEM electric meat grinder, stainless steel (or equivalent)

7.9 Multi-hazard glove box: Labconco

7.10 Bench liner material

7.11 Latex Gloves – Powder Free

7.12 Glass weighing dish/jar, wax paper, aluminum foil, plastic wrap.

7.13 Camera

7.14 Ruler

7.15 Paper towels: Kim Wipes

8. Reagents and Standards

Use reagent grade or trace metals grade chemicals for all reagents. Deionized (DI) water or reagent water is ASTM Type II laboratory reagent grade water. Other grades may be used.

All reagents are stored at room temperature in flammable cabinets, unless otherwise noted. All reagents expire upon manufacturer's expiration date or one year from date of opening, whichever is sooner.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see ALPHA ANALYTICAL SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.2 Methanol: ACS approved, Purge & Trap grade, see ALPHA ANALYTICAL SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see ALPHA ANALYTICAL SOP Reagent. *Solvent and Standard Control* (1816) for additional details regarding solvent purity

8.4 Acetone: ACS approved, HPLC grade, see ALPHA ANALYTICAL SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.5 Nitric acid 50% (1:1): Add 500 mL concentrated HNO_3 to 400 mL of reagent water and dilute to 1 liter in an appropriate beaker or flask. For 25% HNO_3 solution: add 250 mL of concentrated HNO_3 to 400 mL of reagent water and dilute to 1 liter in an appropriate beaker or flask. Store in a corrosion-resistant cabinet.

8.6 10% Bleach solution: Add 100 mL of commercial bleach to 500 mL of reagent water and dilute to 1 liter in an appropriate beaker or flask. Prepare fresh each day of use.

8.7 Alconox cleaning solution. No special storage requirements. No expiration requirements.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

The following quality control samples mayor may not be included with each processing batch. If not included in the tissue processing steps, they must be included in the extraction/digestion batches that follow processing, or as needed, depending upon project specifications.

9.1 Blank(s)

9.1.1 Rinseate Blank/Equipment Blank or Process Blank

Rinseate/Equipment or Process blanks are generated using contaminate-free reagent (DI) water to rinse all processing equipment after completion of the cleaning procedure (see Section 10.1). The volume of water used will be based on project-specific volume requirements for requested analyses.

All processing equipment is rinsed with pre-determined volume of reagent water (DI) into a collection vessel. All rinse water is transferred from collection vessels to larger glass carboy.

Homogenizer/Generator probes are immersed in a pre-determined volume of DI water. The Homogenizing Unit will be turned on and the probe will process the DI water for a pre-determined time, based on project specifications. The DI water will then be transferred into a larger glass carboy and combined with DI from other processing equipment.

After all rinseates are collected into the glass carboy (or appropriate container), mix the DI water using a large glass stirring rod or by swirling the DI water. Transfer the water into the appropriate pre-prepared sample containers.

9.1.2 Method Blank

Not applicable to this method. Refer to analytical SOPs.

9.2 Laboratory Control Sample (LCS)

Not applicable to this method. Refer to analytical SOPs

9.3 Initial Calibration Verification (ICV)

Not applicable to this method. Refer to analytical SOPs.

9.4 Continuing Calibration Verification (CCV)

Not applicable to this method. Refer to analytical SOPs.

9.5 Matrix Spike

Not applicable to this method. Refer to analytical SOPs

9.6 Laboratory Duplicate

Not applicable to this method. Refer to analytical SOPs

9.7 Method-specific Quality Control Samples

Not applicable to this method. Refer to analytical SOPs

9.8 Method Sequence

Not applicable.

10. Procedure

The procedures described below are general cleaning and pre-processing procedures that are to be followed regardless of the type of tissue being processed. Samples are prioritized by the Department Manager or Team Leader based on hold time and client due date. All weights, measurement and other project required observations are recorded on the Tissue Prep Log sheets.

10.1 Equipment Set-up

10.1.1 Wash all utensils, generator probes, sample processor (blades, blade post, cup and lid) and the cutting board(s) with an alconox solution and a sponge. Rinse thoroughly with tap water, then with DI water and allow to dry. Equipment may be dried with a paper towel, if needed.

10.1.2 After drying the equipment, rinse all utensils, processor parts and surfaces with Acetone followed by a rinse with methylene chloride.

10.1.2.1 For *metal analyses only*, rinse all plastic and ceramic utensils with 25% HNO3 followed by another rinse with DI water. Rinse processor parts and surfaces with the alconox solution, followed by a tap water and a DI water rinse. Any metal or titanium surfaces must not come into contact with the 25% HNO3 solution as this may strip some metal alloys from these surfaces and introduce contamination.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Gloves must be worn when handling tissue samples.

10.3.2 Tissue samples should be partially thawed before starting, to the point where it becomes possible to make an incision in, or cut through, the flesh.

10.3.3 Note any morphological abnormalities on the processing records.

10.3.4 Fish Tissue Preparation

10.3.4.1 Determine the wet weight for each individual fish using a calibrated balance and appropriate weighing dish. Follow project specifications for

alternate sample weight determinations.

10.3.4.2 Determine the length of each fish using a ruler, and record with the weight. Some measurements may, or may not be, a part of the project specifications. Additionally, a picture with a ruler in the foreground may be required. Follow project specifications.

10.3.4.3 Removal of Scales or Skin

10.3.4.3.1 If required by project specifications, the scales and/or skin of the fish will be removed prior to filleting. Clean all glassware and utensils as described in Section 10.1.

10.3.4.3.2 Rinse the fish with DI water and dry using a paper towel. Lay the fish on the cleaned, and/or lined, cutting board

Scrape the fish from tail to head using the blade edg**e** of a cleaned stainless steel, ceramic or titanium knife, to remove the scales. Continue until all scales are removed.

10.3.4.3.3 Depending upon the outward condition of the samples, the sample may be rinsed with DI water and pat dry with paper towel. Place the fish on a clean cutting board, for filleting or skinning.

10.3.4.3.4 To skin the fish: Using a stainless steel knife, cut the skin behind the operculum (gill cover). Using the knife blade, pliers or other cleaned utensil, pull the skin off towards the tail of the fish. If necessary, cut lightly along the inside of the skin, slowly separating the skin from the muscle tissue. Removing the skin may require cutting the skin along the backbone or underbelly of the fish. If necessary follow project specifications for weight determinations.

10.3.4.4 Filleting the Fish

10.3.4.4.1 Using fresh gloves and the specified knife, make a cut behind the entire length of the operculum (gill cover), making sure to cut through the skin, if still attached, and the flesh, as close to the bone as possible. Note: If the fish samples are small, and it appears difficult to fillet, or if the amount of the fillet appears to be insufficient for the analysis requested, consult the Department Manager and/or Project Manager prior to filleting. In some cases it may be necessary to homogenize the whole body.

10.3.4.4.2 Make a cut across the caudal peduncle (the base of the tail fin) keeping as close to the caudal (tail) fin as possible. Continue cutting along the underbelly of the fish, moving from the head to the tail.

10.3.4.4.3 Go back to the cut made at the beginning at the operculum, and slice down the entire length of the fish following along the backbone until reaching the cut previously made across the caudal peduncle.

Gently slide the stainless knife along the backbone of the fish and along the rib cage. Remove the fillet from the fish. Be sure to include the belly flap in each fillet and do not remove the dark muscle tissue in the vicinity of the lateral line from the light muscle tissue that makes up the rest of the muscle tissue mass.

10.3.4.4.4 Remove any bones that may be left attached to the fillet. Repeat the fillet steps 10.3.4.4.1 through 10.3.4.4.3, for the opposite side of the specimen.

10.3.4.4.5 Note in the sample processing records if the internal organs were ruptured during freezing or if inadvertent puncture of the internal organs occurred during the filleting process. If the internal organs did rupture or were punctured, notify project manager for further guidance.

10.3.4.4.6 Place a glass plate on the balance. Tare the balance and record the appropriate weights in the appropriate spreadsheet or logbook as determined from the project specific QAPP. This may include weighing the fillet(s), carcass or skin.

10.3.4.4.7 If the fillet(s) and/or the carcass are to be homogenized immediately, proceed to Section 10.3.4.5. If not, store in the appropriate container; see Section 4 for allowable materials. Note that it may be necessary to chop the fillet(s) or carcass into smaller pieces, with the appropriately cleaned knife, before storage, and before homogenization, so the entire sample will fit into the storage container or the homogenization vessel. See the project specific QAPP for additional details.

10.3.4.4.8 If the samples will not be homogenized immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until homogenization

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10.3.4.5 Homogenization

10.3.4.5.1 Allow the fillet(s), carcass or whole body to partially thaw if previously frozen.

10.3.4.5.2 Fillets/Skin/Whole Body: Weigh a glass jar on the balance and record the weight. Tare the same glass jar. Be sure the jar is large enough to allow headspace for freezing after sample homogenization. While wearing the appropriate gloves, place the sample on the cutting board. Using the appropriate knife, slice and cut the sample into small chucks, preferably 1” squares or less. Add the sample to the appropriate size glass container for homogenization. Record the pre homogenization weight or follow project specific QAPP. Immerse the sample into the pre-cleaned generator probe (see section 10.1). Homogenize the sample until it appears fully and consistently homogenized tuning into a fine paste. This procedure may require mixing the sample during the homogenization process with a stainless steel spatula, ensuring all sample is equally processed and no sample remains on the side of the jar.

10.3.4.5.3 Large Whole Body/Carcass: Large sample carcasses may need to be homogenized using a hand held grinder/ electric grinder or food processor. Add the pre-sliced sample to the pre-cleaned blender (see section 10.1) and “push” through the auger part of the grinder. Collect the sample into a pre-tared jar or glass plate. Further processing using

additional equipment may be necessary to achieve a consistently homogenized sample.

10.3.4.5.4 After homogenization, remove as much sample from the processing equipment as possible using a stainless steel spatula or other utensil and add to the processed sample. Re-weigh the sample and record the post-homogenization weight. Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.4.5.5 Place the individual or composite homogenized samples into the appropriate glass jars to be frozen pending future extraction/digestion. If the samples will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. All freezer logbooks must be filled out for hold time tracking purposes. Note the return of the samples to Sample Management must be documented in the LIMS Tracking log.

10.3.4.5.6 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1.

10.3.5 Mollusk Preparation

10.3.5.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes. Note removal of samples in the LIMS Tracking log.

10.3.5.2 If required by the project specifications, measure and record the length of the sample shell.

10.3.5.3 Cover the balance with the proper material as described in Section 4, and weigh and record the sample weight.

10.3.5.4 Wearing the proper gloves, place the sample on the cleaned cutting board. Samples should be partially thawed. If the sample is frozen, it will be difficult to break open the shell. If the sample is excessively thawed, the internal tissue will become soupy and difficult to remove.

10.3.5.5 If preparing *Bivalve* specimens, use the titanium knife to cut the abductor muscle by sliding the knife through the crevice where the two shells meet. Once the abductor muscle is cut the two shell pieces should come apart easily.

10.3.5.6 Carefully remove the top shell, and scoop out the internal tissue that is resting on the mantle. Be careful not to tip the bottom shell. If the sample is

excessively thawed, the sample internal fluids may spill out of the shell. The internal fluids must be retained as part of the sample. If the bivalve is still partially frozen as suggested, the tissue should easily be removed from the shell in one piece.

10.3.5.7 Cover the balance with the proper material and weigh the amount of tissue obtained from the sample. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar, see Section 10.3.5.17. If the sample will be homogenized immediately, proceed to 10.3.5.13.

10.3.5.8 If preparing *Gastropod* specimens, a mallet will be necessary to open the shell.

10.3.5.9 Place a paper towel or piece of lab mat over the shell of the Gastropod specimen

10.3.5.10 Holding the shell still with one hand, use the mallet to hit the paper towel that is over the shell, in order to crush the shell.

10.3.5.11 Using the appropriately cleaned tweezers, remove the tissue from the crushed shell pieces.

10.3.5.12 Cover the balance with the proper material and weigh the amount of tissue obtained from the sample. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar, see Section 10.3.5.17. If the sample will be homogenized immediately, proceed to 10.3.5.13.

10.3.5.13 Since the amount of tissue obtained from one bivalve or gastropod is generally small, several specimens are frequently combined to make one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

10.3.5.14 If several specimens will be composited to make one sample, follow the applicable Sections of 10.3.5.1 through 10.3.5.11, for each of the specimens. The tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight.

10.3.5.15 After the tissue has been removed from all of the specimen shells for one composite or individual sample, place the tissue in the clean small processor with the titanium blade to be homogenized. Grind the sample until it appears to be fully and consistently homogenized and there are no large chunks.

10.3.5.16 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be

recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.5.17 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion, see Section 4. If the samples will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. Record placement of the samples in the freezer, in the freezer storage logbook, for hold time tracking. Note return of the samples to Sample Management in the LIMS Tracking log.

10.3.5.18 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1.

10.3.6 Crustacean Preparation

10.3.6.1 Lobsters

10.3.6.1.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes. Note removal of samples in the LIMS Tracking log.

10.3.6.1.2 If project specifications require gender determination of lobsters, this must be done prior to dissecting. To determine the gender, hold the lobster by the thorax, and flip it over to examine the underneath abdomen. Just below the legs and where the abdomen division begins, there is a first pair of swimmerets. The first pair of swimmerets is what is used to distinguish the lobster's gender. If the first pair is soft, has small hairs, and the swimmerets are crossed, it is **female**. On a **male** lobster, the first pair of swimmerets is hard and stiff, and generally do not touch.

10.3.6.1.3 If the hepatopancreas of the lobster samples is to be analyzed, the samples should be received alive. If the samples are frozen prior to dissection the hepatopancreas could burst upon thawing making it difficult to remove. To remove the hepatopancreas, the live lobster should be placed on a cleaned cutting board. Wearing the proper gloves, one analyst holds the two chelipeds (claws) out in front of the lobster, while also holding down the lower abdomen and telson (tail). The second analyst takes a knife, and places it on the grove in the carapace (outer shell), just behind the head region. Keeping the knife at an angle, the second analyst must push down and forward to remove the head. Once the head is removed the hepatopancreas can be seen lying just under the carapace and running the length of the thorax. The hepatopancreas is generally a greenish-yellow color, but there may be some variation. Scoop the hepatopancreas out gently trying not to break it into pieces. Cover the tray of the balance with the proper material, and weigh and record the weight of the

hepatopancreas on the processing record, and place it into an appropriate sample jar for freezing and future extraction/ digestion.

10.3.6.1.4 To remove the edible meat, remove the two chelipeds from the body of the lobster at the joint. Place a piece of lab mat or paper towel over the cheliped and pound with a mallot. Once the shell is crushed remove the meat, using the appropriately cleaned tweezers or other tool, making sure to get all the meat in the joints and arms. Cover the balance tray with the appropriate material and weigh and record the total tissue weight obtained from the two chelipeds and arms. Record this weight with the previously recorded information on the sample processing record.

10.3.6.1.5 Remove the abdomen and telson from the rest of the carapace by pulling the lobster apart. Using the titanium coated knife, cut through the center underside tissue of the lobster and laterally along the exoskeleton of the tail. Once the abdomen and tail have been cut open, separate the shell from the edible meat using cleaned utensils. Any eggs found in the female lobsters will have to be removed and discarded. Cover the balance tray with the appropriate material, and record the weight of the tissue obtained from the abdomen and telson on the processing record. The sample may now be stored pending homogenization in the appropriate jar.

10.3.6.2 Crabs

10.3.6.2.1 If removing tissue from *crabs* break off all legs and claws. Squeeze, pull, cut or pick all the tissue out of the legs and chelipeds. Pull apart the carapace. The carapace should be easy to remove by pulling up on the holes left from when the legs were broken off. Scoop out the tissue. Cover the balance tray with the appropriate material and record the weight of the tissue obtained from the legs, claws, and carapace on the processing record. The sample may now be stored pending homogenization in the appropriate jar, see Section 10.3.6.7. Any eggs found in the female crabs will have to be removed and discarded.

10.3.6.2.2 If the hepatopancreas of the crab samples is to be analyzed, the samples should be received alive. If the samples are frozen prior to dissection the hepatopancreas could burst upon thawing, making it difficult to remove. In order to remove the hepatopancreas of a frozen crab, remove the legs and claws, and then the top shell can be removed by cutting along the outside edge of the top shell. The top shell can then be removed. It is best if the crab(s) are chilled live in a refrigerator for 30-60 minutes, prior to removal of the hepatopancreas, to slow the crab's movements. To remove the hepatopancreas, the live crab should be placed on a cleaned cutting board. Wearing the proper gloves, the analyst must hold the crab still, with the claws facing away from the analyst. Then grab the back of the top shell with fingers or cleaned pliers, and pull the back shell from the crab. Once the back shell is removed the hepatopancreas can be seen lying inside the body cavity. The hepatopancreas is generally a greenish-yellow color, but there may be some variation. Scoop the hepatopancreas out gently trying not to break it into pieces. Cover the tray of the balance with the

proper material, and weigh and record the weight of the hepatopancreas on the processing record. Place it into an appropriate sample jar for freezing and future extraction/ digestion.

10.3.6.3 Since the amount of tissue obtained from one crustacean may be small, several specimens may be combined to make one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be cleaned and rinsed in between each composite sample.

10.3.6.4 If several specimens will be composited to make one sample, follow the applicable Sections of 10.3.6.1.1 through 10.3.6.1.5 for lobsters, or 10.3.6.2.1 through 10.3.6.2.2 for crabs for each of the specimens. The tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight.

10.3.6.5 After the tissue has been removed from all of the specimen shells for one composite or individual sample, grind the sample until it appears to be fully and consistently homogenized and there are no large chunks. This procedure may require mixing the sample during the homogenization process with a stainless steel spatula, ensuring all sample is equally processed and no sample remains on the side of the jar.

10.3.6.6 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.6.7 Place the processed samples into the appropriate glass jars to be frozen for future extraction/digestion, see Section 4 for allowable materials. If the samples will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. Record placement of the samples in the freezer, in the LIMS, for hold time tracking. Note return of the samples to Sample Management in the LIMS Tracking log.

10.3.6.8 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1. If any processing equipment comes in contact with a crab that is not going to be included in the composite, the equipment must be washed as described in section 10.1 before continuing.

10.3.7 Mammals (Mice and Shrew)

10.3.7.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes.

10.3.7.2 Place the first five, partially thawed samples to be processed, and all equipment needed into the glove box on a freshly laid out lab mat. Equipment needed includes:

- Empty and pre-labeled glass sample containers for the processed homogenate,
- PVC gloves or Latex gloves,
- 10% Bleach solution, 25% HNO3 and methylene chloride, methanol and hexane in squirt bottles,
- Omni grinding unit,
- Balance,
- Nylon bristled brushes,
- Ceramic, titanium,or stainless steel (organic compounds only) knives, spatulas and/or other utensils,
- Cutting board (s),
- Dl water in a squirt bottle and Kim wipes,
- Laboratory waste bottles with caps.

10.3.7.3 Once all materials are in the glove box and set up for use, seal the transfer box and ensure the motor blower is on. Over tightening of the outer or inner door knobs is not necessary to achieve a good seal. Place your hands into the gloves attached to the glove ports and place PVC or Latex gloves over the glove port gloves for use. The outer PVC or Latex gloves will need to be changed in between each sample.

10.3.7.4 If the gender of the mouse or shrew needs to be determined, turn the animal over and note the length of the anus and the distance of the anus from the tail. If the anus is elongated in shape and does not touch the base of the tail, testicles and a large genital papilla are visible, and there are no nipples, the animal is **male**. If the anus is round in shape and almost touches the base of the tail and/or there are nipples (up to five sets), the animal is **female**. If the animal is very small, young or immature and a gender determination cannot be made, note that the gender is *un-determined*. Record the gender observations on the processing records.

10.3.7.5 If skinning of the mammal is required, carefully make an incision at the tail end and cut just below the skin along the back, from one hind leg to the other. Make another cut from one hind leg to one front leg, and repeat the cut on the other side of the animal. Starting from the tail, lift the skin flap, and carefully separate the skin from the muscle tissue below. Pull the skin forward from the tail to the head to expose the back tissue of the animal. Repeat the procedure on the stomach side of the animal. Note: it may be very difficult to remove the skin from the legs, head and the tail. If some skin cannot be removed, note this on the processing records.

10.3.7.6 Weigh and record the weight of the mammal on the processing records. Depending upon the size of the mammal, it may need to be chopped into small pieces before being ground. Generally, mice and shrew can be quartered before homogenization if needed.

10.3.7.7 Put the whole body or chopped sample into the cup of the grinding unit. Ensure the sample is in contact with the blades of the unit and place a bag over the entire grinding unit to help contain and minimize splatter on the walls of the glove box.

10.3.7.8 Turn the grinding unit on low speed and gradually increase the speed to homogenize the sample being careful to minimize any splatter or outside contamination. Homogenize until a uniform consistency is achieved.

10.3.7.9 Transfer the homogenized sample from the cup to the pre-labeled sample jar using the appropriate utensil. Carefully clean the threads of the sample jar with a DI water-soaked Kim wipe. Clean the outside of the sample jar with a 10% bleach-soaked Kim wipe. Set the sample jar inside the transfer box and close the transfer box inner door.

10.3.7.10 To clean the grinding unit in between samples, remove as much residual tissue on the blade as possible by operating the unit at low or medium speed with DI water in the sample cup. Keep a bag over the grinding unit as the primary containment for splashing. If necessary, use the nylon brush to gently scrub the exposed surfaces and to dislodge remaining tissue. Repeat as necessary, until the unit appears clean. Any plastic or ceramic parts must now be given a final rinse with 25% HNO3 then DI water when processing samples for metals analysis. If processing for organic compounds only, rinse with DI water, acetone and then the methylene chloride.

10.3.7.11 Repeat steps 10.3.2.3 through 10.3.7.10 until the five samples have been processed and each placed into the transfer box. Ensure the outer Latex or PVC gloves are changed in between each sample.

10.3.7.12 Since the amount of tissue obtained from one mouse or shrew may be small, several specimens may be combined to make one sample, as required by project specifications. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be cleaned in between each composite sample.

10.3.7.13 If several specimens will be composited to make one sample, follow the applicable Sections of 10.3.7.3 through 10.3.7.10, for each of the specimens. The tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight.

10.3.7.14 Remove the individual or composite sample jars from the transfer box from the outside of the glove box, and return them to the Sample Management office for storage in the freezers until extraction/digestion. At the same time, obtain the next five samples to be processed and homogenized from the Sample Management office freezers. Movement of samples into and out of freezer storage must be documented in the freezer logbooks and in the LIMS Tracking log.

10.3.7.15 Allow the samples to partially thaw and begin again at 10.3.7.3 through 10.3.7.14 until all samples have been processed and homogenized. Clean the outer surfaces of the homogenate sample jars as described in

10.3.7.10, and remove them from the transfer box. If the samples will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. Record placement of the samples in the freezer, in the freezer storage logbook, for hold time tracking. Note return of the samples to Sample Management in the LIMS Tracking log.

10.3.7.16 Before removing any equipment from the glove box, the following disinfection steps must be taken:

- Remove the primary containment bag. Take care not to invert the bag. Place this bag into another bag.
- After the grinding unit, cup and blades have been cleaned with DI water as in 10.3.7.10, rinse the entire unit with the 10% bleach solution. Collect the bleach in a waste bottle.
- Remove the bags that were twist tie secured to the grinding unit, and place them into another bag. Rinse the entire unit again with the bleach solution.
- Roll up the bench liner, and place this into a bag.
- Pour all waste solutions into capped waste bottles. Place these bottles and any other bleach cleaned utensils, into bags, and seal all bags.
- Wipe the inside surfaces of the glove box with Kim wipes soaked in the bleach solution.
- The glove box transfer doors may now be opened to remove the grinding unit and waste. The waste material may be discarded after adding 10% bleach. The utensils and the grinding unit may be re-washed according to the normal cleaning procedures.

10.3.7.17 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.7.18 After individual homogenates have been combined to form the final sample composite homogenate, as requested, all utensils and equipment must be washed, in between samples, according to the procedures described previously in Section 10.1.

10.3.7.19 If the final sample composite homogenates will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. Record placement of the samples in the freezer, in the LIMS, for hold time tracking. Note return of the samples to Sample Management in the LIMS Tracking log.

10.3.8 Reptiles and Amphibians (Frogs and Turtles)

10.3.8.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes. Note removal of samples in the LIMS Tracking log.

10.3.8.2 Wearing the proper gloves, place the *turtle* sample on the cleaned cutting board. The turtle should be partially thawed. If the turtle is frozen, it will be difficult to remove the muscle. If the sample is excessively thawed, the internal tissue will become soupy and difficult to remove.

10.3.8.3 Take all project required measurements. The distance between the anterior and posterior edge of a turtle carapace (top of shell) should be measured with a ruler and recorded on the processing records. If the entire mass of the turtle, including the shell, needs to be recorded, cover the balance with the proper material and weigh and record this weight on the processing records.

10.3.8.4 Since the plastron (bottom of shell) and carapace are extremely dense and difficult to cut through with normal dissecting tools, the muscle tissue of the turtle must be removed by cutting the body of the turtle away from the shell. Insert a knife, made of the proper material, into the skin of the turtle, close to the shell on the lower half of the body. Slowly, cut along the entire circumference of the shell. Repeat the procedure on the upper half of the body, on both sides of the shell.

10.3.8.5 With dissection scissors, or a ceramic or titanium paring knife of the proper material, remove the skin from the hind limbs, tail, fore limbs and neck.

10.3.8.6 Using the appropriate utensils, remove the muscle tissue from the tail, neck, hind limbs, and fore limbs, including the feet, leaving bone and claws behind. Remove any visible muscle tissue within the carapace. Most of this tissue will be found in the upper portion of the carapace around the pectoral area.

10.3.8.7 Cover the balance with the proper material and weigh the amount of tissue obtained from the turtle sample. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar, see Section10.3.8.15. If the sample will be homogenized immediately, proceed to 10.3.8.13.

10.3.8.8 If processing *frogs*, allow the frog to partially thaw, take the project specific measurements, and record them on the processing records. The number of frogs required to make up one sample, and the weight and length of the individual frogs, must be taken and recorded, if specified. In all cases, the skin must be removed from the frog prior to processing and chopped into smaller pieces, due to its thickness. It will then be added to the processor with the whole body of the frog, or it may be discarded depending upon the project specifications.

10.3.8.9 To skin the frog, make an incision, using the proper utensils, and cut into an area where there is an excess of skin, most likely around the neck. Slowly, pull the skin off of the frog using dissecting scissors, or a ceramic or titanium paring knife, as needed. Once skin is removed chop it up into tiny pieces using the appropriate knife and set it aside to be processed with the whole frog body.

10.3.8.10 Cover the balance with the proper material and weigh the amount of tissue obtained from the frog sample, if the tissue and not the whole body will be processed. Record the weight along with the information previously recorded on the processing records. The sample may now be stored pending homogenization in the appropriate jar, see Section 10.3.8.15. If the sample will be homogenized immediately, proceed to 10.3.8.13.

10.3.8.11 Since the amount of tissue obtained from one small turtle or frog may be insignificant, several specimens may be combined to make up one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

10.3.8.12 If several specimens will be composited to make up one sample, follow the applicable Sections of 10.3.8.1 through 10.3.8.10, for each of the specimens. The tissue obtained from each specimen may be weighed and recorded individually, then totaled for the composite weight. If only the composite weight is sufficient for the project specifications, weight the entire composite and record that weight.

10.3.8.13 After the tissue has been removed from all of the specimens, homogenize the muscle tissue, and skin if required, by placing it into the small or large food processor fitted with the appropriate blades (stainless steel for the large processor and titanium for the small processor). See Section 4 for allowable materials. The sample may need to be cut into smaller pieces for processing. Grind the sample until it appears to be fully and consistently homogenous. Continue to grind the sample until there are no chunks present in the homogenate.

10.3.8.14 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.8.15 Individual or composite samples may be returned to the Sample Management office for further storage in freezers pending extraction/digestion. All processed samples are stored in the proper containers noted in Section 4. All freezer logbooks must be filled out for hold time tracking purposes. Return of samples to Sample Management must be documented in the LIMS Tracking log.

10.3.8.16 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1.

10.3.9 Macro Invertebrates

10.3.9.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes. Note removal of samples in the LIMS Tracking log.

10.3.9.2 Cover the balance tray with the appropriate material and record the weight of the invertebrate sample. Since the weight obtained from one invertebrate (benthic worms, insects or biota) may be small, several invertebrates may be combined to make one sample. In many cases, several invertebrates of the same species and sample location are delivered to the laboratory in one sample jar. Each specimen from this jar must be weighed, if requested, and composited to form one homogenized and unique sample. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight. Utensils do not need to be rinsed between the individual samples or specimens that comprise one composite, but utensils must always be rinsed in between each composite sample.

10.3.9.3 Invertebrates such as *eels* must be chopped into smaller pieces before homogenization. This is generally due to the length of the specimen and the thickness of the skin.

10.3.9.3.1 For project specifications requiring eel specimens to be skinned prior to homogenization, first secure eel to cutting board using a stainless steel screw. Using a stainless steel knife, cut the skin behind the operculum (gill cover). Using the knife blade, pliers or other cleaned utensil, pull the skin off towards the tail. If necessary, cut lightly along the inside of the skin, slowly separating the skin from the muscle tissue. Removing the skin may require cutting the skin along the backbone or underbelly. If necessary follow project specifications for weight determinations.

10.3.9.4 Place the weighed specimen into the clean small processor with the titanium blade to be homogenized. Process the sample until it appears to be fully and consistently homogenized and there are no large chunks.

10.3.9.5 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.9.6 Individual or composite samples may be returned to the Sample Management office for further storage in freezers pending

extraction/digestion. All homogenates are stored in the proper containers noted in Section 4. All freezer logbooks must be filled out for hold time tracking purposes. Return of samples to Sample Management must be documented in the LIMS Tracking log.

10.3.9.7 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1.

10.3.10 Plants

10.3.10.1 Wash all utensils, the cutting board, and surfaces as previously described in Section 10.1. Note the allowable materials in Section 4. Obtain samples from the Sample Management office and log them out of the freezer logbooks for hold time tracking purposes. Note removal of samples in the LIMS Tracking log.

10.3.10.2 Wearing the appropriate gloves, plants must be rinsed with DI water to remove soil, silt, small insects, and other debris. Place the plants in a stainless steel or plastic strainer, depending on the determinative sample analysis, and rinse thoroughly with DI water. If analyzing the sample for metals and organic compounds, rinse the plants carefully over a sink, being sure not to touch the sides of the sink with the plant sample.

10.3.10.3 Depending on the size and texture of the plants, some may be homogenized in the small food processor with the titanium blade. Samples such as long grass will have to be chopped into small pieces (approximately 1/2 inch) using titanium or ceramic knives. Leaves can generally be homogenized in the small food processor without pre-cutting.

10.3.10.4 Some project specifications may require the plants to be dried prior to homogenization. A plastic salad spinner may be used to remove excess water from samples, if organic compounds do not need to be determined. If both metals and organic compounds need to be determined, air drying for 48 hours, or oven drying overnight at low temperatures (S 50°C), can be done. Freeze drying the plant is an additional option for the removal of water and may be employed per project specifications.

10.3.10.5 Cover the balance tray with the appropriate material and record the weight of the plant sample. Since the weight obtained from one plant may be small, several plants may be combined to make one sample. Utensils do not need to be rinsed between the individual samples that comprise one composite, but utensils must always be rinsed in between each composite sample.

10.3.10.6 If several plants will be composited to make one sample, follow the applicable Sections of 10.3.10.2 through 10.3.10.5, for each of the specimens. The weight of each specimen may be recorded individually, and then totaled for the composite weight. If only one composite weight is sufficient for the project specifications, weigh the entire composite and record that weight on the processing records.

10.3.10.7 After the plant weight for one composite or individual sample has been recorded, place the plant(s) in the clean small processor with the titanium

blade to be homogenized, or place them onto the cleaned cutting board to be chopped. Grind or chop the plants until they appear to be fully homogenized.

10.3.10.8 Individual homogenates may be processed further to prepare composite homogenates as required by project specifications. Composite homogenates must be prepared from equal weights of individual homogenates. All individual weights that make up one composite must be recorded, if required, or one composite weight may be recorded. If individual or composite homogenates were frozen prior to extraction/digestion, these homogenates must be thawed and re-homogenized by hand mixing prior to being extracted or digested.

10.3.10.9 Place the homogenized plants into the appropriate glass jars to be frozen for future extraction/digestion, see Section 4. If the samples will not be extracted/digested immediately, the samples must be returned to the Sample Management office and placed back into the freezer, until extraction/digestion. Record placement of the samples in the freezer, in the freezer storage logbook, for hold time tracking. Return of samples to Sample Management must be documented in the LIMS Tracking log.

10.3.10.10 All utensils and equipment must be washed in between samples according to the procedures described previously in Section 10.1.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

Not applicable to this method.

11. Data Evaluation, Calculations and Reporting

The processing bench sheets and other relevant laboratory notebooks must follow the specifications in the Alpha Analytical *Logbook Usage Work Instructions* (WI 108-01), and all record keeping and document control practices. Separate project-specific documents may be used in place of Alpha bench sheets, as necessary.

See the appropriate Alpha Analytical SOPs noted in Section 1, for details on sample analysis, data evaluation, calculations and data reporting.

All results for the organic/inorganic compounds of interests are reportable without qualification if extraction/digestion and analytical holding times are met, preservation (including cooler and freezer temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during extraction/digestion and/or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

QC Parameter	Acceptance Criteria

Equipment/Processing Blank	< reporting limit
Method Blank	< reporting limit
Laboratory Control Sample	See the applicable ALPHA analytical SOP for acceptance criteria
Matrix Duplicate	See the applicable ALPHA analytical SOP for acceptance criteria
Matrix Spike	See the applicable ALPHA analytical SOP for acceptance criteria
Matrix Spike Duplicate	See the applicable ALPHA analytical SOP for acceptance criteria
Surrogate Recoveries	See the applicable ALPHA analytical SOP for acceptance criteria
Standard Reference Material	See the applicable ALPHA analytical SOP for acceptance criteria

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9, Quality Control, defines the preparation and/or analytical corrective actions that must be taken in instances where QC outliers exist.

Section 11 outlines sample batch QC acceptance criteria. If non-compliant organic or inorganic compound analytical results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ) – Not Applicable

Not Applicable

13.2 Demonstration of Capability Studies

Not Applicable

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste and Sample Disposal SOP for further pollution prevention and waste management information.

Once satisfactory organic or inorganic compound results have been generated, the extracts/digestates are held for 30 days, or longer, if specified by a client contract. Then, organic extracts are discarded into a 55-gallon drum labeled "Vial Waste" and inorganic digestates are poured into a 55-gallon drum marked "Acid/Non-chlorinated" waste.

All solvent or reagent waste generated during processing and/or extraction/digestion must be stored in satellite containers in the preparation laboratories labeled "Organic Solvent", "Acid/Non-chlorinated" or "Bleach".

Once the organic solvent satellite containers are full, they must be emptied into 55-gallon drums marked "Organic Solvent Waste". Cleanup waste from the HPLC fractionators (silica cleanup) or GPC is emptied into the 55-gallon drum marked "HPLC Solvent Waste". Bleach from disinfection is emptied into the 20-gallon drum marked "Bleach", and reagent waste generated during metals analysis is emptied into a 55-gallon drum marked “Acid/Non-chlorinated" waste.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/G-006 Hazardous Waste and Sample Disposal

16. Attachments

None.

Appendix D.3
Clam Siphon Skin Removal Standard Operating Procedure

STANDARD OPERATING PROCEDURE

CLAM TISSUE – SIPHON SKIN REMOVAL

A Introduction

For inorganic arsenic analysis, clam tissue will be analyzed as siphon skin and remainder tissue. This SOP describes the process for clam siphon skin removal; other aspects of clam tissue processing are described in Analytical Resources Laboratory SOP 3328S, Extraction of Tissue Samples for Organic Extractions, also included as part of this appendix.

B Siphon Skin Removal

Clam siphon skin tissue will be removed as described in the following steps:

1. *Prepare equipment* – Gather laboratory-decontaminated tools to be used for clam dissection. Put on clean gloves between all composite samples.

2. *Prepare clams* – Partially thaw clams. Clean the clam shell by squirting with deionized water and blotting dry with a chem wipe.

3. *Open clam shell* – At a location on one side of the clam shell hinge, insert a clean knife between the two sides of the shell. The depth of penetration of the knife should be only as deep as is needed to reach and cut the muscle attachment (insertion should generally be less than 0.5 in., depending on clam size). Repeat this process on the other side of the hinge, and then pry open the clam from the front.

4. *Cut clam siphon skin* – Make a shallow cut along clam siphon skin, being careful not to bisect the siphon.

5. *Remove siphon skin* – Pull off siphon skin by hand. The siphon skin is the thin, brown tissue layer surrounding the siphon.

6. *Rinse and weigh siphon skin* – Rinse removed siphon skin with deionized water and pat dry using a chem wipe. Place siphon skin in a weight boat (on pre-tarred scale) and record weight on bench sheet. Once measurement is recorded (and any abnormalities are noted and photographed), place siphon skin in the siphon skin composite jar.

7. *Remove remainder tissue* – Remove the remaining clam body tissue (i.e., the mantle and foot) from the shell.

8. *Rinse and weigh remainder tissue* – Rinse remaining tissue with deionized water and pat dry using a chem wipe. Place tissue in a weight boat (on pre-tarred scale) and record weight on bench sheet. Once measurement is recorded (and any abnormalities are noted and photographed), place tissue in the remainder tissue composite jar.

9. *Discard shell and repeat for additional clams* – Discard shell after all tissue has been removed. Repeat with remaining clams to be included in each composite.

Appendix D.4
Analytical Resources Laboratory Extraction of Tissue Samples for Organic Extractions Standard Operating Procedure

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Extraction of Tissue Samples
For
Organic Extractions

SOP 3328S
Revision 0.1
Revision Date: 10/23/19
Effective Date: 10/23/19

Prepared By:

Warren Woodard

Approvals:

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David R. Mitchell, Quality Assurance Manager

Annual Review

SOP Number: 3328S

Title: Extraction of Tissue Samples for Organic Extractions

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

1. Scope and Application

1. This document describes the procedures used at ARI to preform solvent extraction of tissue samples using a Tissumizer extraction for organic analysis and %Lipids.

2. Summary of the Procedure

1. Tissue samples that are not already ground or blended will be chopped, ground and blended as needed to allow for a homogenous aliquot to be taken and properly extracted by the Tissuemizer process.
2. Samples are weighed into Centrifuge bottles.
3. Weighed samples are surrogated and spiked and the appropriate solvent is added.
4. Each sample is dried with sulfate immediately before being Tissuemized. If they are dried too soon it may result in clumping that will hinder the Tissuemizing process.
5. Tissuemizing equipment is thoroughly cleaned between each sample using Soap, Diluted Acid, and DI water, and are then Acetone Rinsed.
6. Samples are Tissuemized, decanted through a funnel with glasswool into a flask with sodium sulfate.
7. Additional solvent is added to the sample and the sample is Tissuemized and Decanted two more times.
8. Concentrations and Cleanups will then be performed using SOP 3327S.

3. Definitions

1. Blank Spike (BS) – A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes or material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system. (May also be listed as LCS or SB short for Laboratory Control Sample and Spike Blank respectively.)
2. BSD (Blank Spike Duplicate) – A duplicate blank fortified with known quantities of selected analytes to monitor extraction efficiency. (May also be listed as LCSD or SBD short for Laboratory Control Sample Duplicate and Spike Blank Duplicate respectively.)
3. Matrix Spike (MS) – A sample prepared by adding a known mass of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of sample matrix on a method's recovery efficiency.
4. Matrix Spike Duplicate (MSD) – A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
5. Method Blank (BLK) – A sample 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, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
7. Minimum Reporting Limit (MRL) - A sample matrix, free from the analytes of interest, spiked with verified amounts of analytes at the LOQ of the instrument curve.

4. Interferences

1. Standard solutions, solvents or supports materials must not contain extraneous compounds or other chemical interferences. All standards are verified by GC/ECD, GC-FID or GC/MS prior to use. All solvent lots and support materials are checked for purity prior to use. (See Bench sheet 3058F for instructions).
2. Glassware used in the procedure is cleaned and kiln fired as described in ARI SOP 301S. Glassware is rinsed with clean solvent just prior to use.
3. Extracting solvent may contain impurities that could interfere with analyses. Use solvents from a supplier's lot that have are verified free of such contaminates.
4. Laboratory supplies and equipment are potential sources of interfering contamination
    1. Items, such as gloves, bench paper, and rubber stoppers, should not come into contact with samples or extracts.
    2. Use glassware that is thoroughly cleaned, kiln fired (ARI SOP 301S) and solvent rinsed.
5. Airborne dust and other debris may contaminate samples. Samples and extracts must be covered at all times.

5. Safety

1. The toxicity or carcinogenicity of each reagent used in this SOP are not precisely defined. Treat each compound as a potential health hazard. Reduce exposure to all chemicals to the lowest possible level by whatever means available.
2. Always wear appropriate PPE (personal protective equipment) when working in the Organics Extraction Laboratory. Gloves, safety glasses, ear protection, lab coats, respirators, face shields, etc. are provided for your protection.
3. DO NOT attempt to cleanup solvent spills in the laboratory. Immediately evacuate the area and contact a member of the Emergency Response Team (ERT) for assistance.
4. Safety Data Sheets (SDS) that outline hazards, exposure limits, treatments and regulatory guidelines are available for all chemicals used in this procedure and should be consulted when such information is required. The SDS file is located in the central project management area. SDS are also available online, at http//www.msds.com

5. Environmental Samples may contain hazardous material; treat them as potential health hazards.
6. Dispose of all unwanted, broken glassware into a broken glassware disposal box. Inspect every piece of glassware prior to use. Do not use glassware that is chipped, cracked, etched, or scratched. Set aside glassware with minor damage for repair.
7. Use nitrile gloves when working with organic solvents; latex gloves are not appropriate for working with methylene chloride or other solvents.

6. Equipment and Supplies

1. Equipment
    1. Personal protective equipment (PPE)
        1. Gloves
        2. Laboratory coat
        3. Goggles
    2. Tissuemizer (Tekmar mark II Type T25-S1 or equivalent)
    3. Blender (Magic Bullet or equivalent.)
    4. Chopper or grinder for tissue samples (Chefmate Model # CC12 or equivalent)
    5. Cleaning station for tissuemizer, blenders or chopper parts to include three deep, gray bins, white plastic drying bins, paper towels, Contrex AP detergent, diluted HCL, deionized water, aluminum foil, brushes and broad-range pH paper.
    6. Drying oven set at 105c (+/- 5)
    7. Analytical Balance - Top Loading, readable to 0.02g
    8. Hamilton gastight syringes – 100µL, 250 µL, 500 µL, 1000 µL, 2500 µL
2. Glassware
    1. Erlenmeyer flask, glass 500mL
    2. Centrifuge bottles, glass 250mL
    3. Beaker, glass 400mL (one for each sample)
    4. Powder Funnel, glass 100mm
3. Supplies
    1. Spatula, stainless steel
    2. Glasswool, neutral, (See SOP 301S)
    3. Wide Sharpie Permanent Pen
    4. Colored label tape
    5. Kim Wipe

7. Reagents and Standards for Tissue extractions: Reagents by Parameter, Matrix, Task Instruction, and Preparation of the solutions is located in SOP 3327S Appendix 20.2, (TI-020)

1. Acetone, high purity
2. Methylene Chloride, high purity
3. Hydrochloric Acid (HCL)
4. 1% Nitric Acid Solution
5. Anhydrous sodium sulfate, (See SOP 301S)
6. Organic Free Water – ASTM Type 1 Water produced using ARI's central water purification system.
7. Spiking solutions prepared and verified by the GC laboratory. Verify that all labeling is clear and complete prior to using one of these solutions. Preparation of the solutions is documented in Element.

8. Sample Collection, Preservation, Shipment and Storage

1. Tissue samples may arrive at the lab frozen. Frozen tissues must be stored at ≤ -10 ° C and thawed just prior to beginning the extraction process.
2. Archived tissue samples must be stored at ≤ -10 ° C
3. Hold times for Tissue Samples is 14 days, however while samples are frozen the hold time does not progress. Only days that the tissue samples are thawed count toward the hold time. Hold times may be different dependent on analysis and client requirements.
4. When hold times are or may be compromised inform your supervisor immediately.
5. Some samples are shared with the metal and or conventional laboratories. These samples are placed in a share bin in Refrigerator 36 when not frozen. SOP 1019S includes procedures for handling shared samples.
6. Some samples are shared with the Dioxins laboratory. These samples are placed in a share bin in Refrigerator 5 when not frozen. Do not Archive or Freeze samples shared with Dioxins without first verifying the Dioxins lab is done with them. If Dioxins will not be extracting the samples right away, they may be frozen until Dioxins is ready to use them. Consult with the Project manager and Dioxin Lab Supervisor before freezing tissue samples that Dioxins is not done with.

9. Quality Control

1. Quality Control requirements are specified on the analysis specific bench sheet
2. Generally, all samples are spiked with surrogate standard(s), and each extraction batch of 20 or fewer samples include a Method Blank (BLK) and a Blank Spike (BS).
3. When requested by ARI's client's or required by published analytical protocol other QA samples such as Matrix Spikes, Replicates, Quantitation Limit Standards, Standard Reference Materials are prepared and extracted.

4. To verify that surrogate and matrix spikes is accurate; all spike additions are witnessed and documented by a second laboratory technician. **(See SOP 3327S Appendix 20.4).**
5. QC samples including Method Blanks and Blank Spike (BS) must be subjected to the clean-up procedures along with any samples that require that technique.
6. Prior to beginning the extraction process, determine what procedures are required by reviewing the bench sheet. Follow all information listed for extraction volumes, surrogate and spike additions, pH requirements and special instructions listed with all bench sheets. **Bench sheet Forms are dynamically generated by Element using the Extraction Instructions Database.**
7. Proceed slowly and carefully while extracting samples. Note all problems, concerns, errors or deviations from the standard procedure on an Analyst Notes Form (Form 3056F). What seem to be minor errors or deviations may have a significant impact on the final results. Analyst must report all deviations from standard procedures to the laboratory supervisor as soon as possible. Failure to do so may result in a disciplinary process.
8. Label all glassware and vials with permanent markers.
9. Verify sample identification is transferred correctly when transferring or vialing samples and extracts.
10. The Element entry person will review all bench sheets before distribution.
11. The laboratory supervisor reviews all logbooks for completeness and accuracy monthly.
12. The QA section periodically reviews the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

10. Calibration and Standardization

1. Verify the accuracy of each analytical balance prior to weighing samples. The procedure is included in SOP 1003S. Record the results of the balance check in the appropriate logbook.

11. Procedure

1. The procedure includes a series of tasks performed sequentially. The tasks are listed in Table 01. A different analyst may be assigned to any task. **Detailed procedures for each task are contained on each unique Bench Sheet and in specific "Task Instructions" (TI).** All tasks are listed in Table 01. Details of the first five task instructions are included in this section (TI 031 thru TI 035). "Task Instructions" (TI 008, TI 009, TI011S thru TI 017, TI 019, TI 020, TI 036) are detailed in SOP 3327S.

Table 01-Task for Extraction of Soil/Sediment samples			
	Preparation Step	**Optional Tasks**	**Task Instruction**
1	Prepare for extraction		TI 031
2	Weigh sample	Tissuemizer	TI 032
3	Spike sample	Tissuemizer	TI 033
4	Dry sample	Tissuemizer	TI 034
5	Extract sample	Tissuemizer	TI 035
6	Concentrate extract & Hexane Exchange	Kudema-Danish (KD)	**SOP 3327S** TI 008
7		TurboVap	**SOP 3327S** TI 009
8		Vacuum Desiccator (%Lipids)	**SOP 3327S** TI 036
9	Extract Cleanup	GPC Cleanup	**SOP 306S**
10		Sulfuric Acid/Silica Gel for TPH-D Soil	**SOP 3327S** TI 011S
11		Sulfuric Acid for Pesticide or PCB	**SOP 3327S** TI 012
12		Sulfur Removal	**SOP 3327S** TI 013
13		Solid Phase Extraction (SPE)-Silica Gel Clean-up	**SOP 3327S** TI 014
14		Manual Column Alumina Clean-up	**SOP 3327S** TI 015
15		Manual Column EPH Fractionation	**SOP 3327S** TI 016
16	Hydrolysis/Derivatization	Hydrolysis/Derivatization for TBT	**SOP 3327S** TI 017
17	Derivatization	Derivatization for PCP or Sim PNA PCP	**SOP 3327S** TI 018
18	Vialing of final extract		**SOP 3327S** TI 019

2. **(TI-031)** Preparing for extraction
    1. Preparing Tissues for Extraction
        1. It may be necessary to initiate some preparatory steps (dissection, manual chopping, removal of unwanted tissue, ext.) prior to extracting the tissue. The portion of tissue to be extracted must be representative and amenable to the extraction process. Dissection must be done by a qualified laboratory technician. See Laboratory Supervisor for specific instructions pertaining to tissue preparation requirements. NOTE: When preparing tissue samples that may be analyzed for trace metals, all metal instruments (spatulas, knives, razor blades, blender, etc.) must be rinsed with copious amounts of 1% nitric acid between each sample to prevent metals contamination of the sample.

2. Clams and mussels should be shucked and homogenized in a blender or chopper. Other small tissues parts such as worms or organic material may also be blended provided there is enough free flowing tissue moisture. It may be necessary to cut or cube some tissue before blending or chopping.
3. Use a stainless-steel spatula to pry open the shell of the clam or mussel and scrape the tissue into a jar with PTFE-lined screw cap labeled with sample ID. Clam or mussel tissue must be homogenized using a pre-cleaned blender. Blend the tissues thoroughly and place them back into the labeled sample jar. Clean the blender between samples by disassembling the blade, and washing as described in section 11.2.2 below.

2. Cleaning Preparation for Tissuemizer, blender or chopper parts
    1. All Tissuemizer, blender and chopper parts that contact the sample must be disassembled completely and cleaned prior to initial use and after each sample to ensure that no cross-contamination will occur.
    2. Prepare the Tissuemizer, blender or chopper parts and associated tools at a sink with hot running water. Disassemble the equipment completely then thoroughly rinse the parts with hot tap water and transfer them to the washing station.
    3. Prepare three water baths for washing the disassembled parts and tools as listed here in order of intended use:
        1. Bin #1 – ½ Cup Contrex AP detergent in approximately 12L hot tap water. The tub will be ¾ full. The resulting solution will be basic (pH ~ 12).
        2. Bin #2 - 20mL concentrated HCL in approximately 12L hot tap water. This rinse water will be pH 2 acidic to remove soapy, basic residue left on parts.
        3. Bin #3 – Deionized water. This rise water should be pH 5-9 neutral to neutralize parts. Check the DI water frequently to ensure correct pH range using broad range pH pater. Change the DI water if it is acidic (<pH 4)
    4. Parts Cleaning
        1. Operating at the washing station, remove all disassembled parts, including the tools used to disassemble the equipment and clean them sequentially in the three baths listed above. In Bin #1, scrub each piece thoroughly with brushes and inspect each part, making sure there is no residual tissue on the part or in any small crevices. Rinse all parts thoroughly with hot tap water, then submerge all pieces in Bin #2 followed by a rinse in Bin #3
        2. Following the final rinse in Bin #3, place each part in a drying bin lined with paper towels to air dry.
        3. Each part must then be rinsed with acetone three times and methylene chloride three times before assembling for use on next tissue sample.

4. Reassemble the parts. Perform one final rinse using methylene chloride. Extract the next sample or store for later use.

2. Extraction Procedures
    1. Review the appropriate bench sheet provided. (Bench Sheets are dynamically created by Element.) to determine sample weight to be extracted, surrogate and spike additions, concentration techniques, clean-ups, final effective volume, reporting limits requested and any other special requirements or instructions.
    2. Obtain an Analyst Notes Form (3056F) and enter required data on the forms. Add header and sample identifications to the forms as appropriate.
    3. Find, remove and log out samples from their current location using Element's Sample Custody system and allow to warm to room temperature.
    4. Verify the client Identification's with the ARI labels. If the identifications do not match, inform the Organic Extractions Supervisor immediately.
    5. If Percent Solids is required, see ARI SOP 359S for details.
    6. Label each 250mL centrifuge bottle with the appropriate color label tape (see Table 02 below) for the analysis containing the following information: Batch or Work Order Number, sample ID number, and type of extraction eg. (Pest or PCB).
    7. Table 02

Table 02: Color coded Label Tape by Matrix and Analysis		
Analysis	**Matrix**	**Label Tape Color**
Pest	Tissue	Green
PCB	Tissue	Green
8270 PNA	Tissue	Green
SIM PNA	Tissue	Green
BAN; SIM BAN; Skydrol/BHT; 1,4 Dioxane	Tissue	Green
Low Level SIM PNA	Tissue	Green
TBT	Tissue	Red
% Lipids	Tissue	White

    8. **Note: All glassware or microwave vessels associated with TBT must be pre-rinsed with 0.10% Tropolone in Methylene Chloride. Note: TBT extracts must be put in HexMgBr Reagent the same day as extraction!**

3. **(TI-032)** Weigh sample for Tissuemizing
    1. Mix samples thoroughly to ensure that a homogeneous and representative aliquot is being used. **Note: Do not decant standing water from samples.**

2. Using a top loading balance, prepare two 250mL centrifuge bottles with anhydrous sodium sulfate volume listed on the bench sheet, one for the method blank and one for the LCS. Weigh appropriate amount of sample listed on the bench sheet into pre-labeled 250mL Centrifuge bottles.
3. Proceed to section 11.4 for spiking (TI-033). **NOTE: Do not Dry samples at this time.**

4. **(TI-033)** Spiking Samples for Tissumizing
    1. Add the appropriate volume of surrogate solution specified on the bench sheet to all samples including MB, BS, BSD, MRL, MS, and MSD. To verify that the surrogate spiking is accurate, the surrogate addition will be witnessed and documented by another laboratory technician. **(See SOP 3327S Appendix 20.4)**
    2. As samples are being surrogated, add enough of the appropriate solvent (see bench sheet) so the solvent layer is about a 1 inch above each sample for Tissumizing.
    3. Add the appropriate volume of spike solution specified on the bench sheet to the BS, BSD, MRL, and any MS, MSD sample. To verify that the matrix spiking is accurate, the spike addition will be witnessed and documented by another laboratory technician. **(See SOP 3327S Appendix 20.4)**.
5. **(TI-034)** Drying soil/sediment samples for Tissumizing.
    1. Add 40-50g of anhydrous sodium sulfate to dry the samples only immediately prior to Tissumizing that sample. **NOTE: Do not dry all samples at once, only add sulfate to the sample that is about to be Tissumized.**
    2. The samples are now ready for extraction by Tissuemizer. Proceed to 11.6. (TI-035)
6. **(TI-035)** Extracting samples by Tissuemizer
    1. Tissuemize the samples as follows
        1. Place the clean assembled shaft onto the tussuemizer and place the shaft into the sample bottle with the tip below the surface and into the tissue layer.
        2. Turn on the tissuemizer and adjust to maximum speed.
        3. Extract each sample with the tissuemizer for approximately 1 to 2 minutes mixing in an up down and around motion making sure the sample and sulfate is thoroughly blended and mixed.
        4. Turn off the Tissuemizer.
        5. Decant the extraction solvent through a 100mm funnel with a neutral glasswool plug in it. Pour the tissuemized extract into the corresponding pre-labeled 500mL Erlenmeyer flask with 100-150g of sodium sulfate at the bottom.
        6. Repeat steps 1-5 two more times using the appropriate solvent as indicated on the bench sheet.

7. After the Third extraction, transfer the sample to the funnel and rinse the 250mL centrifuge bottle with methylene chloride. Pour this rinse through the funnel, then rinse the funnel and extracted sample with methylene chloride. All rinses are collected in the 500mL flask.
8. Transfer the label with sample ID to the flask. Empty the Tissue into the buckets marked for solvent contaminated solids.
9. Clean the Tissuemizer as described in 11.2.2 (TI-031) between each sample.

2. Submit the samples extracted for concentration task as specified on the bench sheet
   1. Concentration and solvent exchange by Kuderna-Danish using SOP 3327S TI-008
   2. Concentration and solvent exchange by TurboVap using SOP 3327S TI-009
3. Concentrated extracts may require clean-up, hydrolysis, derivatization or fractionation as specified on the bench sheet. Extract treatment options/requirements are: **(See bench sheet details)**.
   1. TPH-D Sulfuric Acid/Silica Gel Clean-up using SOP 3327S TI-011S
   2. Pest or PCB Sulfuric Acid Clean-up using SOP 3327S TI-012
   3. Pest, PCB, or PNA Sulfur removal using SOP 3327S TI-013
   4. Silica Gel (SPE) Clean-up using SOP 3327S TI-014
   5. Manual Column Alumina Clean-up for TBT using SOP 3327S TI-015
   6. Manual Column EPH Fractionation using SOP 3327S TI-016
   7. Derivatization and Hydrolysis for TBT using SOP 3327S TI-017
   8. GPC Clean-up using SOP 306S
   9. Derivatization procedures for Sim PNA / PCP using SOP 3327S TI-018
   10. Determination of %Lipids SOP 3327S TI 036

2. Use the following Table 4 for extraction solvent:

Table 04-Tissuemizer Extraction/Rinsing Solvent Requirements		
Analysis	**Extraction Solvent**	**Rinse Solvent**
PEST	2x DCM/ACE 1x DCM	DCM
PCB	2x DCM/ACE 1x DCM	DCM
SVOA; 1,4 Dioxane; Skydrol/BHT	2x DCM/ACE 1x DCM	DCM
(8270) PNA	2x DCM/ACE 1x DCM	DCM
Sim PNA	2x DCM/ACE 1x DCM	DCM
LL Sim PNA	2x DCM/ACE 1x DCM	DCM
% Lipids	2x DCM/ACE 1x DCM	DCM
TPHd	3x DCM	DCM
TBT	1x 0.10% Tropolone 2x DCM	DCM

12. Data Analysis and Calculations

1. Not Applicable

13. Method Performance

1. QA maintains control charts for the recovery of surrogate standards and spiked compounds.
2. Management periodically reviews the charts to detect and correct any negative trends in analyte recovery.

14. Pollution Prevention

1. Do not discard solvent contaminated solid material into trash containers. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to a 55-gallon drum in the Hazardous Waste Room when the accumulation pails are full.
2. Disposed expired standards into appropriate Halogenated or Non-Halogenated Organic Solvent Waste drum, located in the Extractions Lab, as appropriate to solvent used for the standard.
3. Samples that are designated as hazardous waste by the LIMS “Hazardous Report” must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. ARI's Chemical Hygiene Plan discusses Hazardous Waste Disposal.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

1. Not Applicable

16. Corrective Actions for Out of Control Events

1. Promptly report any events that may compromise the extraction process to the Organic Extractions Supervisor who will take appropriate steps to insure data quality. Corrective actions may include, but are not limited to, notation on the Analyst Notes Form (3056F) or re-extraction of the sample.
2. Corrective action procedures for common laboratory issues are provided in the diagrams provided in Appendix 20.2.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

1. Unacceptable QA data noted during GC or GCMS analysis may result in a request for re-extraction. Re-extract parameters (sample volume, final volume etc.) may be modified from the original extraction based on analytical results.

18. Waste Management

1. Place the solids in the designated 5-gallon “satellite accumulation stations” located at various places in the laboratory. This includes spent sodium sulfate, glass wool, solid sample, silica gel and paper wipes. Transfer the solids to the 55-gallon drum labeled “Solvent Contaminated Solids” located in the Hazardous Waste Room when the accumulation pails are full.
2. Discard all Chlorinated waste solvent (solvent waste that contains Methylene Chloride) into the 55 gallon drum labeled “Halogenated Organic Solvent Waste” located in the Extractions Lab.
3. Discard all Non-Chlorinated waste solvent (solvent waste that does not contain Methylene Chloride) into the 55 gallon drum labeled "Non-Halogenated Organic Solvent Waste" located in the Extractions Lab.
4. Waste Solvents that you are unsure if they contain Methylene Chloride should be disposed of in the Halogenated Organic Solvent Waste drum located in the Extractions Lab.
5. Disposed expired standards into appropriate Halogenated or Non-Halogenated Organic Solvent Waste drum, located in the Extractions Lab, as appropriate to solvent used for the standard.
6. Place samples that designate as hazardous using the LIMS “Hazardous Report” in the designated drum in the Hazardous Waste Storage Area when they are disposed. ARI's Chemical Hygiene Plan discusses Hazardous Waste Disposal. Excess extracts and expired spiking solutions must be disposed of in the labeled Halogenated or Non-Halogenated Organic Solvent Waste drum, located in the Extraction Lab, as appropriate to the extract solvent.
7. ARI’s Laboratory Chemical Hygiene Plan (CHP) describes internal hazardous waste handling procedures. All analysts must be familiar with these requirements.

8. ARI properly profiles and disposes all hazardous waste using an EPA registered TSD (Treatment, Storage and Disposal) facility.

19. Method References

1. SW-846, “Ultrasonic Extraction”, Method 3550-C, Revision 3, February, 2007. USEPA Test Methods for Evaluating Solid Waste
2. Washington State Department of Ecology, “Extractable Petroleum Hydrocarbons”, June 1997.
3. Washington State Department of Ecology, “Analytical Methods for Petroleum Hydrocarbons”, June 1997.
4. Alaska Laboratory Method for the Analysis of Diesel Range Organics (DRO), AK102, November 7, 2002.
5. Alaska Laboratory Method for the Analysis of Residual Range Organics (RRO), AK103, November 7, 2002.
6. Krone (TBT), 1988.

20. Appendices

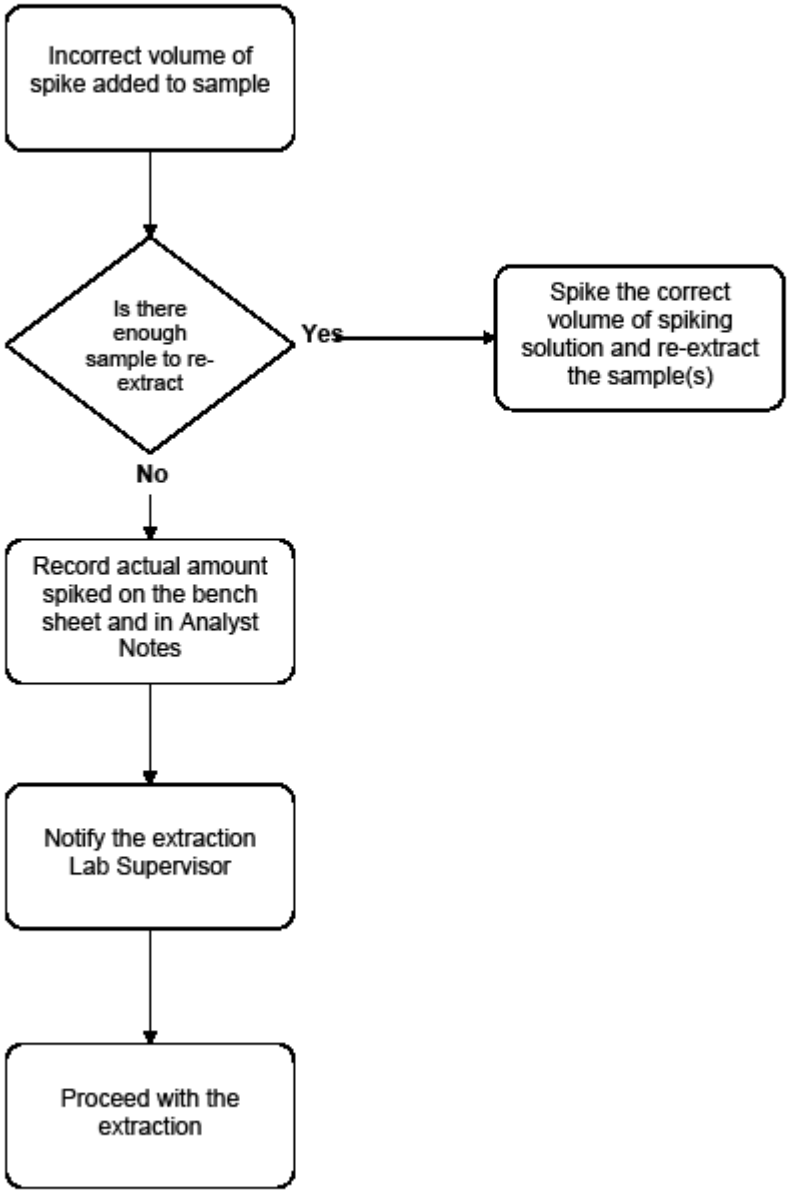
1. Extractions Performed Using SOP 3328S
2. Corrective Action Flow Charts

Appendix 20.1 Extractions Performed Using SOP 3328S							
Description of Analysis	**Bench Sheet**	**Extraction Technique**	**Sample Weight (g)**	**FEV (mL)**	**Solvent**	**Concentration Technique SOP and TI#**	**Cleanup-Separation-Derivatization-Hydrolysis (TI#/or SOP)**
8082A PCB (20ppb)	3027F	Tissuemizer	5g	5.0	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S Acid SOP 3327S TI 012
8082A PCB (4ppb)	3030F	Tissuemizer	12.5g	2.5	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S Acid SOP 3327S TI 012
Pest	*	Tissuemizer	12.5g	2.5	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S
Sim PNA (0.1-5ppb)	3323F	Tissuemizer	10g	0.5	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S Silica Gel Manual Column SOP 3327S TI 016
SVOA (20-200ppb)	3335F	Tissuemizer	10g	1.0	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S
%Lipids	3074F	Tissuemizer	5g	N/A	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	Desiccator Drying SOP 3327S TI 036
Sim PNA Low	3320F	Tissuemizer	10g	0.5	2x DCM/Ace 1x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	GPC SOP 306S Silica Gel Manual Column SOP 3327S TI 016
TBT	3336F	Tissuemizer	10g	1.0	1x 0.10% Tropolone 2x DCM	KD SOP 3327S TI 008 TurboVap SOP 3327S TI 009	Hydrolysis/Derivatization SOP 3327S TI 017 Manual Alumina Column SOP 3327S TI 015

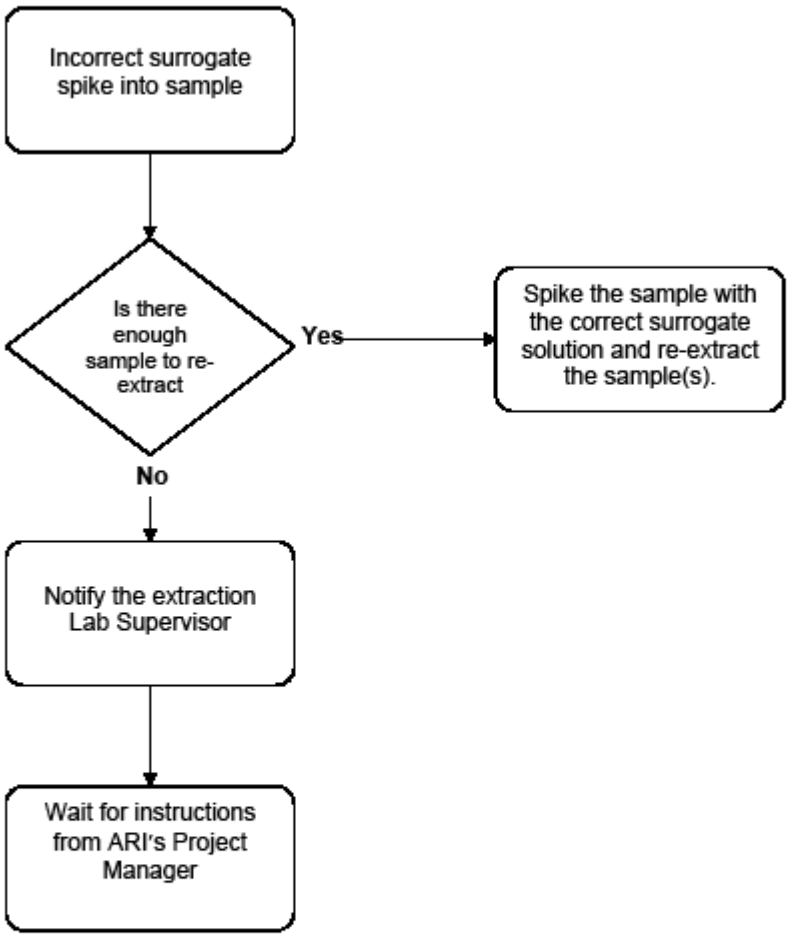
*Other Bench sheets built on request

Appendix 20.2
Corrective Action Charts

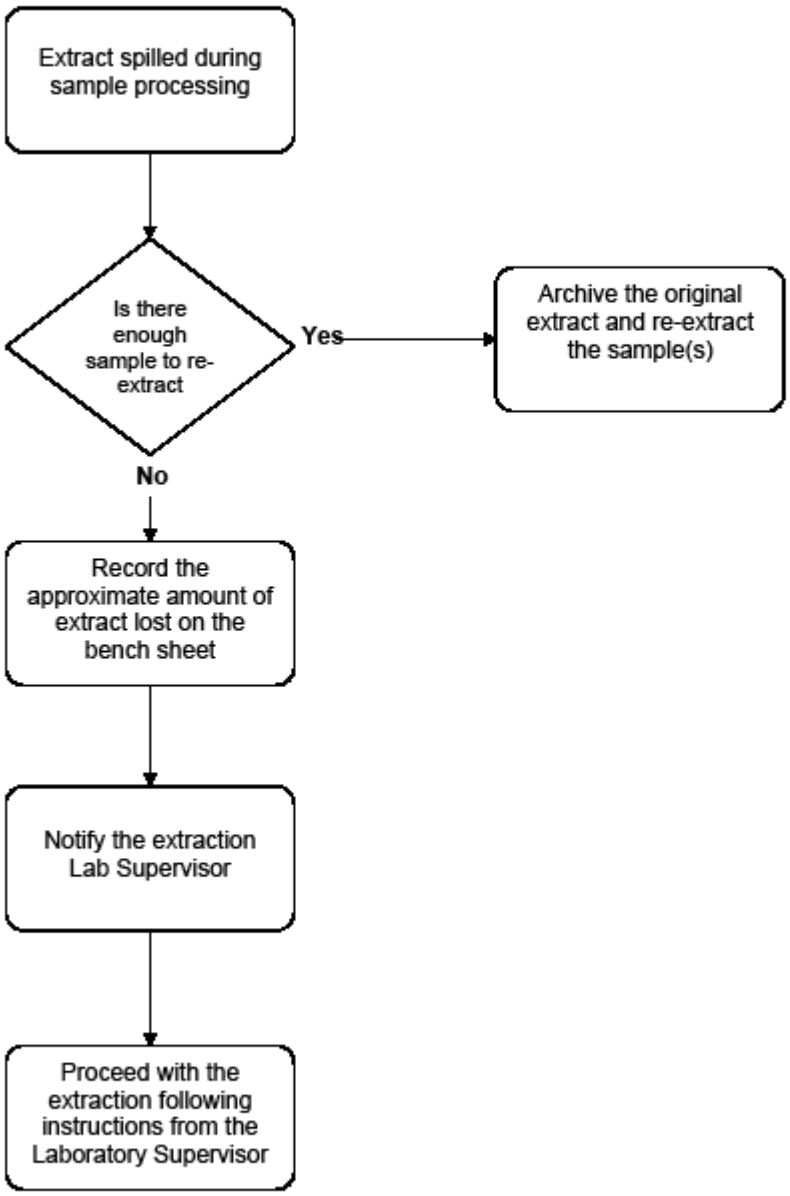
Corrective Action for Incorrect Spike Volume



Corrective Action for Incorrect Surrogate Addition



Corrective Action for an Extract Spill



Appendix D.5
Brooks Applied Labs Arsenic Speciation by IC-ICP-MS Standard Operating Procedure

SOP #BAL-4100

As Speciation for Waters, Tissues, and Sediments (Basic Anion Exchange) by Ion Chromatography - Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)

Brooks Applied Labs

Effective Date: 05/03/2021

Issued By: Apr 19, 2021

Reviewed HelenaNVu HelenaNVu (Jul 26, 2022 09:29 PDT) Jul 26, 2022

Hakan Gurleyuk (Apr 19, 2021 07:50 PDT)
Technical Director — Date: Apr 19, 2021

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HelenaNVu
HelenaNVu (Apr 19, 2021 06:55 PDT)
Chemist — Date: Apr 19, 2021

As Speciation for Waters, Tissues, and Sediments (Basic Anion Exchange) by Ion Chromatography - Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)

1.0 DESCRIPTION

1.1 Definition: This standard operating procedure (SOP) describes the determination of arsenite, arsenate, monomethyl arsonic acid, cacodylic acid, and other anionic/cationic arsenic species in waters and extracts (by BAL-4111, BAL-4112, BAL-4115, or BAL-4117) using ion chromatography coupled to an inductively coupled plasma - mass spectrometer.

1.2 Scope: This method is developed to detect arsenic species in the range of 0.020 – 20.0 ug/L (ppb). Samples that have higher concentrations can be diluted to extend the range of the method.

1.3 Summary: Aqueous samples and extractions are filtered through a 0.45-µm filter. An aliquot of sample is injected onto an anion-exchange column. A trap column removes impurities from the eluent and pump. The analytical column separates the anionic arsenic species according to their interactions with the column resin. The mass-to-charge ratio (m/z) of As at mass 75 is monitored and the area under the arsenic peaks are used for quantitation. Selenium at [redacted] is monitored as an internal standard. It is highly recommended that total filtered arsenic accompany all arsenic speciation analyses to ensure all arsenic species are accounted for in the filtered sample.

2.0 DEFINITIONS

2.1 Arsenite – As(III)

2.2 Arsenate – As(V)

2.3 Monomethyl arsonic acid – MMA

2.4 Cacodylic acid – DMA

2.5 Arsenobetaine – AsB

2.6 Trimethylarsine oxide – TMAO

3.0 INTERFERENCES

3.1 Samples with high levels of Fe, Mn, and Al may precipitate at the column head and cause removal of arsenate and possibly other arsenic species in the sample. Samples containing elevated concentrations of these metals must be diluted

accordingly or another separation method that prevents precipitation of these elements should be applied.

3.2 ICP-MS is prone to spectral interferences. These types of interferences result from the formation of polyatomic species in the plasma interface that have the same mass-to-charge ratio as the analyte of interest. Since arsenic is mono-isotopic (100% abundance at mass 75), formation of the $^{40}Ar^{35}Cl^{+}$ species can be a significant source of interference. Even though this species is resolved from most target arsenic peaks during the ion chromatographic separation, it should be kept in mind that there may be other compounds that contain chlorine, and that they may co-elute with some arsenic species. Therefore, it is recommended to include chlorine in the analytical method. Most of these interferences are eliminated if the ICP-MS instrument is operated with interference reduction technology (IRT).

3.3 AsB and TMAO coelute and so this method should not be used to quantify either AsB or TMAO.

4.0 SAFETY

4.1 The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Safety data sheets (SDS) are maintained for all chemicals used in this method. The SDS are stored at BALLAB\Safety\SDS.

4.2 [redacted] is very corrosive, toxic, and harmful if swallowed, inhaled, or absorbed through skin. It is extremely destructive to the tissues of mucous membranes, eyes, and skin. Users must wear lab coats, safety glasses, and gloves. [redacted] must be handled in a well-ventilated area.

4.3 [redacted] is slightly hazardous to the skin and eyes. Users must wear lab coats, safety glasses, and gloves.

4.4 As(III), As(V), MMA, and DMA have been confirmed as a human carcinogen and possible mutagen. It may cause allergic skin reactions. Users must always wear lab coats, safety glasses, and gloves.

4.5 Analytical plasma sources emit radiofrequency radiation in addition to intense UV radiation. Suitable precautions should be taken to protect personnel from such hazards. The shield on the spray chamber must be kept attached at all times during the plasma operation and the inductively coupled plasma should only be viewed through the viewing window.

4.6 As with any electronic apparatus, great care should be employed to reduce the probability of an electrical shock. Only properly trained and approved (by the Technical Director) BAL employees should attempt to service the RF generator for the instrument. Or a licensed technician may perform any electrical work associated with an ICP-MS; otherwise, severe shock or death may occur.

4.7 Refer to the Chemical Hygiene Plan (CHP) for additional safety precautions and required protective equipment.

5.0 Equipment and Supplies

5.1 Agilent 7700/7800 inductively coupled plasma - mass spectrometer (ICP-MS) which is equipped with a collision reaction cell, reducing spectral interferences.

5.2 Computer capable of running the chromatographic software:

5.2.1 [redacted] software from Dionex to control the pump and the autosampler

5.2.2 MassHunter from Agilent to analyze the data.

5.3 Ion chromatographic system consisting of the following components:

5.3.1 Dual-piston pump ([redacted], or equivalent), capable of generating a quaternary gradient.

5.3.2 Autosampler ([redacted] or equivalent).

5.3.3 Autosampler vials ([redacted]).

5.3.4 6-port switching valve ([redacted] or equivalent).

5.3.5 [redacted]-mL ([redacted]) sample loop (cut to volume from green PEEK tubing).

5.3.6 [redacted] sample loop for column cleaning (cut to volume from green PEEK tubing).

5.3.7 Eluent bottles (2, 4-L capacity).

5.3.8 Chromatographic column (such as a [redacted]).

5.3.9 Trap column (such as a [redacted]).

5.3.10 Peristaltic pump (such as a Gilson Minipuls3 or equivalent) to pull the waste from the spray chamber to the waste vessel.

[redacted]

5.4 Waste container, 5 gallons.

5.5 15-mL plastic centrifuge tubes.

5.6 10-mL disposable syringes with Luer lock.

5.7 PES syringe filters (0.45um) known to be free from any arsenic species.

5.8 Pipettes: All plastic pneumatic fixed volume and variable pipettes in the range of 5µL to 5.0mL.

5.9 SOPs BAL-0020, BAL-0023 and BAL-0600 describe the routine preventative maintenance for the equipment used in this procedure.

6.0 REAGENTS AND STANDARDS

6.1 Reagent Water – deionized water (DIW) tested to be free from the arsenic species of interest.

6.2 Stock Solution Stability and Storage – Unless otherwise specified, all arsenic species stock solutions are stable for at least 5 years, however, the stability of each stock solution species is not well understood; therefore, all stock solutions should be tested routinely to confirm the species information and concentration to the certified values (see BAL-0104). Standards should be stored refrigerated.

6.3 Standard Reference Material 3036-Arsenic Acid (AsV) standard solution – This SRM can be purchased from NIST. This standard is stable for at least 5 years, however, the stability of this SRM is not well understood; NIST will monitor this SRM over the period of its certification on NIST website. Standards should be stored refrigerated. This will help in preventing standard degradation.

6.4 Standard Reference Material 3037-Arsenic Acid (AsIII) standard solution – This SRM can be purchased from NIST. This standard is stable for at least 5 years, however, the stability of this SRM is not well understood; NIST will monitor this SRM over the period of its certification. Standards should be stored refrigerated.

6.5 Cacodylic Acid Stock Solution (DMA) – Cacodylic acid standards can be purchased 10 mg/L from [redacted] or prepared from stock pure solid materials. Solid Cacodylic acid can be purchased from [redacted]. A 1000 mg/L solution can be prepared by adding [redacted] of DMA to a clear 40mL borosilicate

glass vial. The vial should be brought to volume (40mL) with reagent water on a 4-point scale.

6.6 Monomethyl Arsonic Acid Stock Solution (MMA) – Monomethyl arsonic acid standards can be purchased 1000 mg/L from [redacted] or prepared from stock pure solid materials. Solid MMA standards can be purchased from [redacted]. A 1000 mg/L solution can be prepared by adding [redacted] of MMA to a clear 40mL borosilicate glass vial. The vial should be brought to volume (40mL) with reagent water on a 4-point balance.

6.7 Daily Stock Solution (DSS) - A 1000 µg/L daily stock solution for As(III), As(V) and MMA is prepared by adding 9.95mL of diluent (6.21) to a 15mL plastic vial. Then add 10 µL of the 1000 mg/L As(III), As(V), and MMA stock solutions (6.3, 6.4 and 6.6). Diluent must be added prior to the spikes to prevent conversion or precipitation of the individual species. This solution is then used to prepare the Daily Calibration Solution and is made fresh weekly. DSS is prepare based on concentration of the stock standards.

6.8 Daily Calibration Solution (DCS) - A 20 µg/L daily calibration solution for As(III), As(V), MMA, and DMA is prepared by adding 300 µL of the 1000 µg/L Daily Stock Solution (6.7) and 30 µL of 10 mg/L DMA (6.5) to final of 15.0 mL of diluent (6.21) in a 15 mL plastic vial. Diluent must be added prior to the spikes to prevent conversion or precipitation of the individual species. This solution is then used to prepare the daily calibration standards and is made fresh on the date of analysis. DSS is prepare based on concentration of the stock standards.

6.9 As(III) Initial Calibration Verification Stock Solution – A second source 1000 mg/L solution can be purchased from [redacted].

6.10 As(V) Initial Calibration Verification Stock Solution – A second source 1000 mg/L solution can be purchased from [redacted].

6.11 DMA Initial Calibration Verification Stock Solution – Second source cacodylic acid standards can be purchased or made from stock pure solid materials. Solid [redacted] can be purchased from [redacted]. A 1000 mg/L solution can be prepared by [redacted] to a clear 40mL borosilicate glass vial. The vial should be brought to volume (40mL) with reagent water (6.1) using a 4-point balance. The true value DMA in this solution [redacted].

6.12 Initial Calibration Verification Daily Solution - A 1000 µg/L ICV daily stock solution for As(III), As(V), and DMA is prepared by adding 10 µL of each 1000 mg/L stock solutions (6.9, 6.10 and 6.11) into 9.95 mL of diluent (6.21) in a 15-mL plastic tube.

6.13 MMA Initial Calibration Verification Stock Solution – Second source Monomethyl Arsonic acid standards can be purchased 10 mg/L from [redacted] or prepared from stock pure solid materials. Solid [redacted]. A 1000 mg/L solution can be prepared by adding [redacted] to a clear 40mL borosilicate glass vial. The vial should be brought to volume (40mL) with reagent water using a 4-point balance. This solution is then used to prepare the daily MMA ICV standard. This standard has a slight As(V) impurity and thus must be spiked separately from the As(V) standard.

6.14 Initial Calibration Verification Daily Solution for MMA - A 1000 µg/L ICV daily stock solution for MMA is prepared by adding 4.5 mL of diluent (6.21) to a 15 mL plastic tube and adding 500 µL of 10 mg/L MMA stock solution (6.13).

6.15 Methanol – HPLC Grade methanol purchased from a reputable vendor.

6.16 [redacted] ultra-pure electronic grade [redacted] solution purchased from a reputable vendor.

6.17 [redacted] eluent ([redacted] Dilute to the 4-L mark with reagent water (6.1).

6.18 [redacted] Eluent ([redacted]

6.19 [redacted] – can be purchased from Alfa Aesar.

6.20 Internal Standard Stock Solution Enriched Selenium 82 (^{82}Se) – A 10 mg/L solution can be purchased from [redacted].

6.21 Diluent – [redacted]. Dilute to the 1000 mL mark with reagent water (6.1).

6.22 Extract solution – [redacted] (Section 10.2.2)

6.23 Standard Reference Material (SCV1) TMDA- 50.5 – Trace elements in Natural Water – This reference material is also used for the verification of the calibration curve in addition to the ICV solution. While this SRM is preserved with 0.2% HNO_3 and only certified for total arsenic, the arsenic speciation results can be compared to the certified value of total arsenic to confirm the accuracy of the method. A 100 uL aliquot of this SRM is diluted to 5 mL in an autosampler vial with the diluent (6.20). Historical data suggest that the primary arsenic species in this SRM are As(III) and As(V).

6.24 Argon gas - (standard laboratory grade).

6.25 High purity (99.999% minimum purity) Helium gas – Impurities filtered out by a gas clean filter purchased through Agilent.

6.26 Optimization Solution. Using 1000mg/L stock standards of As, Se, Li, Co, Mg, Y, Ce, and Tl make a 10ppb solution to be used for tuning and optimizing the instrument. Add approximately 800mL of 1% HNO_3, 1% HCl to a 1-L bottle, then add 10 µL of each 1000 mg/L stock. Bring up to volume (1L) with 1% HNO_3, 1% HCl.

7.0 SAMPLE COLLECTION, FILTRATION, PRESERVATION, AND STORAGE

7.1 Since the method is only applicable to soluble species of arsenic, samples should be filtered prior to preservation and must be filtered prior to analysis. It is strongly recommended that filtration is performed in the field.

7.2 The sample (60-250 mL) is collected directly into a pre-tested, high-density polyethylene (HDPE) container using sample handling techniques specially designed for collection of arsenic at trace levels. Research has shown that amber HDPE bottles have a propensity to retain certain anionic molecules; therefore, they should NEVER be used for sample collection associated with arsenic speciation or total arsenic analysis.

7.3 Anoxic samples should be collected in 6-mL vacuette containers pre-preserved with [redacted]. Once collected, the sample container is gently inverted several times to fully solubilize the [redacted] before chilling the samples and shipping overnight.

7.4 As(V) is known to co-precipitate with iron, manganese, aluminum, and other trace metals; therefore, historical analysis of the samples for these analytes is highly recommended. Temperature depression decreases the solubility of trace metals in solution; therefore, sample matrices containing elevated concentrations of other trace metals, especially iron, should be field preserved with a buffered solution (using acetic acid to maintain a pH of 4) of [redacted]. The [redacted] chelates with the iron which inhibits co-precipitation. Samples with extremely high

concentrations of iron (greater than 10,000ppm) may necessitate field preservation with degassed HCl.

7.5 Oxidation of As(III) to As(V) can be induced by significant changes in pH and ORP. Samples which are naturally in an anoxic environment (low pH and low dissolved oxygen) must be collected in an anoxic atmosphere to prevent species conversion prior to analysis. It may also be recommended that field preservation with a buffered [redacted] solution is applied for sample matrices with elevated concentrations of iron. Oxidation of ferrous to ferric iron can also be induced by significant changes in pH and ORP which can result in precipitation of insoluble iron hydroxides.

7.6 MMA and DMA are typically formed through the metabolic mechanisms of biological activity. Sample matrices rich in bacteria and other biological organisms may convert inorganic arsenic [As(III) and As(V)] to organic arsenic during sample transport to the laboratory. To reduce the possibility of methylation of inorganic arsenic species for these sample matrices, samples should either be field filtered (using a 0.45µm filter) or preserved with degassed HCl to either remove or inhibit biological activity.

7.7 Aqueous samples should be stored in a refrigerator at 0 – 6 ºC, unfrozen until analysis can commence. Cryofreezing is not recommended for arsenic speciation.

7.8 Sediments undergoing arsenic speciation should be frozen to mitigate any microbial activity or temperature dependent reactions that may induce isotope conversion. These samples should undergo extraction following BAL-4111 and BAL-4112. The aqueous extracts can then be analyzed following this SOP.

7.9 Biotas undergoing arsenic speciation should be frozen to mitigate any microbial activity that may induce isotope conversion. These samples should undergo extraction by BAL-4115. The aqueous extracts can then be analyzed following this SOP.

8.0 CALIBRATION AND STANDARDIZATION

8.1 A calibration curve must be generated at the beginning of each sequence by analyzing various standards that range between 0.018 µg/L and 20 µg /L. Calibration standards are prepared by diluting the daily calibration solution in 5-mL autosampler tubes to 5 mL with diluent (6.21). The standards to be prepared are 0.018, 0.20, 0.50, 2.00, 5.00, and 20.00 µg/L using the Daily Calibration Solution (6.8). ***CAL1 can be a serial dilution from CAL4 due to low aliquot of DCS.

Cal Solution	Concentration (ppb)	Standard	Aliquot (mL)	Final Volume (mL)
SEQ-IBL	0	Diluent	5	5
SEQ-CAL1	0.018	CAL4	0.045	5
SEQ-CAL2	0.200	DCS (6.7)	0.050	5
SEQ-CAL3	0.500	DCS (6.7)	0.125	5
SEQ-CAL4	2.00	DCS (6.7)	0.500	5
SEQ-CAL5	5.00	DCS (6.7)	1.25	5
SEQ-CAL6	20.0	DCS (6.7)	5.00	5

8.2 Calibration curves must conform to linear regression plot with an R value greater than or equal to 0.995. A minimum of 5 data points must be applied to generate a calibration curve. If the calibration curve does not conform to a linear regression plot, the cause must be investigated prior to pursuing sample analyses. The high calibration point may be dropped if recovery criteria is not met and 5 points remain. Low calibration points that do not meet recovery criteria are not removed as the high calibration points are weighed heavily and removal does not affect the slope. Only results with concentrations between passing calibration points can be reported quantified. Mid-level calibration points must pass recovery criteria and cannot be dropped without a scientifically sound reason.

8.3 The calibration is calculated in MassHunter. The nature of IC-ICP-MS allows for a species independent calibration meaning that all arsenic species will generate the same response factor at the instrument. The relative standard deviation between the calibration slopes should be less than 10%. In cases where the RSD between slopes exceed 10%, the most common reasons are mis-prepared eluents (different amounts of MeOH in each bottle), improperly optimized instrument or incorrect standard. Although it is also possible that the stock standards are bad, this is highly unlikely. If the ICV and SCV confirm the calibration for each species, data can be reported.

8.4 All unknowns are quantified by substituting in the DMA calibration curve as these species are not used in the calibration. DMA is the species that is the least likely to convert with pH changes (ie. extractions).

9.0 QUALITY CONTROL

9.1 Instrument optimization is a crucial part of the quality control. The ICP-MS instrument should be optimized for arsenic before analysis is begun.

9.2 Each set of 20 samples must have at least one associated preparation (or method) blank (BLK). For each batch of up to 80 samples, four BLKs are prepared. These are treated the same as the samples and must go through all preparation steps. For samples that were preserved with preservative from BAL, prep blanks must be created at the same percentage of preservative as the samples

at the time of analysis. Prep blanks associated with unpreserved samples are created using only diluent (6.21).

9.3 The percent recovery of the ICV standard must be 80-120% for As(III), As(V), MMA, and DMA. If the percent recovery of the ICV is not within the control limits of 80-120%, results may not be reported from that run since there are no Certified Reference Materials available to validate the calibration.

9.4 Blank Spike (BS) 1 per batch – A BS is prepared per BAL-4115 for TMAH extracts and per BAL-4111 or BAL-4112 for soil extracts. BS for water batches are prepared the same as the ICV, 1 BS is spiked for As(III), As(V), and DMA and a 2nd BS for MMA. The recovery of each spiked species in the BS should be 75-125%.

9.5 The summed percent recovery for As(III) and As(V) for the SCV1 (TMDA- 50.5 SRM (6.21)) must be 80-120% of the certified total arsenic value. This SRM is not certified for speciation analyses but is certified for total arsenic. Historical data suggest that the primary arsenic species in this SRM are As(III) and As(V).

9.6 Continuing calibration verification (CCV) is performed immediately after the calibration, after every 10 injections, and at the end of the analytical run. A 1 µg/L CCV standard is prepared by diluting 250 µL of the 20 ug/L daily calibration solution (6.8) in 5 mL of diluent (6.21) into the autosampler vial. The recovery criteria for the CCV standards is 75-125%.

9.7 Continuing Calibration Blanks (CCB) should be monitored for the effects of carry-over. If the concentration of any arsenic species is higher than the MRL the sample results should be carefully reviewed for possible carry-over effects. Carry-over is usually observed due to high matrix samples so all samples before the failing CCB should be investigated to identify issues with matrix. If the samples are less than 10x more in concentration than the contaminated CCB, both brackets surrounding the failed CCB should be reanalyzed.

9.8 Matrix duplicate (DUP) sample analysis must be performed at a frequency of 10% per batch and for each unique submatrix. The relative percent difference (RPD) for the replicates should be ≤ 25% if the sample concentrations are greater than 10 times the MRL at the instrument. If the source and DUP concentrations are less than 10x the MRL and within 1 MRL for waters or 2 MRLs for solids of each other than the variance can be narrated.

9.8.1 If limited sample volume or mass restricts the application of quality control a matrix spike duplicate (MSD) may be substituted for the DUP, with the same control limit applying. If the RPD for the replicate analyses exceeds 25%, the source samples should be visually inspected for heterogeneity. If the cause of the variability cannot be ascertained, but

other supporting quality control can identify that the variability is not representative of the samples or the analysis, replicates should be reanalyzed for confirmation purposes. If the reanalysis does not confirm the initial analysis, all associated samples must be reanalyzed along with the corresponding quality control. If the reanalysis confirms the original analysis, the variance can be narrated. The project manager must be consulted during the entire investigative process and all corrective actions should be recorded on the respective sequence notes.

9.9 A matrix spike (MS) and a matrix spike duplicate (MSD) analysis should be performed for each target species at a frequency of 10% per batch and for each unique submatrix. The percent recovery of the spike is 75-125% with an RPD ≤ 25%. If the spike recovery is out of the control limits the project manager should be consulted. Samples should be spiked 1 – 20x the source concentration if historical data is available, or at the level of the CCV, whichever is greater.

9.10 With speciation analysis, it has been observed that the sample matrix can induce changes in the speciation of added arsenic spikes. If a mass balance cannot be determined, a fresh source/DUP/MS/MSD should be reanalyzed for confirmation purposes. If the reanalysis of the source/DUP/MS/MSD is within quality control limits, all samples and the corresponding quality control must be reanalyzed. If the reanalysis confirms the initial results the samples should be reprepared and the source sample concentrations should be referenced to identify appropriate spike concentrations. For aqueous matrices, if the sample concentration levels are greater than the spike concentration, the QC sample and all other samples of similar matrix may be diluted to reduce the matrix effect, followed by reanalysis of all associated samples and quality control parameters.

9.11 The internal standard is present in the diluent and so expected recoveries are dependent on the analytical dilution. Injections at ≤ 10x use less diluent, therefore the expected recoveries are adjusted for the ratio of diluent used (e.g. at 2x, 50% of the injection is diluent). Internal standard recoveries are used to evaluate potential misinjections, especially if the recoveries are very low. Selenium is a naturally occurring element and high internal standard recoveries may indicate that the sample contains selenium. For this reason, data is generally not internal standard corrected.

10.0 PROCEDURE

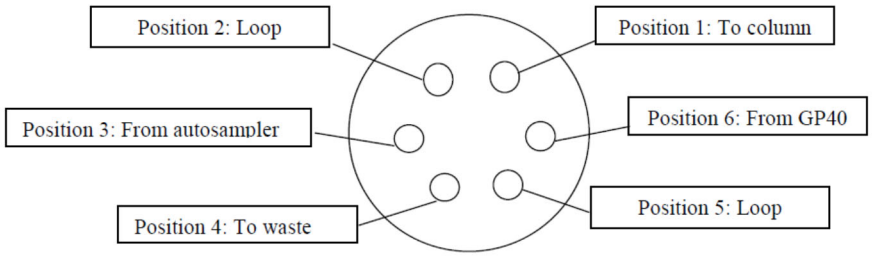
10.1 *Instrument Set Up*

10.1.1 Make new eluants if necessary, load [redacted] pump method, and connect eluant lines to the pump. [redacted]

10.1.2 Prime the eluant lines by opening the transducer knob on the [redacted], set the method to direct control, and change the gradient to 100% of eluant line A. Select the prime button. Allow the pump to prime for 3-5 minutes. Press off. Switch the gradient to 100% of line B, prime, then repeat with lines C and D.

10.1.3 Each week, the switching valve must be cleaned. Disconnect all lines from it and unscrew the rotor face assembly. Rinse the stator, stator face assembly, and the rotor seal under DIW. Check to ensure that each port hole is clear of debris- if it is not continue to rinse with water. Carefully dry each piece under argon gas. Retighten the components and attach lines to each port following Figure 1.

Figure 1. Six port Rheodyne switching valve port map.



10.1.4 Attach the [redacted] column between the [redacted]. Ensure that the flow is heading the correct direction. [redacted] between the switching block and the instrument. Ensure that the flow is heading the correct direction. Attach a waste line to the end of the column and place the other end into the waste container.

10.1.5 While the pump is priming, prepare the column cleaning solutions in Dionex 5mL vials. These are as follows: [redacted] In PeakNet, load the appropriate column cleaning method (i.e. - 5 minute method (131004-IC-5minGP999.sch). Load the racks into the autosampler, connect the cleaning loop to the right ports of the injection valve and press Run.

10.1.6 Keep the [redacted] in direct control. Switch the eluent proportions to be [redacted]. Select on. Pump pressure should be around 2000 psi. Under the PeakNet Run screen, select Run>Start. The first vial of 10% HNO3 should inject now.

10.1.7 While the column is being cleaned, the ICP-MS should be readied for analysis.

10.1.8 Cleaning the cones is part of regular maintenance. It is required once per month to maintain sensitivity and should be done if issues with sensitivity arise. This will also allow the analyst to regularly inspect the condition of the orifice. To access the cones, press the Maintenance button on the left wall of the Interface Chamber until the torch box moves out of the way. Using Agilent tools, unscrew the cones. The procedure for cleaning the cones is described in the Hardware maintenance Manual for Agilent Instruments (W:\Methods_&_Papers\Agilent manuals\Hardware Maintenance Manual.pdf). In short, cones are cleaned by sonication with DIW, 2% Citranox (or Barkeeper's friend), respectively for 3-5 minutes. While most of the debris should be removed with this, if there is significant debris is visible on the cone, fine alumina powder can be mixed with bar keeper's friend and applied with a Q-Tip. If none of these methods clean the cones, a Q-Tip can be dipped into a 2% HNO3 solution and used to scrub the cone gently followed by sonication in DIW for 3-5 minutes. **Never sonicate the cones in an acid solution.** Rinse under DIW and dry under argon.

10.1.9 Inspect the HMI tube and torch for debris. If there is any present, carefully remove the HMI tube and torch and rinse under DIW. It is likely necessary to insert a pipe cleaner into the injector tube on the torch to clear the debris. Once cleared, rinse with MeOH, which evaporates quicker than water. Carefully dry the tubes under argon. Reinstall the HMI tube and torch.

10.1.10 Once the column cleaning is finished, switch the pump from Direct Control to the appropriate method following Table 1 in the Appendix. Connect the end of the [redacted] column to the spray chamber. In PeakNet under Run, select Run> Abort. This will stop the ACI.

10.1.11 Open the most recent As Spec (Basic Anion Exchange) run in MassHunter. Check that masses 75, and 82 are selected in the Realtime display. Save this with the sequence number for the run followed by the SOP number, i.e. S211234-BAL 4100.

10.1.12 Follow the instructions given in SOP BAL-5000 for the optimization of the instrument.

10.2 *Running Samples*

10.2.1 While the instrument is optimizing, begin preparing the calibration in 5mL Dionex autosampler vials. After the samples are loaded into the autosampler, select Run on the autosampler (make sure the autosampler does not start loading the sample immediately which suggests an ACI program is still running).

10.2.2 Load the schedule from the analytical benchsheet into MassHunter. The analytical benchsheet should start with 3-5 replicates of [redacted], 2 CCVs and a low-level calibration. Save this and select Add to Queue. Open PeakNet and load the correct schedule (i.e. 131004-IC-15minGP999.sch). Select Run>Start. The first sample will inject now.

10.2.3 Monitor the system until 2.4 minutes to catch any errors. Make sure that pressure is normal and consistent, there are no leaks, the switching block switches, there is signal at the instrument at 2.4 minutes, proper peak separation, and good sensitivity. Since the IC method is a gradient method first injection may not provide accurate information for separation, so it is important to inspect the separation with the second injection.

10.2.4 Prepare the client samples in 5mL Dionex vials according to the dilutions on the analytical benchsheet.

10.3 *Peak Integration*

10.3.1 Double click on the IcpDataAnalysis icon to open the Offline Mass Hunter Workstation. Select “batch” from the toolbar. Select “new batch folder” and save as the sequence number + “-crunch” ex: “S211234-BAL 4100-crunch”.

10.3.2 Select “file” and select “import samples” from the intended sequence. Highlight all samples to be imported and select “open” and “Ok”. A status bar should show, it can a few minutes for the samples to import depending on the size of the file. After the import is complete, click on “Process Batch” button on the toolbar.

10.3.3 Click on the “DA Method Editor” button. Select “import DA Method Only” from the menu on the left. Select a previous As Speciation Crunch file and import the data analysis settings.

10.3.4 Click on “Open Data File” and select the ICV. The sub-window at the bottom should display the chromatogram for the ICV. On the top left side of the chromatogram, select mass 75. Check the retention times of each species on the “Integration Parameters” window and update as necessary. Click on each peak on the “peak” table and make sure that the peak area counts threshold is set to a very large number (>2,000,000,000,000). This makes sure that no peaks are integrated by the software so each peak is manually integrated by the analyst. If the peak area threshold is not set (defaults is 23,000), enter 2000000000000 into the peak area threshold box and click on “apply to all”

10.3.5 Click on "Full Quant" on the left menu and make sure all standard values are entered in correctly. Note that AsB and TMAO and all other unknown species are quantitated using "Compound Independent Calibration-CIC" so the CIC check box should be checked above the table. To assign a calibration to these species, on the substitute column, select DMA.

10.3.6 Click on "Return to Batch at-a-glance" which should ask if you would like to update the DA method. Select yes if changes were made. After exiting the DA Method Editor, the "Process Batch" button should be RED. Before beginning to integrate peaks, the whole batch must be processed with the new DA Method. Failure to do so, may cause the software to crash and lose any integrations already performed. It is highly recommended to click on the "Process Batch" button as frequently as possible followed by saving the batch file in case the software crashes.

10.3.7 Properly integrate each peak present in each chromatogram excluding "blobs". See BAL-4000 for information on peak integration.

10.3.8 In Batch Table, right-click on the sample header and select Add/Remove Columns. In the dialog box, add "level" and "type" to the box on the right and click OK. Select the appropriate levels and types for calibration and blank. SEQ-ICB1 is type CAL Blk and Level 1. SEQ-CAL1 through 6 are type CAL Std and levels 2-7. Click on "Process Batch" data to calculate concentrations based on the curve. Check the curve recoveries, R values and RSD between the calibration slopes.

10.3.9 If calibration passes QC requirements, scan the whole batch for irregularities that may need re-analysis (mis-injects, failing CCVs, high blanks, etc). If no problems can be found, proceed with data upload.

10.4 *Data Upload*

10.4.1 Once the peaks have been integrated, reprocessed, and saved, ensure that the proper columns are present: Rjt, Acq. Date-Time, Data File, Comment, Sample Name, Dilution, all analytes of interest in units conc. (ug/l), and ISTD counts.

10.4.2 Highlight table and select Export table from the drop down menu. This creates an Excel file. Save this file under BALLAB>Data>YY_Data >Analytical>Sequences> "Assigned sequence number" folder using "Assigned sequence number-BAL4100-Raw Data" as the file ID.

10.4.3 If requested by client, calculate Inorganic Arsenic (As(III)+As(V)), the Sum of Unknowns and Count of Unknown Species in Excel. If the concentration of any unknown is less than the MDL, it must be excluded

from the sum and count calculations. This modified Excel file is then saved under Data to Upload to LIMS and is then uploaded into LIMS.

10.4.4 See BAL-0103 for information on data upload.

11.0 POLLUTION PREVENTION

11.1 Whenever feasible, lab personnel should use pollution prevention techniques to limit waste generation. The cost involved in purifying used acids makes such recycling unpractical at BAL. Instead, every effort is made to reduce any volumes necessary to still produce the best possible results. This analysis requires very small volumes to analyze sample preparations and standards. Standards should be prepared in volumes consistent with the laboratory use to minimize the volume of disposed standards to be disposed.

12.0 WASTE MANAGEMENT

12.1 All waste is disposed of in accordance with state and federal regulations either by sewer disposal (only if concentrations are less than the King County sewer limits) or through a licensed hazardous waste disposal facility according to BAL-2003.

12.2 All sample waste with unknown concentrations of metals are collected in satellite waste containers which are tested and disposed according to BAL-2003

13.0 REFERENCES

13.1 Goldberg, S. and Johnston, C. “Mechanisms of Arsenic Adsorption on Amorphous Oxides Evaluated Using Macroscopic Measurements, Vibrational Spectroscopy, and Surface Complexation Modeling” Journal of Colloid and Interface Science. 234: 203-216.

13.2 Bednar, A., Garborino, J., Ranville, J., and Wildeman, T. “Preserving the Distribution of Inorganic Arsenic Species in Groundwater and Acid Mine Drainage” Environ. Sci. and Tech. 36: 2213-2218.

13.3 Wilkin, R., Wallshlager, D., and Ford, R. “Speciation of Arsenic in Sulfidic Waters” Journal of the Royal Society of Chemistry. 4:1-7.

13.4 Controlled Benchsheet: BAL-4100 Benchsheet Template

14.0 TABLES

Table 1. GP40 pump method for arsenic speciation analysis by BAL-4100.

Time (min)	Lines A&B ([redacted])	Lines C&D ([redacted])	Flow Rate (mL/Min.)
0	[redacted]	[redacted]	[redacted]
6	[redacted]	[redacted]	[redacted]
8	[redacted]	[redacted]	[redacted]
12.9	[redacted]	[redacted]	[redacted]
13	[redacted]	[redacted]	[redacted]
15	[redacted]	[redacted]	[redacted]

Table 2. Quality Control Criteria and Corrective Action Procedures for Arsenic Speciation Analysis BAL-4100.

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Calibration Blanks	System Contamination	1 per sequence	≤ MRL or ≤ 10x all associated samples	Systematically clean sample loop, analytical column, eluent/diluent bottles, glassware, and other components until criteria met prior to any further analysis.
Calibration Standards	Acceptability of calibration curve	Daily (first batch of the sequence) or when ICV/CCV fail	r ≥ 0.995, RSD between slopes should be ≤ 10%; Low CAL Rec: 70-130% All other CALs Rec: 75-125%	Calibration standards should be remade and the instrument recalibrated. All samples should be reanalyzed.
Independent Calibration Verification (ICV)	Independent check of system performance	1 per sequence	As(III), As(V), MMA, DMA: Rec: 80 – 120%	Correct problem if known. Rerun in duplicate, if both pass continue analysis. Otherwise, recalibrate.
Secondary Calibration Verification (SCV/SRM)	Independent check of system performance	1 per sequence	Sum of As(III) + As(V) = 80-120% of total As value. No individual species recovery limits	Correct problem prior to continuing analysis. Otherwise, recalibrate system.
Continuing Calibration Verification (CCV)	Accuracy	1 immediately after calibration then 1 every 10 injections and 1 at the end of the sequence	Rec: 75 – 125%	Reanalyze samples bracketed by failing CCV unless QA Manager approves as reportable.
Continuing Calibration Blank (CCB)	Contamination due to carryover from samples	Performed after each CCV	≤ MRL or ≤ 10x all bracketing samples	Reanalyze bracketing samples < 10x the elevated CCB multiplied by the sample dilution. If contamination continues, rinses must be ran after samples causing contamination. If the problem is systemic then column must be cleaned.
Method Blank (BLK)	Contamination from reagents, lab ware, etc.	4 per batch, minimum of 1 per 20 client samples or one per preservation type.	≤ MRL or ≤ 10x all associated samples	Correct problem until criteria met. All samples affected by high BLK (sample < 10x the highest BLK) must be qualified accordingly.
Blank Spike (BS)	Accuracy	1 per batch	Rec: 75-125%	If recovery criteria not met, reanalyze in duplicate for confirmation.

Table 2. Quality Control Criteria and Corrective Action Procedures for Arsenic Speciation Analysis BAL-4100 continued.

Matrix Spike / Matrix Spike Duplicate (MS/MSD)	Accuracy and precision within a given matrix	1 per 10 client samples	Rec: 75-125% RPD ≤ 25%	If recoveries similar but fail recovery criteria, an interference is present in the sample and the result must be qualified. If RPD criteria not met, then the MS/MSD should be reanalyzed or qualified.
Method Duplicate (DUP)	Precision within a given matrix	1 per 10 client samples	RPD ≤ 25% or if results < 10x the MRL then ± the MRL of one another for waters or ± 2x the MRL for solids.	If RPD criteria not met, then the source sample and extracts must be visually assessed. Reanalyze source and DUP for confirmation and discuss with PM. It may be a heterogeneity issue that is narrated.

FINAL

Appendix E
Archaeological Monitoring and Inadvertent Discovery Plan