

# RMP REGIONAL MONITORING PROGRAM FOR WATER QUALITY IN SAN FRANCISCO BAY

sfei.org/rmp

### Field Operations Manual for the Regional Monitoring Program

### Prepared by

Ila Shimabuku, San Francisco Estuary Institute Sarah Pearce, San Francisco Estuary Institute Phil Trowbridge, San Francisco Estuary Institute Amy Franz, San Francisco Estuary Institute Don Yee, San Francisco Estuary Institute

Paul Salop, Applied Marine Sciences

## Field Operations Manual For the Regional Monitoring Program

Version #1 (Sediment Only)

Contribution #902 August 2018

### **Table of Contents**

Table	of Contents	3
Desc	cription	4
Sam	pling Methods Overview	4
Gen	eral Sampling Equipment and Supplies	5
Gen	eral Cruise Manager Responsibilities	5
1. Se	ediment	6
1.1	Overview and Objectives	6
RN	MP General Equipment Checklist	6
Co	ontractor General Equipment Checklist	6
1.2	Sampling Location Adjustments	7
1.3	Sampling Methods	7
1.3	3.1 CTD Profile	7
	3.2 Collect Sediment Sample with Van Veen Grab Sampler	
	3.3 Measurement of pH Directly in the Van Veen Grab Sampler	
	3.4 ORP Measurement	
	3.5 Pore Water Sample Collection	
1	3.6 Benthic Infauna	14
1.4	Sample Containers, Labeling, and Handling	16
Sa	ample Labeling	16
Sa	ample Handling	16
1.5	Sample Record	17
1.6	Field Sheets	
-	RP Field Sheet	
Se	ediment Cruise Field Datasheet	19

### Description

This Field Operations Manual (FOM) of the Regional Monitoring Program (RMP) is intended as a reference document for each sampling effort. The FOM describes in detail how field staff should collect water, sediment, bivalve, bird egg, and sport fish samples for all RMP sampling efforts.

Sampling conducted by the RMP should also reference the QA Program Plan (QAPP) document, which describes the entire program, and is the repository for details about laboratories, analytical methods, MDLs, sample handling, sample preservation, and quality assurance procedures. The FOM is a companion document to the QAPP, and provides a more detailed description of sampling methods that are only outlined in Section 11 of the QAPP.

In addition, each individual sampling effort will have a unique Cruise Plan or Sampling and Analysis Plan (SAP). The SAP describes the details of each sampling effort, including dates, times, places, and staff, as well as the parameters that are being collected and analyzed. The SAP will reference this FOM for the detailed sampling methods. If different sampling methods are needed for a particular study, the SAP will describe deviations from the normal methods described in the FOM.

### Sampling Methods Overview

The quality of samples collected in the field is addressed through a number of procedures. Proper selection of equipment and supplies and training for use of those items ensures that collection procedures and materials either minimally affect or do not affect samples at all. In addition, collection and analyses of appropriate quality control samples allows measurement and assessment of artifacts or influences of sampling on sample characteristics, to differentiate uncertainties and variability introduced by the sampling process from those inherent in the monitored system.

Field personnel participating in field sampling of surface water, sediment, or biological tissues will strictly adhere to the sampling protocols outlined in this FOM to ensure the collection of representative, uncontaminated, and uncompromised samples, which will provide accurate scientific data.

In general, for all sampling efforts:

- 1. Field personnel understand and follow the vessel operating safety procedures as described by the vessel captain. Any concern or uncertainty about operational procedures or safety practices must be brought to the attention of the vessel captain or the cruise manager immediately.
- 2. Field personnel make use of appropriate personal safety equipment at the discretion of the cruise manager or vessel skipper.
- 3. Field personnel will be thoroughly trained in the proper use of sample collection gear and will be able to distinguish acceptable versus unacceptable samples in accordance with pre-established criteria.
- 4. Field personnel will be thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine emissions, winch wires, surfaces, ice used for cooling).
- 5. Samplers and utensils which come in direct contact with the sample will be made of inert materials that do not contaminate for the particular analytes measured in that sample and will be thoroughly cleaned between sampling stations.
- 6. Field personnel follow established procedures in this document and in Sampling and Analysis Plans for sample collection, processing, documentation, and distribution.

- 7. Sample containers will be pre-cleaned and of the recommended type for minimizing contamination for the analytes measured. Sample containers will either be provided by contract laboratories or by project staff according to contract specifications. Bottles will be labeled prior to sample collection according to each site- and project-specific sampling plan. Spare bottles and labels will also be taken to the field.
- 8. Samples will be sealed, labeled, preserved as needed, and stored under appropriate conditions as soon as practicable.

### General Sampling Equipment and Supplies

Water and sediment ancillary samples will be collected in appropriate containers provided by contract laboratories or by project staff according to contract specifications. Samples measured in the laboratory will be processed within the relevant holding time. The appropriate volume and bottle type for samples are denoted on the Field Reference Sheet which will be included in the Cruise Plan or SAP. The appropriate equipment will be used to collect samples (e.g., peristaltic pump, van Veen grab) and, in some cases, a clean-hands dirty-hands protocol will be followed. Recommended preservation conditions and holding times for samples for chemical analyses are listed in the QAPP and in the SAP.

Sampling equipment and supplies will vary depending on the project element. Sample containers appropriate to the matrices being sampled and the analyses to which they will be subjected will be chosen. All containers should meet or exceed the required trace limits established by the US EPA in the document EPA/540/R-93/051, Specifications and Guidance for Contaminant-Free Sample Containers. Chemical-resistant powder-free nitrile and/or polyethylene gloves will be worn and, in some cases, a clean-hands dirty-hands protocol will be followed to minimize contamination of exposed samples. Field cleaning procedures of sampling equipment will be employed between stations to minimize cross-contamination between samples for the parameters of interest.

### General Cruise Manager Responsibilities

It is the responsibility of the Cruise Manager to ensure that all field personnel are capable of sampling safely and complying with the quality assurance guidelines. The Cruise Manager is required to ensure that:

- 1. Field personnel understand and follow the vessel operating safety procedures as described by the vessel captain. Any concern or uncertainty about operational procedures or safety practices must be brought to the attention of the vessel captain or the cruise manager immediately.
- 2. Field personnel will strictly adhere to the quality assurance protocols (see QAPP plan) to ensure the collection of representative, uncontaminated samples.
- 3. Field personnel are thoroughly trained in the proper use of sample collection gear and are able to distinguish acceptable versus unacceptable samples in accordance with pre-established criteria.
- 4. Field personnel are thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine exhaust, winch wires, deck surfaces, ice used for cooling samples, etc.).
- 5. Field personnel follow established procedures for sample collection, processing, documentation, and distribution.
- 6. Field personnel make use of appropriate personal safety equipment at the discretion of the cruise manager or vessel skipper.

### 1. Sediment

### 1.1 Overview and Objectives

Contaminants are analyzed at regular intervals in surface sediment as part of the RMP Status and Trends monitoring program. This Field Operations Manual (FOM) details standard sampling and sample handling methods associated with typical sediment sampling operations. However, due to unique special studies and add-ons for each sediment cruise, these methods may require modification.

Therefore, always review the Sampling and Analysis Plan for each unique monitoring effort as it will reference which of the following protocols are necessary for each cruise. The exact order and content of sampling tasks will be determined for each sampling effort once any and all add-ons have been factored in. Section 1.3 lists equipment checklists and detailed procedures for sediment cruise tasks.

Sampling personnel wear nitrile and/or polyethylene gloves whenever taking or processing samples to avoid contact contamination (and for personal protection). In addition, airborne contamination is avoided by keeping sample containers, sample scoops and compositing bucket inside the vessel cabin with door closed or appropriately covered when not in use.

RMP G	eneral Equipment Checklist
	Camera (if needed)
	Boots
	Foul/wet weather gear
	Personal items (sunscreen, water/drinks, snacks, sunglasses, etc.)
	Bag of pens & scissors
	Field Log
	☐ Sediment section of the Field Operations Manual (this document)
	☐ Cruise Plan & Maps
	☐ Blank ORP log sheets – see section 1.6 (print on waterproof paper if possible)
Contra	ctor General Equipment Checklist
	Laptop and cables
	Sample label printer
	Sample labels
	Cruise plan
	Sample collection forms
	Chain of custody forms (backup)
	2 x Aluminum foil, 100 square feet
	Ziploc(tm) bags, 1 gallon size, as needed
	2 x Sharpie pens, thin and wide
	400 x Nitrile gloves, non-powdered (M + L)
	Splash-proof eye protection
	Plastic squirt bottle of DI water
	Whirl-pak bags
	Bubble bags for organics samples

4 x Tape, rolls for sealing labels
Plywood benchtop
Sunscreen (Titanium dioxide)
Cable ties
Paper and cloth towels
Foul weather gear for splash protection, boots

### 1.2 Sampling Location Adjustments

When the vessel reaches a sampling station and the anchor has been deployed, the captain notifies personnel that the vessel is on site. Sampling procedures will ensure that samples are collected from a localized area at each site. The goal of the sampling program design is to collect relatively fine sediments at the target coordinates. If fine sediments (>1/3 fine by visual estimate) are not present at the target coordinates, the cruise vessel skipper will attempt to locate finer substrate within a 100 m radius of the given coordinates. If no appropriate sediments are located within this radius, the sampling operations will proceed to the next scheduled site and the site in question will be replaced with a site from the oversample list included in the Cruise Plan or SAP. The coordinates will be checked throughout sampling as necessary to ensure that the anchor has not dragged. One set of coordinates will be recorded for each site.

### 1.3 Sampling Methods

### 1.3.1 CTD Profile

Contrac	ctor CTD Profile Equipment Checklist:
	SBE 19 CTD
	Rope for CTD, 50'
	Gloves for CTD (neoprene, leather, etc.)
	CTD processing laptop and cables
	6 x Spare CTD "D" batteries

☐ Distilled water and syringe

The CTD profile measures dissolved oxygen, pH, temperature, salinity, specific conductance and/or electrical conductance. A CTD cast is recorded at each station. The CTD is checked for proper operation at least 48 hours before use.

The following steps describe the CTD deployment and data management process:

- 1. At the beginning of the day, Initialize CTD via laptop.
- 2. Disconnect communication cord from CTD and replace rubber cap.
- 3. Ensure that rope is securely fastened to vessel and to CTD containment cage.
- 4. Ensure that DI syringe is disconnected from CTD intake.
- 5. Before sediment samples are collected, turn CTD on by moving switch completely to on position (fully up).
- 6. Place CTD into the water, with intake approximately 1 meter below water surface (typically a bit lower in the column to allow for any rough seas).

- 7. Leave CTD at surface for approximately 1-2 minutes to equilibrate.
- 8. Slowly lower CTD to the bottom (at a rate less than 1' per second) until rope goes slack or the end of the rope is reached. With strong currents, the rope may extend at a severe angle precluding its reaching the bottom. As soon as the CTD reaches the bottom, immediately begin moving to surface so as to minimize the amount of sediment pulled into the intake. The CTD can be moved to the surface at any rate as data is only collected on the downcast.
- 9. When CTD is at the surface, return to the vessel deck and place the switch in the off position (fully down).
- 10. Download the data at least once daily; check battery level at download, and replace batteries when Vmain drops below 7V.
- 11. At day's end (or after stations where CTD intake may have become fouled with sediment or vegetation) rinse the CTD with distilled water, flush the intake with a minimum of three syringes full of distilled water, and store the CTD with a full syringe of DI inserted onto the intake and partially emptied into the CTD.

### 1.3.2 Collect Sediment Sample with Van Veen Grab Sampler

Contra	ctor Sediment Sample Equipment Checklist:
	Van Veen grab, 0.1 square meter capacity, Kynar(tm) coated
	Van Veen grab stand
	2 x Plastic floats for Van Veen grab
	2 x Weights for Van Veen grab (50 lbs each), with associated cotter pins
	Punch for removing hinge
	Timers for grab collection
	Sediment overflow bucket
	4 x Insulated plastic coolers for sample storage, (pre-cleaned)
	Dry ice, to be replenished
	Wet ice, to be replenished
	Insulated plastic cooler for dry ice storage
	3 x Plastic brushes
	2 x Hydrochloric acid 1% - 2%, 4 L amber bottle, reagent grade
	2 x Methanol, 4 L amber bottle, reagent grade
	3 x Deionized/reverse osmosis water, 2.5 gallon
	Liquinox detergent
	$4 \text{ x Teflon}^{\text{\tiny{TM}}}$ squeeze bottles, (pre-cleaned) in the laboratory (labeled for distilled water (1)
	liquinox soap (1), hydrochloric acid (1), and methanol (1))
	3 x Kynar(tm) coated scoops, (pre-cleaned) in the laboratory
	Kynar(tm) coated bucket, (pre-cleaned) in the laboratory
	Eye protection
	Personal flotation devices

### Pre-Cruise Sampling Equipment Cleaning

The Van Veen grab, buckets, scoops, and squeeze bottles used for collection of RMP sediment samples are to be prepared by the contractor at least four days prior to sampling by following these steps:

- 1. Soak equipment (fully immersed) for three days in a 0.5 % solution of Liquinox™ detergent and deionized water.
- 2. Rinse equipment three times with deionized water and let dry in a clean place.
- 3. Rinse equipment with 1.0 % solution of hydrochloric acid, followed by a rinse with petroleum ether, followed by another set of three rinses with deionized water. All equipment is then allowed to dry in a clean place.
- 4. The cleaned grab is wrapped in aluminum foil until used in the field. All other equipment is stored in clean Ziploc™ bags until used in the field.

### Collection of a Sediment Grab

Sediment sampling is performed using a Young-modified, Van Veen Grab Sampler with a surface area of 0.1 m<sup>2</sup>. The grab is constructed entirely of stainless steel and the jaws and doors are coated with Kynar<sup>™</sup> (or equivalent) to improve chemical inertness. A scoop and bucket used to remove and composite sediments are also constructed of stainless steel and coated with Kynar<sup>™</sup>.

The A-frame at the stern of the vessel (or alternatively hydraulic crane) will be used for deploying the Van Veen grab. The quality of grab samples will be ensured by requiring each sample to satisfy a set of criteria concerning the depth of penetration and disturbance of the sediment within the grab. In this way, each sample will normally contain the top 5-cm of sediment within the area of the grab jaws.

Samples will be rejected for the following conditions:

- There is a rock or shell fragment wedged between the jaws of the grab allowing the sample to wash out.
- The sample surface is significantly disturbed.
- The sample is uneven from side to side, indicating that the grab was tilted when it penetrated the sediment.
- The surface of the sample is in contact with the top doors of the grab and is spilling out of the grab, indicating over-penetration of the grab and possible loss of material around the doors.

If the sediments at a station are considerably fine, plastic floats may be attached to the grab frame and secured so they do not interfere with grab operation. Likewise, if the sediments are considerably coarse, weights can be added to the grab frame to assist penetration of the sediments. The quality of grab samples is ensured by requiring each sample to satisfy acceptance criteria concerning the depth of penetration and disturbance of the sediment within the grab.

A pH measurement (section 1.3.3) is taken on each side of the grab hinge during the first and second grab. No pH measurements are needed on the third and consecutive grabs. An ORP sample (section 1.3.4) is collected from the first grab before sediment is removed and processed for analysis. Depending on type of analysis being conducted, samples are collected from the Van Veen Grab Sampler in one of two methods, as detailed below.

### 1.2.2.1 Collection of Individual Samples Directly from the Van Veen Grab Sampler

The Kynar™ coating applied to the Van Veen grab, scoops, and compositing bucket is a fluorinated polymer product, which makes it unsuitable for samples to be analyzed for (or archived for potential analysis of) per- and polyfluoroalkyl substances (PFAS). Sediment collected for these analyses therefore

need to be collected directly from the grab, at locations removed from its sides. After the overlying water has been drained off, the samples needed for analyses (e.g., PFASs) will be collected from the center of the Van Veen, avoiding contact with the grab or sediment that may have been in contact with the grab. The sample bottle or specified scoops will be used to collect the sample directly into the container. Submerge the jar or scoops directly into the sediment surface and scrape along the surface, to the appropriate depth, until containers are filled, avoiding areas that have been disturbed by the pH probe or the ORP core.

### 1.2.2.2 Collection of Composite Sediment Samples

Using the Van Veen, a minimum of two acceptable grabs will be composited for distribution to analytical laboratories for remaining sediment chemistry parameters. Each sediment grab, after removing sediment that has been in contact with and possibly contaminated from the sampler, yields about 1 liter of clean sediment for analyses. Samples of bulk surficial sediments (top 5 cm) for analysis of chemical constituents must be collected in a manner such that surface layers are not disrupted when removed for processing. Disruption may cause mixing of surficial layers with lower layers in the sample, and may lead to dilution or concentration of the contaminants of concern, depending on the chemical content of the various layers of sediment. Therefore, a minimum amount of scraping should occur while still ensuring that only uncontaminated sediment is collected and, if excessive scraping or mixing of layers cause significant disruption, the grab should be discarded. Whole or partial grabs will be collected and allocated for bulk sediment collection using the following protocol.

After retrieval of the grab and after any overlying water has been drained off, the top 5 cm of sediment will be scooped into a pre-cleaned compositing bucket. Sediment touching uncoated surfaces or disturbed by pH measurement, ORP coring, or other sample collection activities will be avoided. Sampling personnel will continue collection of acceptable grabs until a sufficient volume of sample material has been generated to support all requested analyses (or until site conditions are deemed too dangerous to continue).

Once a sufficient volume of sediment is collected, the compositing bucket and scoops are transferred into the vessel cabin and the doors closed for processing; this is done so that the vessel may get underway while minimizing potential effect of vessel exhaust upon sample material. Sample material is then mixed using coated scoops / spoons until achieving a consistent appearance, which may be difficult with particular substrate types (i.e., heavily consolidated materials). While conducting mixing, sampling personnel should take care to avoid scraping the coated bucket in the interest of maintaining the coating's integrity. Particular attention should be paid to the edges of scoops and spoons — when bare metal shows, they should be replaced with backups. Portions of the composited sample are then aliquoted into pre-labeled containers provided by each laboratory. Typically, the containers are filled in the order of freezing preservation requirements and required cleanliness (e.g., some samples should be placed on dry ice within 20 minutes of collecting and trace metals should be aliquoted prior to organics and organics prior to TOC, particle size, etc). Samples are then transferred to cold storage (i.e., wet / blue ice or dry ice) per the requirements identified in the cruise plan. Samples are taken to secure refrigerators and freezers at the end of each sampling day.

Once the material in the compositing bucket has been transferred inside the vessel cabin for processing, the deck personnel can rinse the grab, bucket, scoops, and spoon to remove sediment. The sediment-catch bucket should then be emptied. They may begin the full cleaning procedures (see Section below on "During Cruise (Between Samples at Next Station)" equipment cleaning) once the boat has arrived at the next sampling station.

### During Cruise (Between Samples at Next Station)

Between stations for each successive station, the following cleaning protocol will be enacted for the Van Veen, buckets, and scoops:

- 1. Rinse equipment with site water.
- 2. Scrub equipment with a dilute Liquinox™ detergent solution and scrub brush. Rinse thoroughly with site water.
- 3. Rinse equipment with a dilute solution of hydrochloric acid, followed by a rinse with methanol, followed by a distilled water rinse.

### 1.3.3 Measurement of pH Directly in the Van Veen Grab Sampler

# Contractor pH Measurement Equipment Checklist: pH meter and standards pH calibration standards – 1 each: 4, 7, 10 If a grab is deemed acceptable, pH measurements from two poir recorded by submerging a pH probe into the sediment to a depi

If a grab is deemed acceptable, pH measurements from two points (for a minimum of two grabs) will be recorded by submerging a pH probe into the sediment to a depth of approximately 2 cm and allowing it to equilibrate while the water is draining from the sample. The pH meter is calibrated twice per day: once in the morning and once half way through the day. Procedures for sample collection then follow, as described in section 1.3.2.

### 1.3.4 ORP Measurement

### RMP ORP Measurement Equipment Checklist:

2 x WTW meter with mV readings (WTW Multi 340i)
2-3 x ORP probe (WTW Sentix ORP, platinum electrode, Ag free)
2 x clear plastic core (~5cm diameter or larger, ~12-15cm height)
Plastic lid to set the core on (can use a lid from a sediment container)
Clamps that attach to stand to hold ORP probe
12 inch ruler to visualize depth of core
White plastic stand (base & pole)
Camera to photograph each core

Oxidation/reduction potential (ORP) readings are taken at each station from a grab core or direct insertion in exposed sediment. Additional readings can also be taken, time permitting. Instrument ORP readings are offset from true "Eh" readings, by an amount specific to the particular electrode type: the Sentix ORP (platinum) probe for the WTW meter is -~210mV relative to true Eh (hydrogen electrode): Eh= ORPreading + 210mV (at 20C). DO NOT make correction to the ORP reading in the field- record what

you read. Readings are taken at 3 depths (1 & 6cm from top, and 1cm from bottom of core) in the standard RMP method.

### Pre cruise testing:

Testing should be conducted one to two weeks prior to cruise to allow time for ordering replacement parts if needed.

- 1. Obtain a testing standard solution for the ORP probe. All the available commercial ones are + (positive) potential
- 2. Insert probe into solution for 10 min, vent port at top of sensor open
- 3. Attach tape label to probe indicating the reading and date, mark "good +" if within 10mV
- 4. If reading for solution is >10mV from target, in addition to std reading, mark/note as potentially "bad +"
- 5. Repeat for other used probes, if all are similarly off do step 6
- 6. Repeat with a brand new probe (should always have 1, order if none). If this is off too, it may be a wire problem, or bad standard. Try other wires with the new probe. Mark bad wires with tape label.
- 7. Order new wire, new standard, or test standard with different manufacturer ORP meter if available. (There are now ORP meters with unchangeable sensors, almost same cost as new sensor alone, which we may transition to eventually)
- 8. For all the probes that pass the initial test with standard, do a secondary test in estuarine/marine field sediment, mudflat areas around edge of the Bay (e.g. mudflat near SFEI offices, or near site if already away from office). Poke/dig into sediment to find area and depth where sediment goes black/gray. Once found, insert good probe(s) into undisturbed adjacent sediment to same depth. Mark mV reading, and those that go to <0mV mark "good –" as well.
- 9. If readings don't go <0mV after ~10min, mark those probes as potentially bad "bad -".
- 10. Keep and use for the field, probe & wire combos that have both good + and good -.

### Collection Method

- 1. Push the corer tube into the grab, let crew collect the rest of the material.
- 2. Dig a spoon/spatula under to help lift it out
- 3. Once out, place on a jar lid or other flat surface (to prevent core sliding out of tube).
- 4. Do readings with vent port at top of ORP sensor **open**. Be sure to close at the end of the day.
- 1. Make a note in the field log of depth below surface any transitions or notable features in the core or surrounding grab (e.g. gray below 4 cm, fine shell fragments throughout). Optionally take a picture of core/grab/in situ sediment cross section.
- 2. Uncap & push the ORP probe, to target depth ("0" point is bottom of cylindrical portion, roughly midway between platinum tip and small hole near tip).
  - a. If probe hits something hard like shell, rock, or wood fragment, do not force through, as probe tip may break. If close to target depth (within 0.5cm of target), keep that location. If a long way from target depth, note the depth of the obstruction, and pull out the probe. In site sediment, just insert at another point.
  - b. In a core, there is less space to relocate so use a wire or skinny screwdriver to poke at locations to find a way around the object, but do not poke all the way to the target depth (or you may expose that point to air).
  - c. If a clear path is found with test wire/screwdriver, insert the probe along that path. If near the core edge, be sure the ORP probe orifice (small hole in the probe side about 0.5cm from the tip) is facing toward the core center.

- 3. Note time/set timer. Record reading @10 min (will continually drift, so consistently read at 10 because drift is slow by then). Record raw ORP, **NOT** Eh conversion.
- 4. Take a photo of the core with a ruler to show the length and a label showing the site number (e.g. if showing a prelabeled sample container for the picture, be sure to return to samplers to allow filling).
- 5. If ORP >0 in anoxic (black/sulfidic) sediment probe may be broken. Switch probe.
- 6. Dump core, rinse probe in DI water, re-cap, and get ready for the next station, or take another reading from the same station if there is enough time.
- 7. Close the port unless another sample is due soon. Clean well, rinse/store with DI water in cap at day end.

### 1.3.5 Pore Water Sample Collection

From the initial van Veen sediment grab, the pore water samples are collected. Using pre-cleaned glass cores, three 5-cm deep cores are taken from each side of the grab. These cores are used for measurement of pH, ammonia, and total sulfides in pore water. These cores are centrifuged onboard the vessel. Part of the supernatant is then used for analysis of ammonia and pH (performed on-board the vessel by AMS) and part is preserved for analysis of sulfides (analyzed in the laboratory by MPSL). Use the following guidelines for pore water sample collection and analysis:

- 1. Prior to fieldwork, wash coring tubes using the same procedures as outlined for preparation of the Van Veen grab and wrap in clean aluminum foil.
- 2. After retrieving the Van Veen grab and determining the sample suitability, insert a coring tube into the sediment so that there is 5.0 cm of sediment in the tube after it has been withdrawn. Three cores are removed from each side of the grab for a total of six cores taken per sampling site. Coring tubes may be reused at the same station without washing.
- 3. Place the sediment from each core into a separate clean centrifuge tube and transport the tubes into the vessel cabin. Purge the headspace of each tube with nitrogen then cap each tube tightly.
- 4. Place the six centrifuge tubes into the centrifuge and spin at a rate of 400-1,000 RPM. Be aware that the centrifuge may transfer excessive heat to the sample if processed for too long a time.
- 5. Rinse a collection pipette with DI water and flush it dry.
- 6. Pipette or pour off the overlaying pore water from the top of each centrifuge tube into one empty centrifuge tube.
- 7. Pipette out enough pore water from the composited centrifuge tube to fill the sulfides vial with no headspace or bubbles. This requires about 1.5-2.0 ml of pore water. Do not spill the preservative in the sulfide bottle and do not contact the pipette with the sample water in the sulfide vial (it contains acid and could corrupt ammonia measurements). Record the station code and date on the sulfides vial and store it in a cool, dark place (do not freeze the sample).
- 8. Follow instructions in Hach Datalogging Colorimeter Handbook for running either the Nitrogen, Ammonia, Low Range (p. 219) or Nitrogen, Ammonia, High Range (p. 225) tests as needed. Use care with reagent powder packets, as reagents can be hazardous to human health. Clean up all spills immediately and empty liquid wastes into appropriate hazardous materials container.
- 9. Rinse the pH probe with DI water, dry, and place directly into the composite centrifuge tube. Record the pH reading when the meter stabilizes. This normally takes less than a minute.
- 10. Dispose all wastes and materials properly.

### Measuring Pore Water Ammonia Using the Hach Colorimeter

The pore water ammonia probe must be assembled and calibrated at least 24 hours before use.

Measuring ammonia concentrations in pore water is a standard procedure using the portable Hach DR/820 colorimeter. There are two calorimetric tests available, depending on the predicted pore water ammonia concentration. The low range test (0-2.5 mg/L total ammonia) is applicable for most RMP Central Bay sites and sites where sediment grain size is coarse. The high range test (2-5-10.0 mg/L total ammonia) is applicable at most RMP South Bay and North Bay sites. It is standard to run a low range pore water ammonia test first, switching to a high range test if the colorimeter can not provide an accurate result. Use the following guidelines for measuring pore water ammonia with the Hach colorimeter:

- 1. Use method #10023, Nitrogen Ammonia Salicylate Method for analysis of pore water total ammonia as referenced in the Hach colorimeter owner's manual.
- 2. Turn on the colorimeter and enter the program number for the test being performed, for low range ammonia the program number is "66".
- 3. The colorimeter requires that two standards be prepared, a blank and an ammonia standard of known concentration. The ammonia test kit prepared by Hach includes standardized ampules used for preparation of the standard. Refer to the Hach DR/820 colorimeter owner's manual, page 219 for preparation of the standards.
- 4. After a blank and ammonia standard has been analyzed, the colorimeter will be ready to read the ammonia concentration of the pore water sample. Refer to Hach DR/820 colorimeter owner's manual for preparation of the pore water sample using the reagents provided in the Hach ammonia test kit. Measure the ammonia concentration of the pore water sample and note the measurement in the ship sample logbook. If the pore water ammonia value is over the limit for the low range test, repeat the procedure using the high range test kit.

### 1.3.6 Benthic Infauna

Benthic infauna primarily comprises sedentary, invertebrate organisms that burrow in or live on the surface of sediments. Benthic infauna communities fluctuate in response to natural and human induced environmental perturbations and therefore can be important indicators of environmental health. For this reason they often are an important component of many ecological monitoring programs.

Benthic infauna is sampled with a Ponar grab with a surface area of 0.05 square meters. The grab is equipped with hinged stainless steel mesh lids with rubber flaps to allow flow-through of water during descent and thus minimize disturbance of surface sediments. The rubber flaps close upon retrieval and prevent winnowing of the sample. Sampling procedures will insure that samples are collected from a localized area at each station to reduce uncontrolled temporal and spatial variations. Lead weights are added to or removed from the outside of the grab as appropriate for sediment type to control depth of penetration.

After deployment and retrieval, the grab is placed on a stand for processing. The grab lids are opened and the sample is examined for suitability using the following criteria:

- Complete closure of the grab jaws.
- No evidence of sediment washout through the grab doors.
- An even distribution of the sediment in the grab.
- Minimum disturbance of the sediment surface.
- Minimum overall sediment depth appropriate for the sediment type: 4 cm in coarse sands and gravel, 5 cm in medium sands, 7 cm in fine sands, and 10 cm in silty sands, silts, and clay.

If the sample passes all of the criteria, the grab jaws are opened and the sample is dumped into a five gallon plastic bucket placed beneath the grab stand. Estuary water is used to wash all sediment from the grab and grab stand into the bucket. Care is exercised not to lose sediment by overfilling the bucket. The sample bucket is then moved to a wash table for sample sieving.

When a sample bucket arrives at the sieving station, it is lifted to the sieve table and poured slowly onto the nested sieve screens. The sea water hose with a flow control nozzle is used to slowly wash sediment from the sample bucket onto the sieve screens. The sieving process is aided by keeping sediment in suspension as it reaches the screen. The sample is washed from the sample bucket until the bucket is empty and well rinsed. Sediment is washed through the nested sieve screens by gently running seawater over the top screen. Use of high water pressure damages organisms impinged on the sieve screen mesh.

When all material smaller than 1.0 mm has passed through the top screen, the process is repeated with the finer screen until all material smaller than 0.5 mm has passed through. The material retained on each screen is gently washed into one corner of the screen and with the aid of a canning funnel, washed into separate appropriately labeled sample jars. A wash bottle with seawater is used to rinse any material on the inside screen frame and canning funnel into the sample jar. Any organisms remaining on the screens are carefully removed with forceps and placed in the appropriate sample jars. The sample jars are then capped with dome lids and bands, labeled with indelible ink inside and out, and delivered to the on-board formalin station. Great care is exercised to avoid creating fragments when removing organisms from the sieve screens. The sieve screens are rinsed with high-pressure seawater and scrubbed clean with a stiff-bristle brush between samples.

If the sample contains many shell fragments and/or worm tubes, the sediment sample is added to the top (1.0 mm) screen in stages so that the screen does not become too full. If the bottom screen (0.5 mm) begins to clog with sediment, the field crew ceases adding sample and gently runs the hose nozzle with low flow along the outside bottom of the 0.5 mm screen being careful not to lose sample by allowing water to escape over the top of the sieve. The material retained on a sieve screen is not allowed to fill the sample jar more than half full. In such a case, the material is divided among two or more jars and each jar is labeled as jar 1 of 2, jar 2 of 2, etc., as required.

At the formalin station, each sample jar lid is replaced with screen lids fitted with 0.25 mm Nitex™ mesh and the Estuary water is decanted from the sample jars through the screen lids. Relaxant (isotonic MgCl₂) is added to the sample through the screen lid to a level approximately one third higher than the sample level. A wash bottle of relaxant is used to wash down the screen lid and sides of the sample jar. The sample jar is recapped with the sample jar lid and gently rotated several times in a tilted position to ensure mixing of the relaxant throughout the sample. The sample is allowed to sit in the relaxant for 15-30 minutes. After this period, the sample jar lid is replaced with a screen lid and the MgCl₂ is decanted out of the sample jar in preparation for fixing the sample.

At the formalin station, relaxant is decanted out and fixative (10% buffered formalin in seawater) is added to the sample through the screen lid. Fixative is added to a level approximately one third higher than the sample level. A wash bottle of fixative is used to wash down the screen lid and sides of the

sample jar. The screen lid is removed, 2 or 3 drops of stain (rose bengal solution) are added to the sample and the sample jar is recapped with the sample jar lid. The jar is gently rotated several times in a tilted position to ensure mixing of the fixative and stain with the sample. Safety glasses and nitrile gloves are worn when working with fixative.

While onboard the survey vessel, benthic infauna samples are stored in plastic trays with dividers, then transferred to cardboard cartons with dividers for travel to the laboratory for sample sorting. Benthic infauna samples fixed in formalin are washed in tap water and transferred to 70% ethyl alcohol between 24 and 72 hours after fixation. Samples can then be held indefinitely in 70% ethyl alcohol.

A sample collection log records sample date, station, depth of grab penetration, number of grabs, number of bottles per sample, and any problems encountered.

### 1.4 Sample Containers, Labeling, and Handling

All sample containers will either be factory cleaned, or cleaned by the lab conducting the analyses. The details of sample cleaning and responsibilities for this task will be specified in the Sampling and Analysis Plan.

Containers should be pre-labeled, and packed into the appropriate clean cooler before the cruise begins. During sampling, a container list is used to verify that all samples are properly collected in the field. At least two personnel verify that the proper sample containers for each station have been filled, and that the labels correspond to the proper station name and code.

### Sample Labeling

Sample IDs are assigned prior to the sampling event based upon the number and type of samples to be collected. The standard sample ID labeling system used is as follows:

### RMP-YYSC-XXXX-##

Where:

RMP = Project

YY = Year sampling conducted (20YY)

SC = Matrix (Sediment Cruise)

XXXX = Unique ID number

## = Aliquot number (for archive samples only)

In some cases, the IDs may be modified to identify a specific subsample number. To-date, this has applied to archive samples only.

### Sample Handling

Analyses and analytical laboratories vary on an ongoing basis. Individual cruise plans developed specific to a sampling event will detail laboratories, contact info, and handling requirements and should be referenced prior to and during sampling activities. Individual handling requirements may require deviations from the above procedures. These deviations will be identified within the cruise plan.

### 1.5 Sample Record

In addition to the ship's log, a sample record is maintained for each site. The sample record contains the following information:

- 1. Station name and code
- 2. Collection date
- 3. Arrival and departure time at each station
- 4. Station coordinates (latitude and longitude) from the survey vessel's GPS
- 5. Depth at time of sampling from the ship's depth meter
- 6. A record of every sample bottle filled, with bottle identification code and quantity (Optional)
- 7. Collecting personnel
- 8. Other remarks (i.e. any conditions that could possibly influence sample analysis or data interpretation or notation of the general performance of equipment involved with the sampling.)

The sample collection form, coupled with a chain of custody record and a laboratory analysis record, allows tracing of the complete history of a sample from time of collection to final entry of data to a computer database. In addition to the sample collection form, some of the laboratories may use a bottle labeling system that catalogs the preparation of bottles prior to their use in the field. This system is particularly important for the Teflon™ bottles used in trace element analysis, where exhaustive cleaning procedures are employed before releasing them for field sampling.

### 1.6 Field Sheets

The PDF and Excel file for the field sheets on the following pages can be found in the Google Drive S&T folder under the appropriate year and folder for the sediment cruise.

### ORP Field Sheet

Site:	Date:		Sampler:				
Photo #s (remember to include a photo w/ jar k	abel):						
Total Core Depth (cm):	Total Core Depth (cm):  Anoxic (Gray/Blank) transition depth (cm):						
Description at surface:							
Description below surface:							
*Probe depths are measured from tip midpoint (~1 cm from tip) to the surface  Probe depth (cm) ORP (mV) Equilibration Time (10 min default)							
	1						
	6						
*last depth should be total depth minus 1 cm if	possible	•					
			T				
Site:	Date:		Sampler:				
Photo #s (remember to include a photo w/ jar k	abel):						
Total Core Depth (cm):		Anoxic (Gray/Blank) transition dept	h (cm):				
Description at surface:							
Description below surface:							
bescription below surface.							
*Probe depths are measured from tip midpoint	(~1 cm from tip) to the	surface					
Pro	be depth (cm)	ORP (mV)	Equilibration Time (10 min default)				
	1						
	6						
*last depth should be total depth minus 1 cm if	possible						
Site:	Date:		Sampler:				
Photo #s (remember to include a photo w/ jar k	abel):	1					
Total Core Depth (cm):	h (cm):						
Description at surface:							
Description below surface:							
*Probe depths are measured from tip midpoint (~1 cm from tip) to the surface							
Pro	be depth (cm)	ORP (mV)	Equilibration Time (10 min default)				
	1						
	6						
*last depth should be total depth minus 1 cm if possible							

### Sediment Cruise Field Datasheet

Site Code:	Date:		Ini	tial:			
Time On Station:	Time Off	Station:					
Lat: Long:			Depth (m):				
CTD Time On:	CTD File:	FB:	Y/N	FD:	Y/N		
Analyte Code	Analyte	Identi	fication		(	Commen	ts
ALS_N	N						
ALS_TOC_TS	TOC, TS						
ALS_PCMSC	PSD						
TE_CCSF	TE						
TE_BAL	TE						
PCB_AXYS	Organics						
ARCH_TE_250	Archive			250ml	, 2 per site		
ARCH_ORG_60	Archive			60ml,	3 per site		
ARCH_NIST_22	Archive			22ml,	3 per site		
ARCH_PFC_10	Archive			10ml,	10ml, 3 per site (Fill from grab)		
Silox_DTSC	Siloxanes						
QAC/AB_UMN	QACs/ABs						
NTA_SFEI	Non-targeted						
Gadolinium_UFB	Gadolinium			Water Grab			
Sed pH (4)							Avg.
Sea State:			Wind:				
% Overcast:			Current:				
Photos:							
Sediment Character							
Comments							