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Addendum 7 to Quality Assurance Project Plan

The Puget Sound Assessment and Monitoring Program: Sediment Monitoring Component, 2015 Monitoring in the Bainbridge Basin

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The Puget Sound Assessment and Monitoring Program: Sediment Monitoring Component, 2015 Monitoring in the Bainbridge Basin

July 2015

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1.0 Table of Contents

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2.0 Abstract

This addendum to the 2009 PSEMP Sediment Monitoring Component Quality Assurance Project Plan (QAPP) (Dutch et al., 2009) provides details about sampling locations, parameters, and sampling/analysis schedules for the 2015 Urban Bays sampling program. All other quality assurance elements, including sampling methods, quality control, and data management, are as described in Dutch et al., 2009 and remain unchanged for the Urban Bays sampling program.

4.0 Project Description

Ongoing Sediment Monitoring Programs

The Washington State Department of Ecology (Ecology) Marine Sediment Monitoring Team conducts sediment sampling as part of the Puget Sound Ecosystem Monitoring Program (PSEMP)[1]. The PSEMP Sediment Component consists of three annual monitoring programs:

- Long-term[2] – Conducted at ten stations located throughout Puget Sound and sampled annually each April since 1989 (or longer).
- Regional[3] – Forty stations sampled within one of eight geographic regions annually each June since 1997. Sampling rotates among the regions over a ten-year period. A new set of randomly selected stations are sampled each time a region is revisited. NOTE: Regional sampling will not be conducted in 2015, as per the program's long-range planning schedule.
- Urban Bays[4] – Thirty randomly selected stations sampled within one of six urban bays annually each June since 2007. Sampling rotates among the bays over a six-year period. The same set of randomly selected stations is sampled each time an urban bay is revisited.

2015 Urban Bays survey: Bainbridge Basin

This Quality Assurance Project Plan addendum provides detailed information about the schedule, budget, measurement quality objectives, parameter list, sampling procedures, and measurement methods for the 2015 sediment survey in the Bainbridge Basin of Puget Sound that differs from the original QAPP (Dutch, 2009). The numbering scheme for the sections of this addendum reflects Ecology's current required formatting for QAPPs and is not found in the original QAPP.

[1] Formerly known as the "Puget Sound Assessment and Monitoring Program (PSAMP)"
[2] Formerly known as "Long-term/Temporal"
[3] Formerly known as "Spatial/Temporal"
[4] An expansion of Ecology's "Urban Waters Initiative"

4.1 Objectives

The objectives of the 2015 Bainbridge Basin sediment survey are to (1) recharacterize sediment quality in the Urban Bays Bainbridge Basin sampling frame and (2) compare these data to 1998 baseline and 2009 data to determine change over time.

4.4 Target Population

The target population of the 2015 Bainbridge Basin sediment survey is the surface soft sediments in the Urban Bays Bainbridge Basin sample frame (Figure 1).

4.5 Study Boundaries

The Urban Bays Bainbridge Basin sample frame consists of the subtidal areas ≥ 6 feet deep, west of Bainbridge Island, including Rich Passage, Sinclair Inlet, Dyes Inlet, Liberty Bay, and Port Madison (Figure 1).

5.0 Organization and Schedule

5.4 Project Schedule

Key activities for the PSEMP Long-term sediment monitoring work are listed in Table 1.

Table 1. Proposed schedule for completing the field and laboratory work, data entry into EIM, and reports for the 2015 PSEMP Urban Bays sediment monitoring program.

Field and laboratory work	Due date	Lead staff
Field work completed	May 2015	Margaret Dutch
Laboratory analyses completed	Ammonia, Total Sulfides – June 2015 Pharmaceuticals, Personal Care Products – June 2015 Perfluoroalkyl Substances – June 2015 Total Organic Carbon – July 2015 Grain size – September 2015 Chemistry – March 2016 Toxicity – March 2016 Taxonomy – June 2016	
Environmental Information System (EIM) database		
EIM Study ID	UWI2015	
Product	Due date	Lead staff
EIM data loaded	July 2016	Sandra Weakland
EIM QA	August 2016	Margaret Dutch
EIM complete	September 2016	Sandra Weakland
Final report		
Author lead / support staff	Valerie Partridge / Sandra Weakland, Margaret Dutch	

Schedule	
Draft due to supervisor	October 2016
Draft due to client/peer reviewer	November 2016
Draft due to external reviewer(s)	December 2016
Final (all reviews done) due to publications coordinator	January 2017
Final report due on web	February 2017

5.5 Limitations on Schedule

PSEMP Urban Bays sampling is usually conducted in June each year. In 2015, sampling will be conducted in May to ensure that PPCP and PFAS laboratory analyses can be completed by the end of the biennium in June.

5.6 Budget

The proposed budget for the PSEMP Urban Bays Bainbridge Basin sediment survey is provided in Table 2.

Table 2. Project budget.

Parameter	Number of Samples	Number of QA Samples	Total Number of Samples	Cost Per Sample	MEL 25% Overhead/Sample	Lab	Total
TOC	33	3 (field split)	36	$43.60	--	Manchester Environmental Laboratory	$1,569.60*
Metals	33	3 (field split)	36	$197.00	--		$7,092.00*
PAHs and phthalates	33	3 (field split)	36	$415.00	--		$14,940.00*
PCB Aroclors and congeners	33	3 (field split)	36	$175.00	--		$6,300.00*
PBDEs	33	3 (field split)	36	$190.00	--		$6,840.00*
MS/MSD QC	1/batch of 20, all MEL analyses	2/MEL analysis	2/MEL analysis	$1,020.60	--		$2,041.20*
Pharmaceuticals and Personal Care Products	33	0	33	$1,650.00	$412.50	AXYS Environmental Laboratory	$68,062.50*
Perfluoroalkyl Substances	33	0	33	$350.00	$87.50		$14,437.50*
Total Solids	33	3 (field split)	36	$5.00†	--	Analytical Resources, Inc.	$180.00*
Total Sulfides (bulk sediment)	33	3 (field split)	36	$30.00†	--		$1,080.00*
Total Sulfides (porewater)	33	3 (field split)	36	$20.00†	--		$720.00*
Ammonia (NH_3) (bulk sediment)	33	3 (field split)	36	$25.00†	--		$900.00*
Ammonia (NH_3) (porewater)	33	3 (field split)	36	$15.00†	--		$540.00*
Porewater Extraction			36	$75.00	--	Materials Testing and Consulting, Inc. (MTC)	$2,700.00*
Archive bottles			36	$2.50	--	--	$90.00*
Grain Size	33	3 (field split)	36	$80.00	--	MTC	$2,880.00**

Parameter	Number of Samples	Number of QA Samples	Total Number of Samples	Cost Per Sample	MEL 25% Overhead/Sample	Lab	Total
Amphipod survival toxicity test	33	0	33	$450.00	--	Northwestern Aquatic Services	$14,850.00**
Sea urchin fertilization toxicity test	33	0	33	$450.00	--		$14,850.00**
Sample courier			2 trips	$425.00	--	Dependable Courier Service	$850.00*
Taxonomic identification	33	0	33	$420.00	--	Contract Regional Taxonomists	$13,860.00**
						Total:	$174,782.80

† QC samples included in cost
* 2013-2015 biennium
** 2015-2017 biennium

6.0 Quality Objectives

6.2 Measurement Quality Objectives

The Measurement Quality Objectives (MQOs) for ammonia and total sulfides are given in Table 3; the MQOs for pharmaceuticals, personal care products, and perfluoroalkyl substances are given in Table 4. MQOs for all other parameters are given in the original QAPP (Dutch et al., 2009) and remain unchanged.

Table 3. Laboratory measurement quality objectives for laboratory analysis for ammonia (NH_3) and total sulfides (TS) in bulk sediments and in porewater.

<table>
<tr><th>Parameter</th><th>Field Blank</th><th>Field Replicate</th><th>Initial Calibration</th><th>Continuing Calibration</th><th>Calibration Blanks</th><th>Laboratory Control Samples</th><th>Matrix Spikes</th><th>Laboratory Replicates</th><th>Method Blank</th></tr>
<tr><td>Total Solids</td><td rowspan="5">RPD < 20%</td><td rowspan="5">Duplicate analysis for 10% of samples, RPD < 20%</td><td>Not applicable</td><td>Not applicable</td><td>Not applicable</td><td>Not applicable</td><td>Not applicable</td><td rowspan="5">Triplicate analyses on one of every 20 samples; 20% Relative Standard Deviation (RSD)</td><td rowspan="5">Analyte concentration < PQL</td></tr>
<tr><td>Ammonia (bulk sediments)</td><td rowspan="3">Correlation coefficient for the standard curve > 0.990</td><td rowspan="2">90 -110% recovery (calibration verification blank should return a value within 10% of its prepared concentration)</td><td rowspan="2">Analyte concentration < Practical Quantitation Limit (PQL)</td><td rowspan="2">80 -120% recovery</td><td rowspan="3">75 -125% recovery</td></tr>
<tr><td>Ammonia (porewater)</td></tr>
<tr><td>Total Sulfides (bulk sediments)</td><td rowspan="2">85 -115% recovery</td><td rowspan="2">Not applicable</td><td rowspan="2">65 -135% recovery</td></tr>
<tr><td>Total Sulfides (porewater)</td></tr>
</table>

Table 4. Laboratory measurement quality objectives for pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs).

Parameter	Field Blank	Field Replicate (Split Sample)	Analytical (Laboratory) Replicate	Laboratory Control Sample	Reference Material	Method Blank	Matrix Spike (and Matrix Spike Duplicate)	Surrogate Spike
Pharmaceuticals and Personal Care Products (PPCPs)	RPD < 20%	RPD < 20%	Compound-specific RPD < 40%	Compound-specific	Not applicable	Analyte concentration < MDL; if ≥ MDL, lowest analyte conc'n must be ≥ 10x blank conc'n	Not applicable	Compound-specific
Perfluoroalkyl Substances (PFASs)	RPD ≤ 20%	RPD ≤ 20%	Compound-specific RPD ≤ 40%	Compound-specific	Not applicable	Analyte concentration < MDL; if ≥ MDL, lowest analyte conc'n must be ≥ 10x blank conc'n	Recovery compound-specific; RPDs < 40%	Compound-specific

RPD: Relative Percent Difference

MDL: Method Detection Limit

Method Blanks - analyzed to assess possible laboratory contamination of samples associated with all stages of preparation and analysis of sample extracts.

Surrogate Spike Compounds - a type of check standard that is added to each sample in a known amount prior to extraction or purging.

Analytical replicates - provide precision information on the actual samples; useful in assessing potential samples heterogeneity and matrix effects.

Matrix Spikes - percent recoveries of matrix spikes are reported, should include a wide range of representative analyte types, compounds should be spiked about 5x the concentration of compounds in the sample or 5x the quantification limit.

Laboratory Control Samples - sometimes called check standards or laboratory control samples, are method blanks spiked with surrogate compounds and analytes; useful in verifying acceptable method performance prior to and during routine analysis of samples.

Reference Materials - a material or substance whose property values are sufficiently well established to be used for calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

7.0 Sampling Process Design

7.1 Study Design

7.1.2 Station Locations

A total of 33 stations will be sampled. Those same 33 sites were originally selected and sampled in 1998, and resampled in 2009 (Figure 1, Table 5). Alternate station locations are proposed, in case a station location cannot be sampled (Figure 2, Table 6).

Table 5. Locations (latitude/longitude) for 2015 PSEMP Urban Bays Bainbridge Basin sediment survey.

Target Station	Stratum	Location	Station Location (NAD 83, decimal degrees)	
			Latitude	Longitude
124	Rural	Port Madison	47.71381	-122.52732
125	Rural	Port Madison	47.73306	-122.53726
126	Rural	Port Madison	47.72603	-122.53051
142	Urban	Liberty Bay	47.72316	-122.64702
143	Urban	Liberty Bay	47.72035	-122.64899
144	Urban	Liberty Bay	47.72183	-122.64211
145	Passage	Keyport	47.71468	-122.62932
146	Passage	Keyport	47.71939	-122.64130
147	Passage	Keyport	47.70651	-122.63555
148	Passage	North West Bainbridge	47.69294	-122.61013
149	Passage	North West Bainbridge	47.68877	-122.58892
150	Passage	North West Bainbridge	47.68123	-122.58550
151	Passage	South West Bainbridge	47.64943	-122.60349
152	Passage	South West Bainbridge	47.60237	-122.58907
153	Passage	South West Bainbridge	47.62584	-122.58124
154	Passage	Rich Passage	47.59342	-122.53736
155	Passage	Rich Passage	47.60060	-122.55375
156	Passage	Rich Passage	47.57922	-122.58412
157	Passage	Port Orchard	47.56905	-122.60235
158	Passage	Port Orchard	47.56951	-122.58731
159	Passage	Port Orchard	47.56620	-122.61089
160	Harbor	Sinclair Inlet	47.53423	-122.67688
161	Harbor	Sinclair Inlet	47.54373	-122.64146
162	Harbor	Sinclair Inlet	47.54724	-122.64148
163	Harbor	Sinclair Inlet	47.54572	-122.65406
164	Harbor	Sinclair Inlet	47.54900	-122.66538

Target Station	Stratum	Location	Station Location (NAD 83, decimal degrees)	
			Latitude	Longitude
165	Harbor	Sinclair Inlet	47.54726	-122.66643
166	Passage	Port Washington Narrows	47.60889	-122.66347
167	Passage	Port Washington Narrows	47.58473	-122.66301
168	Passage	Port Washington Narrows	47.58835	-122.65993
169	Urban	Dyes Inlet	47.63572	-122.67908
170	Urban	Dyes Inlet	47.61308	-122.70134
171	Urban	Dyes Inlet	47.62739	-122.69190

Table 6. Alternate locations (latitude/longitude) for the 2015 PSEMP Urban Bays Bainbridge Basin sediment survey.

Alternate Station	Station Location (NAD 83, decimal degrees)	
	Latitude	Longitude
10	47.60193	-122.67997
26	47.59712	-122.55054
28	47.61891	-122.67585
40	47.73365	-122.65264
74	47.53151	-122.67685
106	47.54641	-122.64810
108	47.63527	-122.69844
136	47.72145	-122.50603
138	47.59444	-122.68152
156	47.61800	-122.69088
168	47.71895	-122.53153
170	47.54814	-122.65487
202	47.56099	-122.59580
232	47.69089	-122.59153
264	47.68104	-122.60264
306	47.54440	-122.63915
322	47.53619	-122.66865

7.1.3 Parameters Sampled

Standard sediment quality field measurements, macroinvertebrate abundance, grain size, total organic carbon, and metals will continue to be collected as per Dutch et al., 2009. A reduced list of organics will also be collected. Ammonia (NH_3) and total sulfides in bulk sediments and porewater are being added due to their potential toxicity to the benthic invertebrate assemblages (Table 7). A comparison will be conducted to determine differences in the levels of these parameters in the two sediment fractions. The parameter of total solids is added to allow for calculation of these ammonia and total sulfide values for bulk sediments in dry weight. Pharmaceuticals and personal care products (PPCP), and perfluoroalkyl substances (PFAS) are added to establish baseline data for these chemicals.

Table 7. Parameters measured in sediments for the 2015 PSEMP Urban Bays Bainbridge Basin sediment survey.

Field Measurements
Sediment temperature
Salinity of overlying water

Macroinvertebrate Abundance
Total Abundance
Major Taxa Abundance
Taxa Richness
Calculated values:
- Pielou's Evenness
- Swartz Dominance Index

Conventionals
Grain size
Total organic carbon
Ammonia
Total sulfides
Total solids

Toxicity
Sea urchin fertilization success (porewater)
Amphipod survival (bulk sediment)

Metals
Priority Pollutant Metals
Arsenic
Cadmium
Chromium
Copper
Lead
Mercury
Nickel
Selenium
Silver
Zinc

Element
Tin

Organics

Phthalate Esters
Bis(2-Ethylhexyl) Phthalate
Butylbenzylphthalate
Diethylphthalate
Dimethylphthalate
Di-N-Butylphthalate
Di-N-Octyl Phthalate

Polycyclic Aromatic Hydrocarbons

LPAHs
1,6,7-Trimethylnaphthalene
1-Methylnaphthalene
1-Methylphenanthrene
2,6-Dimethylnaphthalene
2-Methylnaphthalene
2-Methylphenanthrene
Acenaphthene
Acenaphthylene
Anthracene
Biphenyl
Dibenzothiophene
Fluorene
Naphthalene
Phenanthrene
Retene
Calculated values:
- total LPAH

HPAHs
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo[e]pyrene
Benzo(g,h,i)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Indeno(1,2,3-c,d)pyrene
Perylene
Pyrene
Calculated values:
- total HPAH
- total Benzofluoranthenes

Polybrominated Diphenylethers
PBDE-47
PBDE-49
PBDE-66
PBDE-71
PBDE-99
PBDE-100
PBDE-138
PBDE-153
PBDE-154
PBDE-183
PBDE-184
PBDE-191
PBDE-209

Polychlorinated Biphenyls

Aroclors
PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260
PCB-1262
PCB-1268

Congeners
PCB-8
PCB-18
PCB-28
PCB-44
PCB-52
PCB-66
PCB-77
PCB-101
PCB-105
PCB-118
PCB-126
PCB-128
PCB-138
PCB-153
PCB-169
PCB-170
PCB-180
PCB-187
PCB-195
PCB-206
PCB-209

Personal Care Products and Pharmaceuticals

List 1 - Acid Extraction in Positive Ionization

Acetaminophen
Ampicillin 1
Azithromycin
Caffeine
Carbadox
Carbamazepine
Cefotaxime
Ciprofloxacin
Clarithromycin
Clinafloxacin
Cloxacillin
Dehydronifedipine
Digoxigenin
Digoxin
Diltiazem
1,7-Dimethylxanthine
Diphenhydramine
Enrofloxacin
Erythromycin-H20
Flumequine
Fluoxetine
Lincomycin
Lomefloxacin
Miconazole
Norfloxacin
Norgestimate
Ofloxacin
Ormetoprim
Oxacillin
Oxolinic acid
Penicillin G
Penicillin V
Roxithromycin
Sarafloxacin
Sulfachloropyridazine
Sulfadiazine
Sulfadimethoxine
Sulfamerazine
Sulfamethazine
Sulfamethizole
Sulfamethoxazole
Sulfanilamide
Sulfathiazole
Thiabendazole
Trimethoprim
Tylosin
Virginiamycin

List 2 - Tetracyclines in Positive Ionization

Anhydrochlortetracycline
Anhydrotetracycline
Chlortetracycline
Demeclocycline
Doxycycline
4-Epianhydrochlortetracycline
4-Epianhydrotetracycline
4-Epichlortetracycline
4-Epioxytetracycline
4-Epitetracycline
Isochlortetracycline
Minocycline
Oxytetracycline
Tetracycline

List 3 - Acid Extraction in Negative Ionization

Bisphenol A
Furosemide
Gemfibrozil
Glipizide
Glyburide
Hydrochlorothiazide
2-hydroxy-ibuprofen
Ibuprofen
Naproxen
Triclocarban
Triclosan
Warfarin

List 4 - Basic Extraction in Positive Ionization

Albuterol
Amphetamine
Atenolol
Atorvastatin
Cimetidine
Clonidine
Codeine
Cotinine
Enalapril
Hydrocodone
Metformin
Oxycodone
Ranitidine
Triamterene

List 5 - Acid Extraction in Positive Ionization

Alprazolam
Amitriptyline
Amlodipine
Benzoylecgonine
Benztropine
Betamethasone
Cocaine
DEET
Desmethyldiltiazem
Diazepam
Fluocinonide
Fluticasone propionate
Hydrocortisone
10-hydroxy-amitriptyline
Meprobamate
Methylprednisolone
Metoprolol
Norfluoxetine
Norverapamil
Paroxetine
Prednisolone
Prednisone
Promethazine
Propoxyphene
Propranolol
Sertraline
Simvastatin
Theophylline
Trenbolone
Trenbolone acetate
Valsartan
Verapamil

Perfluoroalkyl Substances

Carboxylic Acids

Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorohexanoate (PFHxA)
Perfluoroheptanoate (PFHpA)
Perfluorooctanoate (PFOA)
Perfluorononanoate (PFNA)
Perfluorodecanoate (PFDA)
Perfluoroundecanoate (PFUnA)
Perfluorododecanoate (PFDoA)

Sulphonic Acids

Perfluorobutanesulfonate (PFBS)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanesulfonate (PFOS)
Perfluorooctane sulfonamide (PFOSA)

7.2 Maps

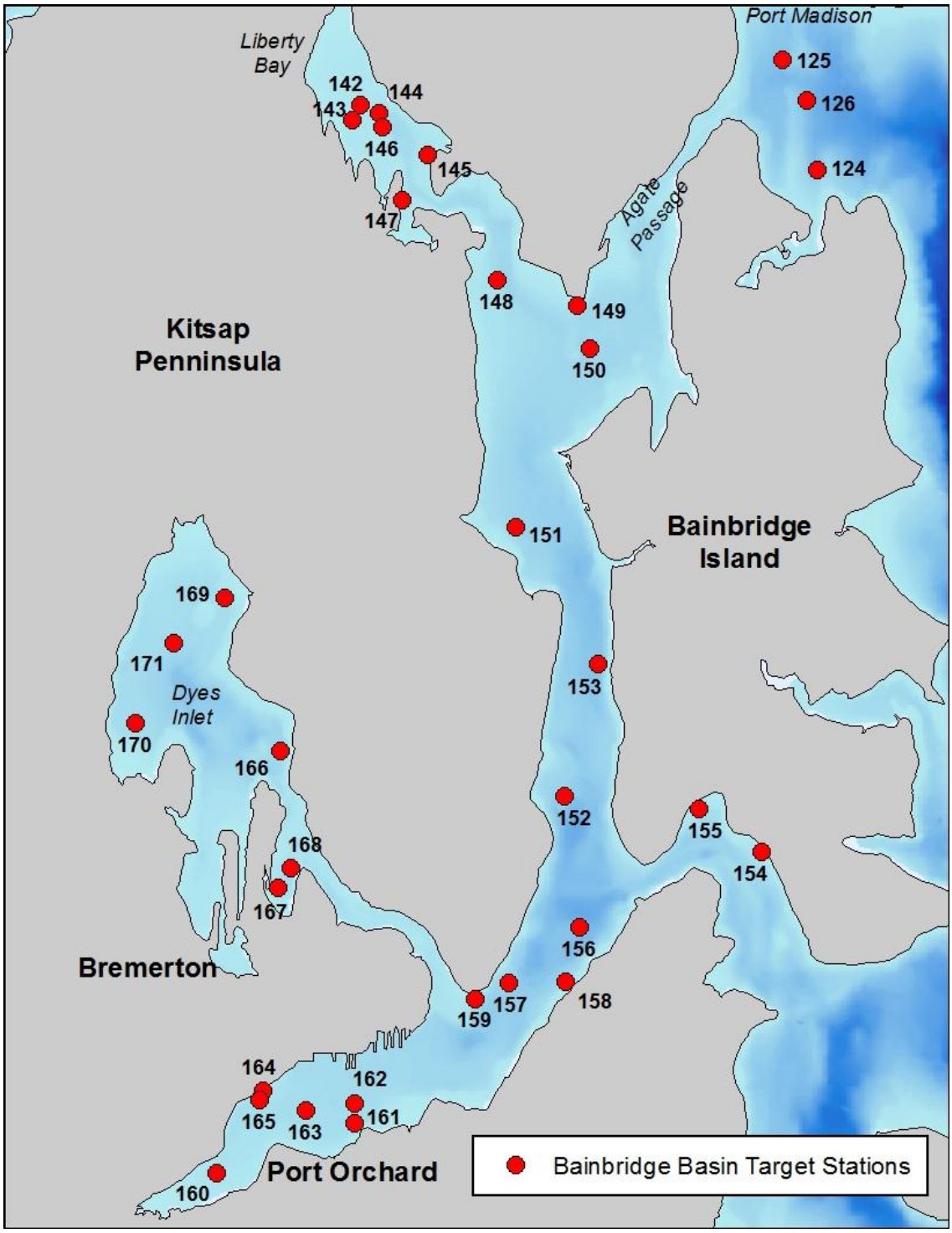

Figure 1. Target stations for the 2015 PSEMP Urban Bays Bainbridge Basin sediment survey.

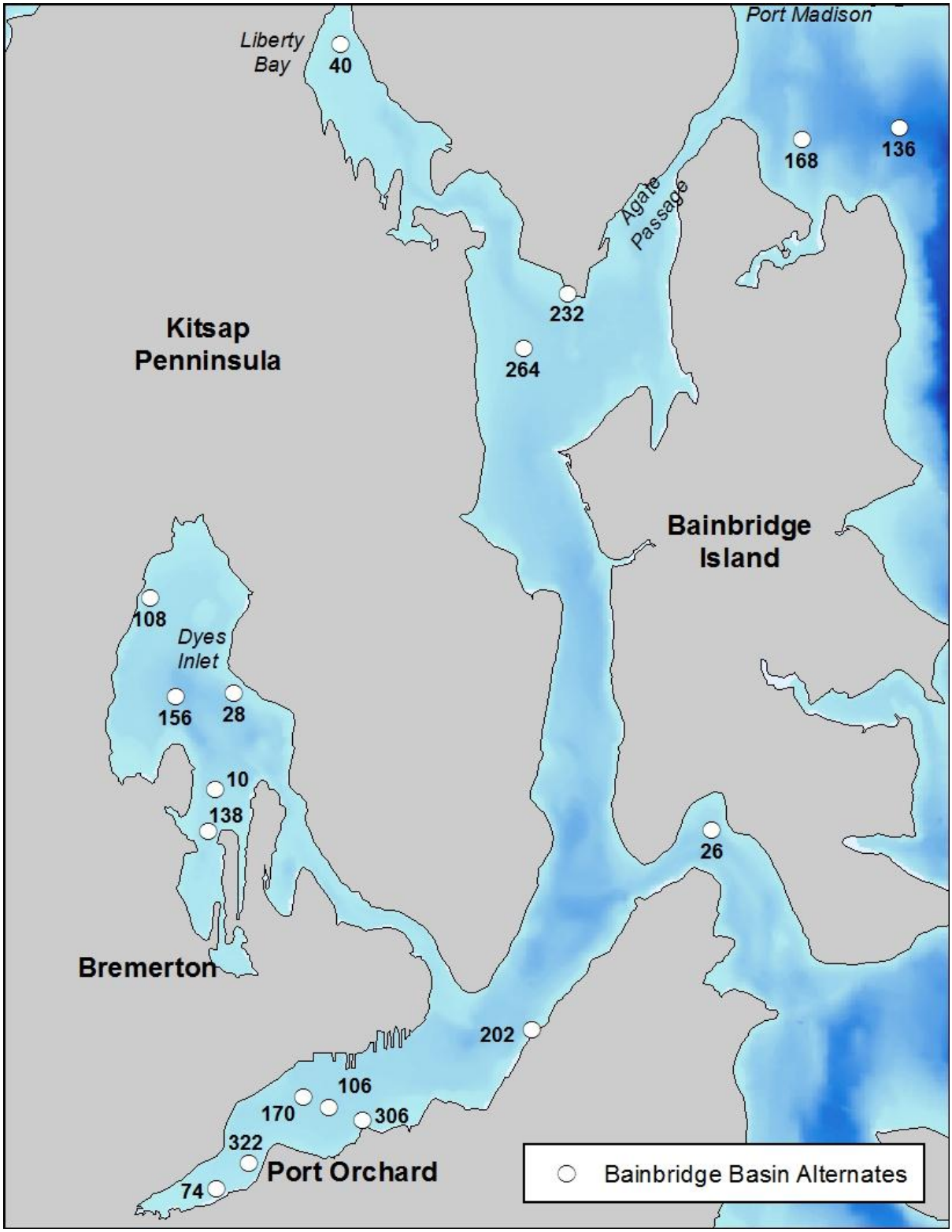

Figure 2. Alternate stations for the 2015 PSEMP Urban Bays Bainbridge Basin sediment survey.

8.0 Sampling Procedures

8.2 Containers, Preservation Methods, Holding Times

Sample collection and preservation of sediment samples for analyses for ammonia, total sulfides, pharmaceuticals, personal care products, and perfluoroalkyl substances are given in Table 8. Sample collection and preservation of sediment samples for all other analyses are given in the original QAPP (Dutch et al., 2009).

9.0 Measurement Methods

9.2 Lab Procedures

Laboratory analysis and reporting requirements for analyses for ammonia, total sulfides, pharmaceuticals, personal care products, and perfluoroalkyl substances are given in Table 9. Laboratory analysis and reporting requirements for all other analyses are given in the original QAPP (Dutch et al., 2009).

Table 8. Sample collection and preservation for analyses for ammonia (NH3) and total sulfides (TS) in bulk sediments and in porewater, and for analyses for pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs) in homogenized sediment.

Parameter	Size of Sediment Sample	Container	Preservation	Maximum Holding Time
Total Solids (bulk sediments)	4 oz (50g for lab work)	4 oz wide-mouth glass jar with Teflon-lined lid	Refrigerate at 4ºC	14 days
Ammonia (bulk sediments)	4 oz (25g for lab work)	4 oz wide-mouth glass jar with Teflon-lined lid	Refrigerate at 4ºC, sample should not be homogenized in field, no headspace or air pockets should remain	7 days
Total Sulfides (bulk sediments)	2 oz (50g for lab work)	2 oz wide-mouth glass jar with Teflon-lined lid	4°C, 5ml of 2 N zinc acetate for a 250 ml sample, sample should not be homogenized in field, no headspace or air pockets should remain	7 days
Ammonia/ Total Sulfides (porewater)	32 oz (600g for lab work)	32 oz wide mouth glass jar with Teflon-lined lid	Refrigerate at 4ºC, sample should not be homogenized in field, no headspace or air pockets should remain	7 days
Pharmaceuticals and Personal Care Products (PPCPs) (homogenized sediment)	8 oz	8 oz HDPE internally certified by contract lab	Wrap in aluminum foil and place in ice chest with dry ice immediately after field collection. Freeze as soon as possible. Store in dark at less than -10°C until analyzed	* Freezing encouraged to minimize degradation. Extract within 48 hours if not frozen or within 7 days of collection if frozen. Extract within 48 hours of removal from freezer. Analyze extracts within 40 days of extraction.
Perfluoroalkyl Substances (PFASs) (homogenized sediment)	8 oz	8 oz HDPE internally certified by contract lab	Refrigerate at 4ºC±2ºC (CAS)	* 14 days to extraction (CAS)

* These are suggested holding times only. Formal holding time studies have not been performed or published for this analysis.

Table 9. Laboratory analysis and reporting requirements for ammonia (NH3) and total sulfides (TS) in bulk sediments and in porewater, and for pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs) in sediments.

Parameter	Extraction Method	Clean-up Method	Analysis Method	Technique/ Instrument	Expected Range of Results	Required Reporting Limit
Total Solids	Not applicable	Not applicable	PSEP, 1986/ASTM D-422	Muffle furnace – 550°C	0.01 – 100%	0.01%
Ammonia (bulk sediments)	Not applicable	Not applicable	Plumb, 1981/EPA 350.1M (sed); Standard Methods, 1995 4500-NH3 H or EPA 350.1M (water)	Automated phenate, flow injection analysis (FIA). Measures ammonia as NH3-N under alkaline conditions.	Unknown	0.1 mg/Kg
Ammonia (porewater)	Centrifugation of bulk sediments (DMMP/SMS, 1998)	Not applicable			0.01 – 1.00 mg/L	0.01 mg/L
Total Sulfides (bulk sediments) (PSEP, 1986)	Sediment is acidified under anoxic conditions to release sulfide as H_2S. The released H_2S gas is then trapped in zinc acetate solution to precipitate sulfide (as zinc or sodium sulfide). Finish analysis is conducted on the trapping solution.	Not applicable	Plumb, 1981; Standard Methods, 1995 4500-S^{2-} D-00; PSEP, 1986	Iodometric titration and methylene blue colorimetry	1.0 mg/kg	10.0 mg/kg dry weight (to nearest 0.1 unit)
Total Sulfides (porewater)	Centrifugation of bulk sediments (DMMP/SMS, 1998)	Not applicable			0.05 mg/L	
Pharmaceuticals and Personal Care Products (PPCPs)	Sonication with aqueous buffered acetonitrile and pure acetonitrile, concentrate then dilute with ultra pure water.	Solid-phase extraction cartridge then filtered	USEPA 1694	HPLC/ESI-MS/MS. High performance liquid chromatography with triple quadrupole mass spectrometer in positive and negative electrospray ionization modes using isotope dilution and internal standard quantitation techniques	Unknown	1-1,000 µg/kg dry weight

Parameter	Extraction Method	Clean-up Method	Analysis Method	Technique/ Instrument	Expected Range of Results	Required Reporting Limit
Perfluoroalkyl Substances (PFASs)	Shake extraction with dilute acetic acid solution then methanolic ammonium hydroxide solution. Combine supernatants and treat with ultra pure carbon powder and diluted with ultra pure water.	Weak anion exchange sorbent solid-phase extraction	MLA-041. Internal AXYS method	HPLC/ESI-MS/MS. High performance liquid chromatography with triple quadrupole mass spectrometer in negative electrospray ionization mode using internal standard.	Unknown	0.1 µg/kg dry weight

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15.0 References

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