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ECOLOGY
State of Washington

2019 Addendum to Quality Assurance Monitoring Plan

The Puget Sound Sediment Monitoring Program

February 2020

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2019 Addendum to Quality Assurance Monitoring Plan

The Puget Sound Sediment Monitoring Program

February 2020

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EAP: Environmental Assessment Program

NEP: National Estuary Program

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2.0 Abstract

This addendum to the 2018 Quality Assurance Monitoring Plan (QAMP) developed for the Puget Sound Sediment Monitoring Program (Sediment Program) provides details about changes in 2019 to the Long-Term and Urban Bays elements of the program. It also includes information regarding three additional projects for 2019 and leveraged sampling to be conducted for several regional partners. These projects include:

- **Pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs) in two urban bays:** A suite of 24 PFASs will be measured in the Sediment Program's Port Gardner/Everett Harbor and Budd Inlet sampling frames. Also, 119 PPCPs will be measured in the Budd Inlet sampling frame.
- **Cornwall Avenue Landfill sediment sampling:** Sediment grab samples will be collected from three Bellingham Bay intertidal stations at the request of the Washington State Department of Ecology's (Ecology) Toxics Cleanup Program (TCP).
- **Invertebrate Genomics Initiative:** Benthic invertebrates will be collected at Long-Term stations for photographic image archiving, DNA sequencing, and morphological identification, as part of the Western Association of Marine Laboratories (WAML) and Smithsonian Institution's West Coast Invertebrate Genomics Initiative.
- **Leveraged sampling with regional partners:** Additional sediment samples will be collected during Sediment Program sampling and analyzed by other regional researchers to further our understanding of Puget Sound sediment quality.

Quality assurance elements not mentioned in this addendum remain unchanged for the Sediment Program and are as described in the 2018 QAMP (Dutch et al., 2018).

4.0 Project Description

Puget Sound Sediment Monitoring Program

The two annual monitoring elements of the Sediment Program include:

- **Long-Term monitoring:** Annual characterization and change over time of sediment quality and the condition of benthic invertebrates (benthos) Puget Sound-wide as estimated from samples collected from 50 randomly and non-randomly selected stations. These samples will be collected in April 2019 and measured for the standard suite of parameters as described in Dutch et al., 2018.
- **Urban Bays monitoring:** Periodic characterization and change over time of sediment quality and benthos condition bay-wide as estimated from samples collected from one of six urban bays sampled on an annual rotational basis. Thirty samples will be collected from the Port Gardner/Everett Harbor sampling frame in June 2019 and measured for the standard suite of parameters as described in Dutch et al., 2018.

Pharmaceuticals and Personal Care Products (PPCPs) and Perfluoroalkyl Substances (PFASs) in Urban Bays

A suite of 24 perfluoroalkyl substances (PFASs) will be measured in sediments from 30 stations in Port Gardner/Everett Harbor and from 30 stations in Budd Inlet. A suite of 119 personal care products and pharmaceuticals (PPCPs) will also be measured for Budd Inlet sediments. If additional funding becomes available, the PPCPs will also be measured for sediments from the Port Gardner/Everett Harbor stations.

Cornwall Avenue Landfill

The [Cornwall Avenue Landfill](#) is a contaminated site on the Bellingham waterfront. Cleanup of this site is regulated by Washington's Model Toxics Control Act (MTCA; [Chapter 70.105D RCW](#)) and overseen by Ecology's TCP (Washington State Department of Ecology 2014, 2015). The TCP has requested collection of three intertidal sediment grab samples from a beach adjacent to this site. These sediments will be tested for biogeochemical and chemical parameters that will inform ongoing cleanup work at this site.

Invertebrate Genomics Initiative

We will also participate in the West Coast Invertebrate Genomics Initiative during the April 2019 Long-Term field sampling effort. The Initiative was developed as a partnership between members of the [Western Association of Marine Laboratories](#) (WAML) and the [Smithsonian National Museum of Natural History's Global Genome Initiative](#) (Smithsonian) to collect and sequence DNA material from benthic invertebrates collected along the west coast of North America, from Southern California through Alaska.

We will take advantage of our normal field sampling opportunities to collect invertebrates and submit tissue for genetic sequencing, while the Smithsonian will conduct all genetic sequencing for these tissue samples. Genetic information generated by the Smithsonian will be used for:

- **Enhancement of the Puget Sound DNA Barcode Library:** DNA barcode information for Puget Sound invertebrates will be available to regional researchers working on genomics-based studies such as development of eDNA rapid bioassessment tools.
- **Validation of Cosmopolitan Species:** DNA barcode information from selected species of cosmopolitan North American west coast invertebrates will be used to validate their morphological taxonomic designations. Such information will be useful for determining geographic shifts in coastal species distribution over time due to climate change-related pressures.

Partnerships with Other Monitoring Programs: Leveraged Sampling and Data

To gain additional scientific knowledge from our fieldwork and data, integrated partnerships, both long-standing and new, have been formed with regional scientists to generate sediment quality-related data that will help us interpret the conditions and changes over time in Puget Sound sediments and benthos. For the 2019 Sediment Program, topics and collaborators include:

eDNA Analysis of Puget Sound Sediments

Partner: Dr. Carol Stepien, NOAA Pacific Marine Environmental Lab (PMEL), Seattle, WA (new for 2019)

Sediment Program samples will be analyzed for eDNA by Dr. Stepien at NOAA’s PMEL for generation of qualitative species composition information at each station. Results will be verified by comparison with our benthos data collected at the same stations. These eDNA methods will be considered for future development of a rapid biodiversity assessment tool for Puget Sound benthos.

Microplastics

Partner: Julie Masura, University of Washington (since 2015)

We will continue to provide Masura and her students with approximately 200 ml of sediment collected from each station sampled. Plastics are recovered from the sediments, counted, measured, and mapped annually. When available, we will use these data to calculate the spatial extent (# pieces/km²) of the plastics for the Puget Sound-wide and Urban Bays sampling frames.

Harmful Algal Blooms

Partners: Dr. Cheryl Greengrove and Julie Masura, University of Washington—Tacoma (since 2013)

We will continue to provide Dr. Greengrove and Masura with approximately 4 oz of sediment collected from each station sampled. They will examine the abundance and distribution of *Alexandrium* sp. cysts in these sediments (e.g., PSEMP Marine Waters Workgroup, 2018). *Alexandrium* sp. is a dinoflagellate that spends part of its life cycle as a cyst in the sediment before germinating to become a vegetative cell. This species produces a suite of neurotoxins that can accumulate in the tissues of filter-feeding shellfish and can be lethal to humans if ingested. This ongoing study evaluates whether the location or concentration of cysts exhibit patterns that

can be associated with shellfish bed closures due to the presence of paralytic shellfish toxins (PSTs) in shellfish above regulatory limits.

Foraminifera Monitoring

Partners: Dr. Liz Nesbitt and Dr. Ruth Martin, University of Washington (since 1997)

We will continue to provide Dr. Nesbitt and Dr. Martin with approximately 4 oz of sediment collected from each Sediment Program station sampled. They and their students will examine the type, abundance, and distribution of foraminifera identified in these samples. Foraminifera, marine protozoans with calcium carbonate or agglutinated sediment particle tests (shells), are an important component of the benthos. Their community structure and physical condition are sensitive indicators of chemical pollution and ocean acidification. Information generated is published in student reports and posters as well as in the primary literature (Martin and Nesbitt, 2015; Nesbitt et al., 2015; Martin and Nesbitt, 2017).

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Puget Sound Sediment Monitoring Program

- Dr. Arati Kaza, Environmental Assessment Program, 360-407-6964.

Dr. Kaza is the new Quality Assurance Officer for Ecology's Environmental Assessment Program and for all of Ecology.

Other key Ecology staff and their responsibilities, listed in Table 2 in Dutch et al. (2018), remain unchanged.

PPCPs and PFASs in Urban Bays

- Dr. Ginna Grepo-Grove, Manchester Environmental Laboratory (MEL), 360-871-8829.
- John Weakland, MEL, 360-871-6543.

Dr. Ginna Grepo-Grove and John Weakland will be responsible for contracting with a commercial chemistry laboratory for PPCP analysis, reviewing all lab results, and generating a final data package and electronic data distribution (EDD) file.

Cornwall Avenue Landfill

Clients

- Lucy McInerney, Ecology's Toxic Cleanup Program, Northwest Regional Office (NWRO), 425-649-7272.
- Mark Adams, Toxic Cleanup Program, NWRO, 425-649-7107.
- Dr. Ginna Grepo-Grove, MEL, 360-871-8829.
- John Weakland, MEL, 360-871-6543.

Responsibilities of the clients include defining the scope of the project and internal review and also approving the QAMP addendum. Dr. Ginna Grepo-Grove or John Weakland will review contract lab PCB results and prepare the final narrative and EDD for the clients.

Invertebrate Genomics Initiative

Partners

- Dr. Gustav Paulay, Research Associate, Friday Harbor Marine Laboratories and Curator of Mollusca, Florida Museum of Natural History (FMNH), paulay@flmnh.ufl.edu.

We will work in partnership with Dr. Gustav Paulay on the Invertebrate Genomics Initiative in April 2019. Dr. Paulay will share his expertise and train Ecology staff in methods for sieving, sorting, photography, and cataloguing of invertebrate specimens and also in collection and preservation of tissue for DNA barcoding (see Appendix).

- Dr. David Gillett, Benthic Ecologist, Southern California Coastal Water Resource Project, davidg@sccwrp.org.

Dr. Gillett will manage the Cosmopolitan Species Barcoding Project for the Invertebrate Genomics Initiative. His responsibilities include oversight of all aspects of the project and coordination of data review and report writing.

- Dana Schultz, Lab Technician, Southern California Coastal Water Resource Project, danas@sccwrp.org.

Dana Schultz will be responsible for receipt of specimens from all collection events for the Cosmopolitan Species Barcoding Project, as well as coordination of taxonomic identification, tissue preparation, and shipping to the Smithsonian.

- Dr. Chris Meyer, Research Zoologist and Curator of Mollusks, National Museum of Natural History, MeyerC@si.edu.
- Michael O'Mahoney, Museum Technician, Invertebrate Zoology, National Museum of Natural History, OmahoneyM@si.edu.

Dr. Chris Meyer and Michael O'Mahoney will be responsible for receipt and processing of all tissue collected for DNA barcoding, and for generation and sharing of the barcoding data produced.

5.4 Proposed project schedule

Puget Sound Sediment Monitoring Program

The proposed project schedule remains unchanged for the annual Long-Term and Urban Bays sediment monitoring elements (Tables 3 and 4, respectively, in Dutch et al., 2018).

PPCPs and PFASs in Urban Bays

Table 1. Proposed schedule for completing the PPCPs and PFASs project.

| Field and laboratory work. | Due date | Lead staff |
|--|---|--|
| Sample collection completed | June 2019 | Margaret Dutch |
| Laboratory analyses completed by contract lab (PPCP) | July 2019 | Ginna Grepo-Grove, MEL and/or John Weakland, MEL |
| Laboratory analyses completed by MEL (PFAS) | December 2019 | John Weakland, MEL |
| Data review | January 2020 | Ginna Grepo-Grove, MEL and/or John Weakland, MEL |
| Environmental Information System (EIM) database | | |
| EIM data loaded | February 2020 | Sandra Weakland |
| EIM data entry review | February 2020 | MSMT staff — will vary |
| EIM complete | February 2020 | Sandra Weakland |
| Final report | | |
| Author lead / Support staff | Margaret Dutch/Sandra Weakland, Valerie Partridge | |
| Schedule | | |
| Draft due to supervisor | September 2020 | |
| Draft due to client/peer reviewer | October 2020 | |
| Draft due to external reviewer(s) | October 2020 | |
| Final (all reviews done) due to publications coordinator | November 2020 | |
| Final report due on web | December 2020 | |

MEL: Ecology's Manchester Environmental Laboratory

MSMT: Marine Sediment Monitoring Team

Cornwall Avenue Landfill

Table 2. Proposed schedule for completing the Cornwall Avenue Landfill project.

| Field and laboratory work | Due date | Lead staff |
|--|-----------------|--|
| Sample collection completed | March 2019 | Margaret Dutch |
| Laboratory analyses completed at Manchester Environmental Laboratory (MEL) — TOC, PAHs | June 2019 | Dean Momohara, John Weakland, MEL |
| Laboratory analyses completed — PCBs (contract lab) | June 2019 | GINNA GREPO-GROVE, MEL and/or John Weakland, MEL |
| Environmental Information System (EIM) database | | |
| EIM Study ID | MDUT0001 | |
| Product | Due date | Lead staff |
| EIM data loaded | August 2019 | Sandra Weakland |
| EIM data entry review | August 2019 | MSMT staff — will vary |
| EIM complete | August 2019 | Sandra Weakland |

Invertebrate Genomics Initiative

Table 3. Proposed schedule for completing the Invertebrate Genomics Initiative.

| Field and laboratory work | Due date | Lead staff |
|---|-----------------|---|
| Field sampling — benthos collection | April 2019 | Sandra Weakland Valerie Partridge |
| Photography, tissue sampling, specimen preservation | April 2019 | Dr. Gustav Paulay Margaret Dutch Dany Burgess Angela Eagleston |
| Species-level identification of benthos | June 2019 | Dany Burgess Angela Eagleston |
| Data/product management | | |
| DNA barcode data — archived in the Bar Code of Life (BoLD) and other databases. Photographic images — archived with the MSMT and at the FMNH. Voucher specimens — archived at the FMNH and at Ecology's Benthic Laboratory. | | |
| Data/Product | Due date | Lead staff |
| Specimen photographs | April 2019 | Dr. Gustav Paulay Dany Burgess |
| Voucher specimens and tissue samples | April 2019 | Dr. Gustav Paulay Dr. Chris Meyer Michael O'Mahoney |
| DNA barcode data set | December 2019 | Dr. Chris Meyer Michael O'Mahoney |
| Voucher specimen ID verification | December 2019 | Dany Burgess Angela Eagleston |

5.5 Budget and funding

Puget Sound Sediment Monitoring Program

The expected budget for all sampling and analyses for the 2019 Sediment Program is provided in Table 4. Payments will be made in Fiscal Years 2018 and 2019.

Table 4. 2019 Puget Sound Sediment Monitoring Program budget.

| Budget category/ parameter | Long-Term | Urban Bays | Grand Total |
|-------------------------------|-----------------|-----------------|------------------|
| Lab (MEL) | \$18,320 | \$40,190 | \$58,510 |
| Chemistry QA | \$2,170 | \$1,085 | \$3,255 |
| Convent/Nutr | \$5,300 | \$3,300 | \$8,600 |
| Metals/Organics | \$10,850 | \$35,805 | \$46,655 |
| Research vessel | \$6,675 | \$2,225 | \$8,900 |
| Skookum | \$6,675 | \$2,225 | \$8,900 |
| Sediment contracts | \$21,017 | \$14,357 | \$35,374 |
| Convent/Nutr | \$8,427 | \$5,247 | \$13,674 |
| Grain Size | \$5,035 | \$3,135 | \$8,170 |
| QA Taxonomy | \$7,555 | \$5,975 | \$13,530 |
| Travel | \$7,752 | \$3,620 | \$11,372 |
| Field travel | \$7,752 | \$3,620 | \$11,372 |
| Grand Total | \$53,764 | \$60,392 | \$114,156 |

PPCPs and PFASs in Urban Bays

The expected budget for analysis of PPCPs from Budd Inlet and PFASs from Pt. Gardner/Everett Harbor and Budd Inlet is provided in Table 5. PPCPs will be measured in Pt. Gardner/Everett Harbor as well, if additional funding becomes available.

Table 5. PPCPs and PFASs budget.

| Sampling frame/ parameter | # Samples +QA | Cost/ sample | Subtotal |
|----------------------------------|------------------|--------------------|------------------|
| Pt Gardner/Everett Harbor | | | |
| PFAS (MEL) | 35 | \$500 | \$17,500 |
| | | Subtotal | \$17,500 |
| Budd Inlet | | | |
| PFAS (MEL) | 35 | \$500 | \$17,500 |
| PPCPs (contract lab) | 35 | \$1,665 | \$58,275 |
| PPCPs (MEL overhead) | 35 | \$500 | \$17,500 |
| | | Subtotal | \$93,275 |
| | | Grand total | \$110,775 |

Cornwall Avenue Landfill

Table 6. Cornwall Avenue Landfill budget.

| Lab Analysis (TCP expense) | No. samples | Total # analyses | Cost/ sample | Total |
|---|---------------------------|-----------------------------|---------------------------|----------------|
| TOC | 3 | 3 | \$50 | \$150 |
| PAH - isotopic dilution extended (NOAA) list + C1-C4 | 3 | 3 | \$540 | \$1,620 |
| PCB congeners (high resolution) - contract lab | 3 | 3 | \$920 | \$2,760 |
| MEL 30% overhead (contracts, QA, validation) | | | | \$828 |
| | | | Subtotal: | \$5,358 |
| Sample courier (TCP expense) | No. deliveries | | Cost/ delivery | |
| Ground courier to drive samples to contract lab | 1 | | \$622 | \$622 |
| | | | Grand Total: | \$5,980 |

Invertebrate Genomics Initiative

Table 7. Invertebrate Genomics Initiative budget.

| Lab crew travel | No. staff | No. days, nights | Cost/day, night | Total |
|--|------------------|-----------------------------|----------------------------|-----------------|
| Lodging - 4/9-17/19 | 1 | 7 | \$127 | \$889 |
| Meals - 4/9-17/19 | 1 | 7 | \$66 | \$462 |
| Lodging - 4/22-30/19 | 3 | 5 | na | \$540 |
| Meals - 4/22-30/19 | 3 | 5 | \$55 | \$825 |
| Lodging - 5/1-2/19 | 1 | 2 | \$127 | \$254 |
| Meals - 5/1-2/19 | 1 | 2 | \$66 | \$132 |
| R/V Skookum | | | | |
| Captain (additional boat days, if needed) | 1 | 3 | \$425 | \$1,275 |
| Supplies | | | | |
| lab supplies | | | | \$5,000 |
| Sample courier | | | | |
| Estimate only | | 5 | \$200 | \$1,000 |
| | | | Grand Total: | \$10,377 |

Leveraged partnership sampling – eDNA, microplastics, harmful algal blooms, foraminifera

A small amount of sample collected from each Long-Term and Urban Bays station will be set aside for the eDNA, microplastics, harmful algal blooms, and foraminifera partnership projects described earlier. Costs incurred by the MSMT for collection of these samples are negligible.

6.0 Quality Objectives

6.2 Measurement quality objectives

6.2.1 Targets for precision, bias, sensitivity

Puget Sound Sediment Monitoring Program

The MQOs for Sediment Program project results are as described in Table 6 in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

The MQOs for the PPCPs and PFASs measured in the Port Gardner/Everett Harbor and Budd Inlet sampling locations are as described in Table 8, below.

Cornwall Avenue Landfill

The MQOs for the Cornwall Avenue Landfill project results are summarized in Table 9, below, for total organic carbon, PAHs, and high resolution PCB congeners.

Table 8. Laboratory measurement quality objectives for PPCPs and PFASs.

| MQO | Precision | | | Bias | | | | | Sensitivity |
|-----------|--|--|--|----------------------------------|--|-----------------------------------|-----------------------|---|---|
| Parameter | Field Replicate (Split Sample) | Analytical (Laboratory) Replicate | Matrix Spike Duplicates (MSD) | Laboratory Control Sample (LCS) | Standard or Certified Reference Material (SRM/CRM) | Matrix Spikes (MS) | Surrogate Spike | Method Blank | Method Detection Limit (MDL) ² or Lowest Concentration of Interest |
| | Relative Percent Difference (RPD) or Relative Standard Deviation (RSD) | | | Recovery Limits (%) ¹ | | | | Comparison of analyte concentration in blank to quantification limit | Concentration Units |
| PPCPs | RPD < 40% | If conc. < 5 times R.L., RPD < 40% for 60% of analytes | Not applicable | Compound-specific | Not applicable | Not applicable | Compound-specific | Analyte concentration < MDL; if ≥ MDL, lowest analyte conc'n must be ≥ 10x blank conc'n | 0-348 ng/g based on 0.5 g sample |
| PFASs | RPD ≤ 40% | Compound-specific RPD ≤ 40% | Recovery compound-specific; RPDs < 40% | 50%-150% ³ | Not applicable | 40%-160%; RPDs < 40% ³ | 20%-200% ³ | Analyte concentration < MDL; if ≥ MDL, lowest analyte conc'n must be ≥ 10x blank conc'n | 0.5 µg/kg dry weight (0.25 ug/Kg wet weight) |

Table 9. Measurement quality objectives for the Cornwall Avenue Landfill project.

| MQO | Precision | | | Bias | | | | Sensitivity | |
|--|--|-----------------------------------|-------------------------------|----------------------------------|-----------------------------------|--------------------|-----------------|--|---|
| Parameter | Field Replicate (Split Sample) | Analytical (Laboratory) Replicate | Matrix Spike Duplicates (MSD) | Laboratory Control Sample (LCS) | Standard Reference Material (SRM) | Matrix Spikes (MS) | Surrogate Spike | Method Blank | Method Detection Limit (MDL) ² or Lowest Concentration of Interest |
| | Relative Percent Difference (RPD) or Relative Standard Deviation (RSD) | | | Recovery Limits (%) ¹ | | | | Comparison of analyte concentration in blank to quantification limit | Concentration Units |
| Total organic carbon | RPD ≤ 20% | RSD ≤ 20% | RPD ≤ 20% | Reference material serves as LCS | 70 – 130% | NA | NA | Analyte concentration <Reporting Limit (RL) | 0.1% dry wt |
| Polycyclic aromatic hydrocarbons (PAHs) | RPD ≤ 40% | RPD ≤ 40% | RPD ≤ 40% | 50 – 150% | See detail in Table 9a | 50 – 150% | 20 – 200% | Analyte concentration <MDL; if > MDL, lowest analyte conc'n. must be >5x method blank conc'n. or qualified as an estimate. | 0.07-0.94 µg/kg dry wt |
| Polychlorinated biphenols (PCBs) – Congeners (high resolution) | Not requested | Not requested | Not requested | Compound specific | Not requested | Not requested | Not requested | | 0.16-3.39 µg/kg dry wt |

NA = not applicable

¹ Recovery limits are based on the low and high confidence limits for each analyte.

² Method Detection Limit is compound specific. See Appendix E-1 in Dutch et al., 2018.

³ PFAS RLs are considered default limits at this point as MEL has not done enough samples to develop in-house limits.

Table 9a. Standard (Certified) Reference Material (NIST 1944) recovery limits of PAHs – MEL.

| Analyte | SRM Limits (%) |
|------------------------|----------------|
| Benz[a]anthracene | 52-96 |
| Benzo(a)pyrene | 50-106 |
| Benzo(b)fluoranthene | 58-111 |
| Benzo(ghi)perylene | 71-127 |
| Benzo(k)fluoranthene | 47-220 |
| Benzo[e]pyrene | 68-123 |
| Chrysene | 61-149 |
| Dibenzo(a,h)anthracene | 110-265 |
| Fluoranthene | 44-95 |
| Indeno(1,2,3-cd)pyrene | 52-140 |
| Perylene | 18-127 |
| Phenanthrene | 60-122 |
| Pyrene | 44-98 |

7.0 Study Design

7.2 Field data collection

7.2.1 *Sampling locations and frequency*

Puget Sound Sediment Monitoring Program

Target coordinates for the 50 Long-Term stations are provided in Figure 1 and Table 10. Alternate coordinates remain as indicated in Dutch et al. (2018), and are provided in Figure 2 and Table 11.

In 2018, sediment could not be collected with the van Veen grab sampler at the target coordinates for Long-Term station 40025 in West Sound. Rocks and shell debris prevented full closure of the grab. As per protocol, the target coordinates were moved 100 meters seaward, where a sample was successfully collected. In addition, due to challenging sampling logistics, water column particulates will no longer be sampled at station 13 in North Hood Canal. They will instead be sampled at station 40022 in the Bainbridge Basin. These changes are reflected in Figure 1 and Table 10.

In 2019, the Urban Bays sampling rotation falls in the Port Gardner/Everett Harbor sampling frame. Target coordinates for these 30 stations and alternate locations remain as indicated in Dutch et al. (2018), and are provided in Figures 3 and 4 and in Tables 12 and 13.

PPCPs and PFASs in Urban Bays

PPCPs and PFASs will be measured in sediments from 30 Budd Inlet target and/or alternate locations provided in Figures 5 and 6 and in Tables 14 and 15. PFASs will also be measured in the Port Gardner/Everett Harbor monitoring stations or alternate locations (Figures 3 and 4, Tables 12 and 13).

Cornwall Avenue Landfill

Ecology's TCP personnel have requested sediment collection from three intertidal stations adjacent to the Cornwall Avenue Landfill in Bellingham, Washington. Coordinates for these stations and a map of locations are provided in Figure 7 and Table 16.

Invertebrate Genomics Initiative

A replicate grab sample of benthic invertebrates will be collected at up to 2 of the Long-Term monitoring stations each day during the April 2019 sampling event (Figure 1, Table 10). Up to 30 stations will be sampled for DNA barcoding of benthos tissue.

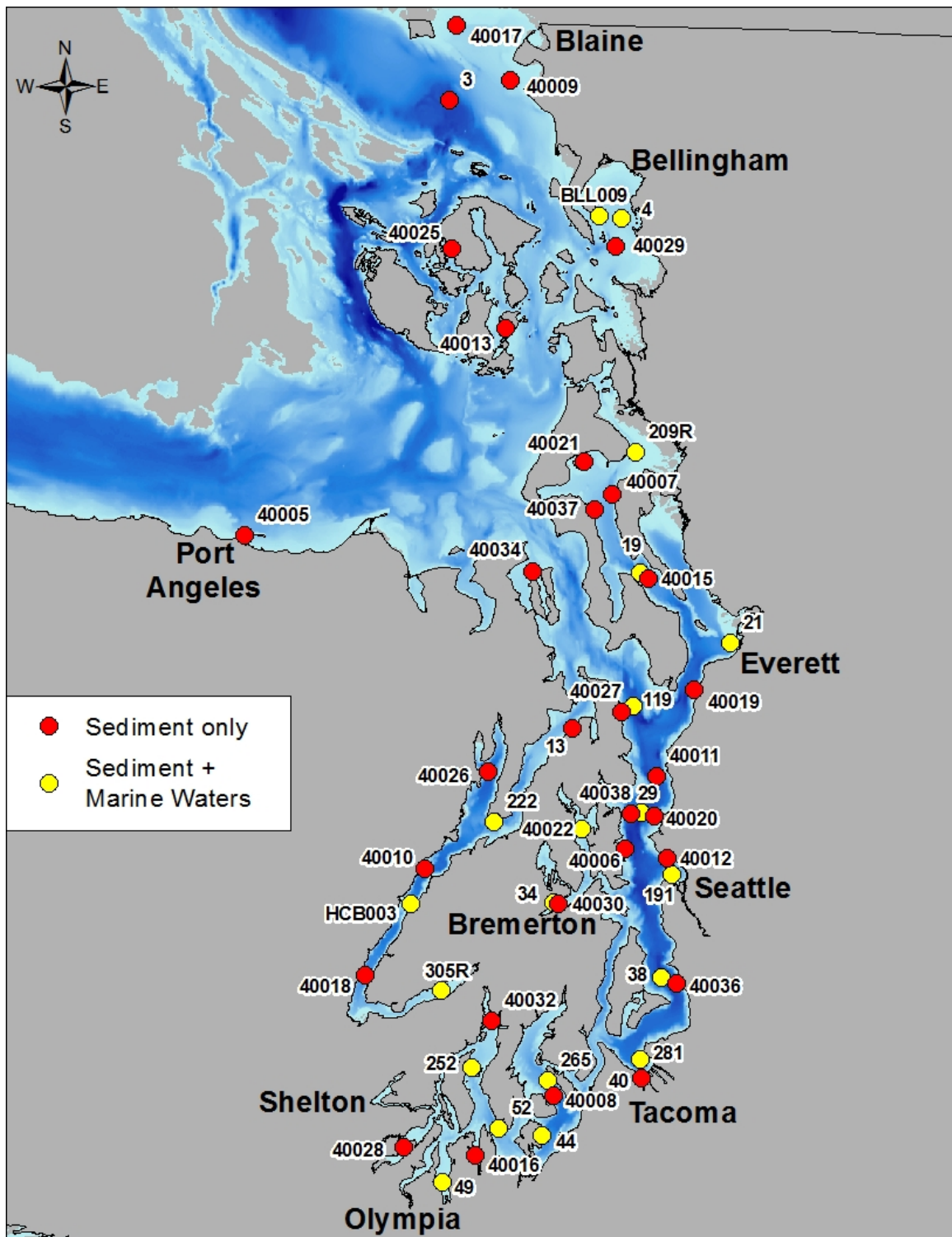


Figure 1. Long-Term monitoring station locations, including co-occurrence with Ecology Marine Waters stations.

Table 10. Target coordinates for 50 Long-Term monitoring stations.

“Station Type” includes: LT = 10 original Long-Term stations, MW = 21 co-located sediment and Marine Waters stations, R = 28 randomly selected stations.

| Station | Location | Target (NAD 83, decimal degrees) | | Station Type |
|---------|--|-------------------------------------|------------|-----------------|
| | | Latitude | Longitude | |
| 3 | Strait of Georgia, N of Patos Island | 48.87025 | -122.97842 | LT |
| 4 | Bellingham Bay | 48.68397 | -122.53820 | LT, MW |
| 13 | North Hood Canal, S of Bridge | 47.83758 | -122.62895 | LT |
| 19 | Saratoga Passage | 48.09792 | -122.47134 | MW |
| 21 | Port Gardner/Everett Harbor | 47.98547 | -122.24283 | LT, MW |
| 29 | Shilshole | 47.70075 | -122.45403 | LT, MW |
| 34 | Sinclair Inlet | 47.54708 | -122.66208 | LT, MW |
| 38 | Point Pully (3-Tree Point) | 47.42833 | -122.39363 | LT, MW |
| 40 | Thea Foss Waterway | 47.26130 | -122.43730 | LT, MW |
| 44 | East Anderson Island | 47.16133 | -122.67358 | LT, MW |
| 49 | Inner Budd Inlet | 47.07997 | -122.91347 | LT, MW |
| 52 | W of Devils Head, E end Nisqually Reach | 47.17060 | -122.78051 | MW |
| 119 | Admiralty Inlet, south | 47.87616 | -122.47816 | MW |
| 191 | Central Elliott Bay | 47.59842 | -122.37581 | MW |
| 209R | Skagit Bay | 48.29533 | -122.48850 | MW |
| 222 | Hood Canal, N of Seabeck | 47.67821 | -122.81466 | MW |
| 252 | Case Inlet | 47.26957 | -122.85101 | MW |
| 265 | Carr Inlet | 47.25240 | -122.66572 | MW |
| 281 | Commencement Bay | 47.29229 | -122.44193 | MW |
| 305R | Lynch Cove | 47.39717 | -122.93124 | MW |
| BLL009 | Bellingham Bay, Pt. Frances (Portage Is.) | 48.68593 | -122.59420 | MW |
| HCB003 | Hood Canal, Central | 47.53787 | -123.00960 | MW |
| 40005 | Inner Port Angeles Harbor | 48.13872 | -123.44985 | R |
| 40006 | Murden Cove | 47.63971 | -122.49046 | R |
| 40007 | Saratoga Passage, north, Camano Island | 48.22609 | -122.54375 | R |
| 40008 | Carr Inlet, NE of Gertrude Island | 47.22686 | -122.64787 | R |
| 40009 | Strait of Georgia, outer Birch Bay | 48.90625 | -122.82638 | R |
| 40010 | Central Hood Canal, S of Triton Cove | 47.59743 | -122.97830 | R |
| 40011 | Central Basin, N of Shilshole | 47.76108 | -122.41759 | R |
| 40012 | Elliott Bay, Smith Cove | 47.62590 | -122.38563 | R |
| 40013 | Reads Bay | 48.49626 | -122.82139 | R |
| 40015 | Saratoga Passage, South | 48.08877 | -122.44853 | R |
| 40016 | Henderson Inlet | 47.12549 | -122.83635 | R |
| 40017 | Boundary Bay | 48.99473 | -122.96789 | R |
| 40018 | Hood Canal, Hoodspout | 47.41787 | -123.11736 | R |
| 40019 | South Possession Sound | 47.90607 | -122.33076 | R |
| 40020 | Shilshole Bay | 47.69588 | -122.42252 | R |
| 40021 | Crescent Harbor | 48.27948 | -122.61517 | R |
| 40022 | Brownsville | 47.67154 | -122.59952 | R, MW |
| 40025 | West Sound (<i>station moved 100m seaward</i>) | 48.62446 | -122.96331 | R |
| 40026 | Dabob Bay | 47.76217 | -122.83153 | R |
| 40027 | Admiralty Inlet, N of Rose Point | 47.86624 | -122.50820 | R |
| 40028 | Totten Inlet | 47.13600 | -123.01006 | R |

| Station | Location | Target (NAD 83, decimal degrees) | | Station Type |
|---------|--|-------------------------------------|------------|-----------------|
| | | Latitude | Longitude | |
| 40029 | North Samish Bay | 48.63718 | -122.55226 | R |
| 40030 | Sinclair Inlet | 47.54500 | -122.65102 | R |
| 40032 | Inner Case Inlet, Rocky Bay | 47.34949 | -122.80550 | R |
| 40034 | Port Townsend, mouth of Kilisut Harbor | 48.09479 | -122.73513 | R |
| 40036 | Des Moines | 47.41975 | -122.35733 | R |
| 40037 | Saratoga Passage, Race Lagoon | 48.19991 | -122.58646 | R |
| 40038 | North Central Basin | 47.69895 | -122.47829 | R |

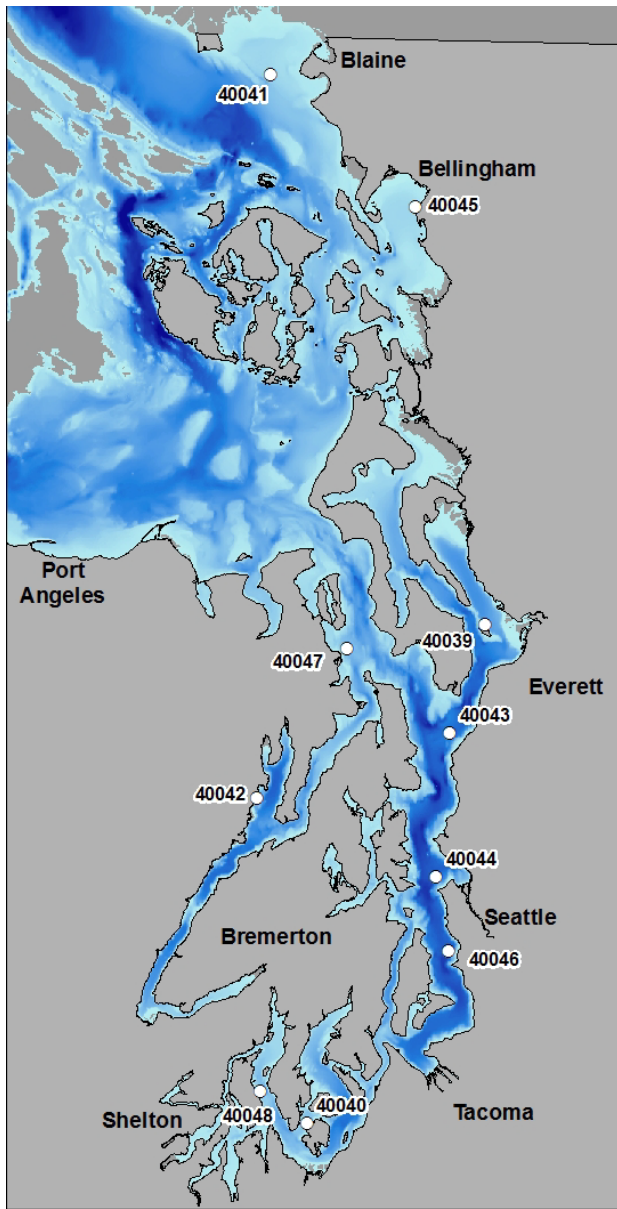


Figure 2. Locations for 10 alternate Long-Term monitoring stations.

Table 11. Alternate coordinates for 10 alternate Long-Term monitoring stations.

| Station | Station location | Target (NAD 83, decimal degrees) | |
|---------|---------------------------------------|-------------------------------------|------------|
| | | Latitude | Longitude |
| 40039 | Gedney Island | 48.02425 | -122.31831 |
| 40040 | NW Anderson Island, Drayton Passage | 47.17831 | -122.72910 |
| 40041 | South Boundary Bay | 48.93582 | -122.89714 |
| 40042 | Hood Canal, Right Smart Cove | 47.72126 | -122.87476 |
| 40043 | South Possession Sound | 47.83918 | -122.39813 |
| 40044 | Central Basin, north of Alki | 47.59770 | -122.42488 |
| 40045 | Bellingham Bay, Fairhaven | 48.72049 | -122.51920 |
| 40046 | Central Basin, north of Normandy Park | 47.47329 | -122.38814 |
| 40047 | Admiralty Inlet, Outer Oak Bay | 47.97690 | -122.66036 |
| 40048 | Case Inlet | 47.23001 | -122.84642 |

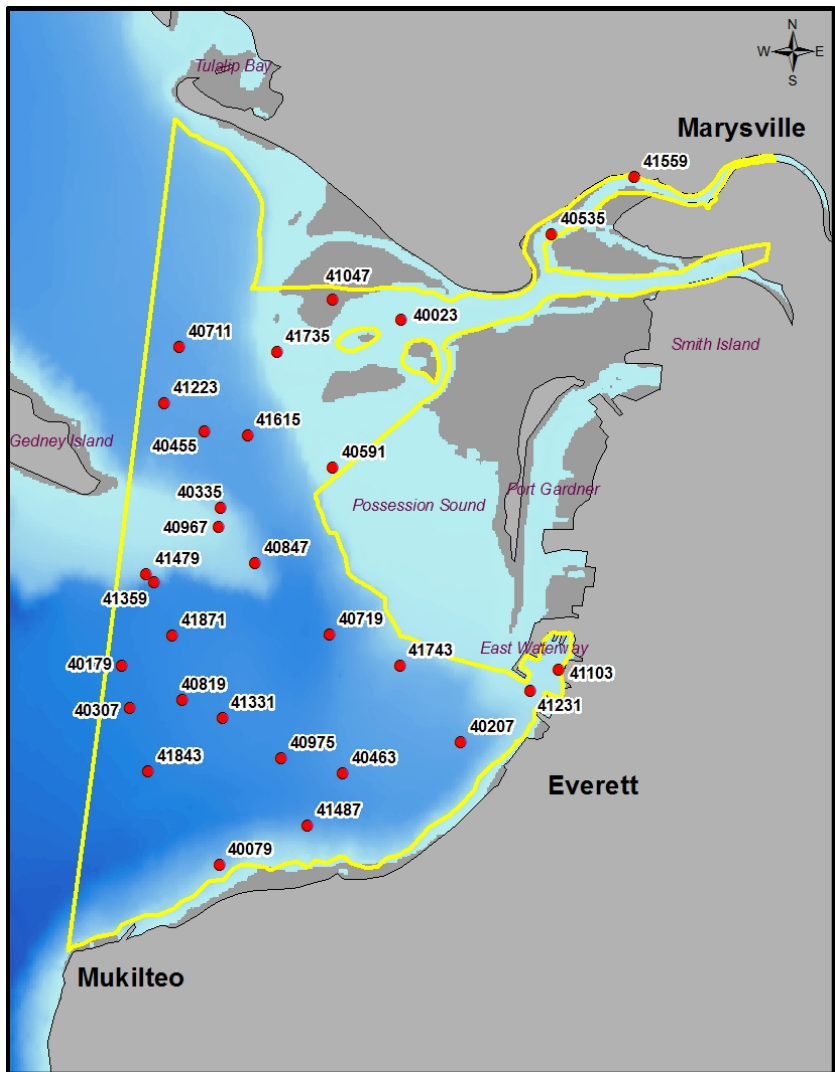


Figure 3. Port Gardner/Everett Harbor sampling frame and 30 monitoring station locations.

Table 12. Target coordinates for 30 Port Gardner/Everett Harbor monitoring stations.
All stations are equally weighted, each representing 1.27 km² of the total 38.1 km² area.

| Station | Target (NAD 83, decimal degrees) | |
|---------|-------------------------------------|------------|
| | Latitude | Longitude |
| 40023 | 48.02659 | -122.24986 |
| 40079 | 47.95991 | -122.28059 |
| 40179 | 47.98380 | -122.29893 |
| 40207 | 47.97551 | -122.23749 |
| 40307 | 47.97868 | -122.29727 |
| 40335 | 48.00329 | -122.28179 |
| 40455 | 48.01256 | -122.28495 |
| 40463 | 47.97142 | -122.25867 |
| 40535 | 48.03742 | -122.22310 |
| 40591 | 48.00846 | -122.26178 |
| 40711 | 48.02266 | -122.28990 |
| 40719 | 47.98817 | -122.26163 |
| 40819 | 47.97988 | -122.28787 |
| 40847 | 47.99670 | -122.27528 |
| 40967 | 48.00091 | -122.28202 |
| 40975 | 47.97300 | -122.26977 |
| 41047 | 48.02892 | -122.26236 |
| 41103 | 47.98450 | -122.22021 |
| 41223 | 48.01578 | -122.29239 |
| 41231 | 47.98299 | -122.22734 |
| 41331 | 47.97773 | -122.28064 |
| 41359 | 47.99404 | -122.29348 |
| 41479 | 47.99496 | -122.29489 |
| 41487 | 47.96496 | -122.26487 |
| 41559 | 48.04457 | -122.20839 |
| 41615 | 48.01214 | -122.27724 |
| 41735 | 48.02243 | -122.27231 |
| 41743 | 47.98462 | -122.24882 |
| 41843 | 47.97108 | -122.29379 |
| 41871 | 47.98766 | -122.28994 |

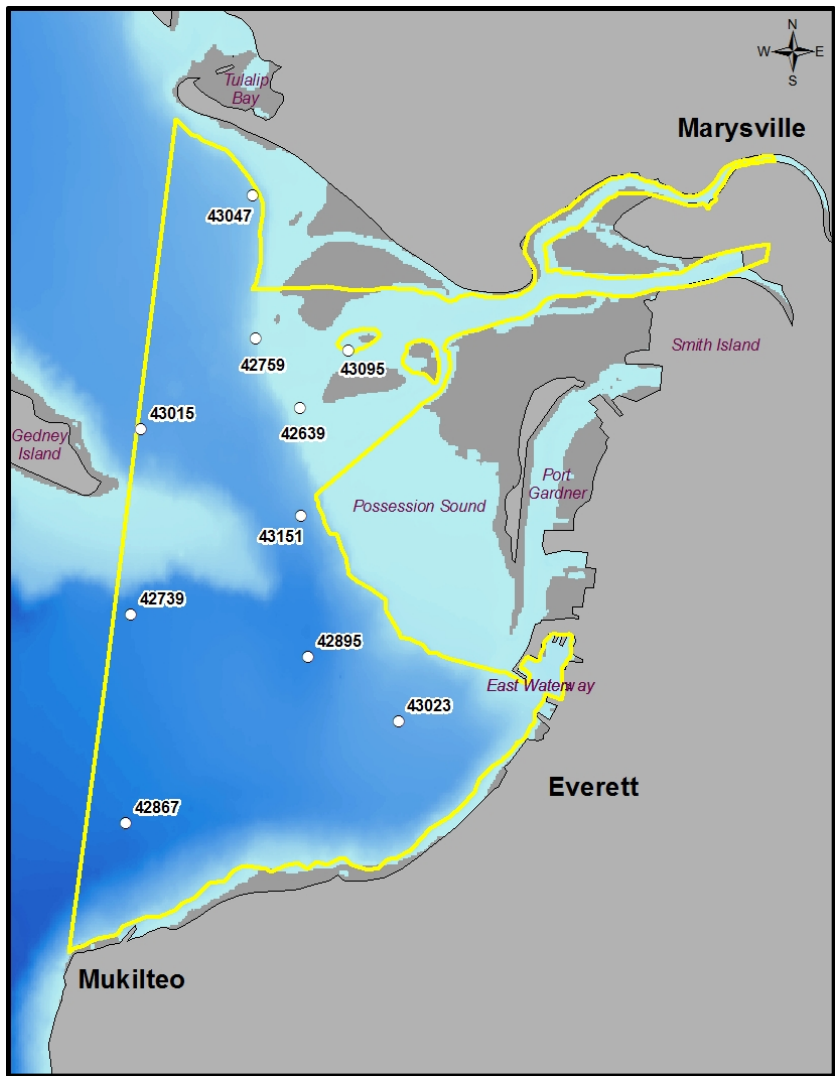


Figure 4. Port Gardner/Everett Harbor sampling frame and 10 alternate monitoring station locations.

Table 13. Target coordinates for 10 Port Gardner/Everett Harbor alternate monitoring stations.

| Station | Target (NAD 83, decimal degrees) | |
|---------|-------------------------------------|------------|
| | Latitude | Longitude |
| 41880 | 47.07653 | -122.92005 |
| 42008 | 47.04648 | -122.90604 |
| 42064 | 47.10261 | -122.90759 |
| 42264 | 47.10007 | -122.92464 |
| 42320 | 47.09653 | -122.90369 |
| 42576 | 47.12388 | -122.90569 |
| 42704 | 47.12982 | -122.91889 |
| 42776 | 47.08882 | -122.92458 |
| 42904 | 47.06903 | -122.91651 |
| 43032 | 47.07466 | -122.91610 |

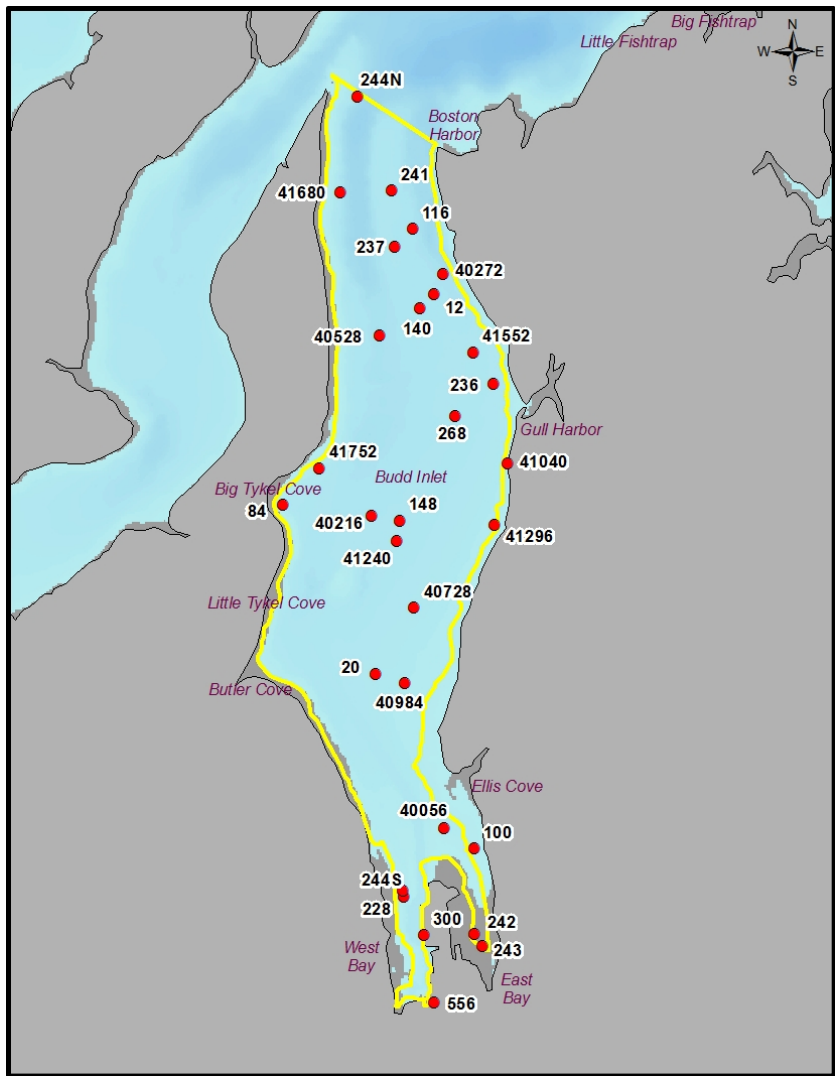


Figure 5. Budd Inlet sampling frame and 30 monitoring station locations.

Table 14. Target coordinates for 30 Budd Inlet monitoring stations.

All stations are equally weighted, each representing 0.578 km² of the total 17.35 km² area.

| Station | Target (NAD 83, decimal degrees) | |
|---------|-------------------------------------|------------|
| | Latitude | Longitude |
| 12 | 47.12407 | -122.90705 |
| 20 | 47.08154 | -122.91473 |
| 84 | 47.10008 | -122.93065 |
| 100 | 47.06241 | -122.89778 |
| 116 | 47.13127 | -122.91092 |
| 140 | 47.12242 | -122.90933 |
| 148 | 47.09875 | -122.91161 |
| 228 | 47.0568 | -122.90899 |
| 236 | 47.11424 | -122.89695 |
| 237 | 47.12927 | -122.91379 |
| 241 | 47.13547 | -122.91450 |
| 242 | 47.05286 | -122.89736 |
| 243 | 47.05164 | -122.89589 |
| 244N | 47.14588 | -122.92064 |
| 244S | 47.05751 | -122.90913 |
| 268 | 47.1106 | -122.90308 |
| 300 | 47.05261 | -122.90552 |
| 556 | 47.04513 | -122.90357 |
| 40056 | 47.06458 | -122.90270 |
| 40216 | 47.09917 | -122.91611 |
| 40272 | 47.12633 | -122.90571 |
| 40528 | 47.11928 | -122.91573 |
| 40728 | 47.08906 | -122.90877 |
| 40984 | 47.08067 | -122.90988 |
| 41040 | 47.10551 | -122.89420 |
| 41240 | 47.0964 | -122.91197 |
| 41296 | 47.09853 | -122.89604 |
| 41552 | 47.11775 | -122.90043 |
| 41680 | 47.13508 | -122.92285 |
| 41752 | 47.10428 | -122.92496 |

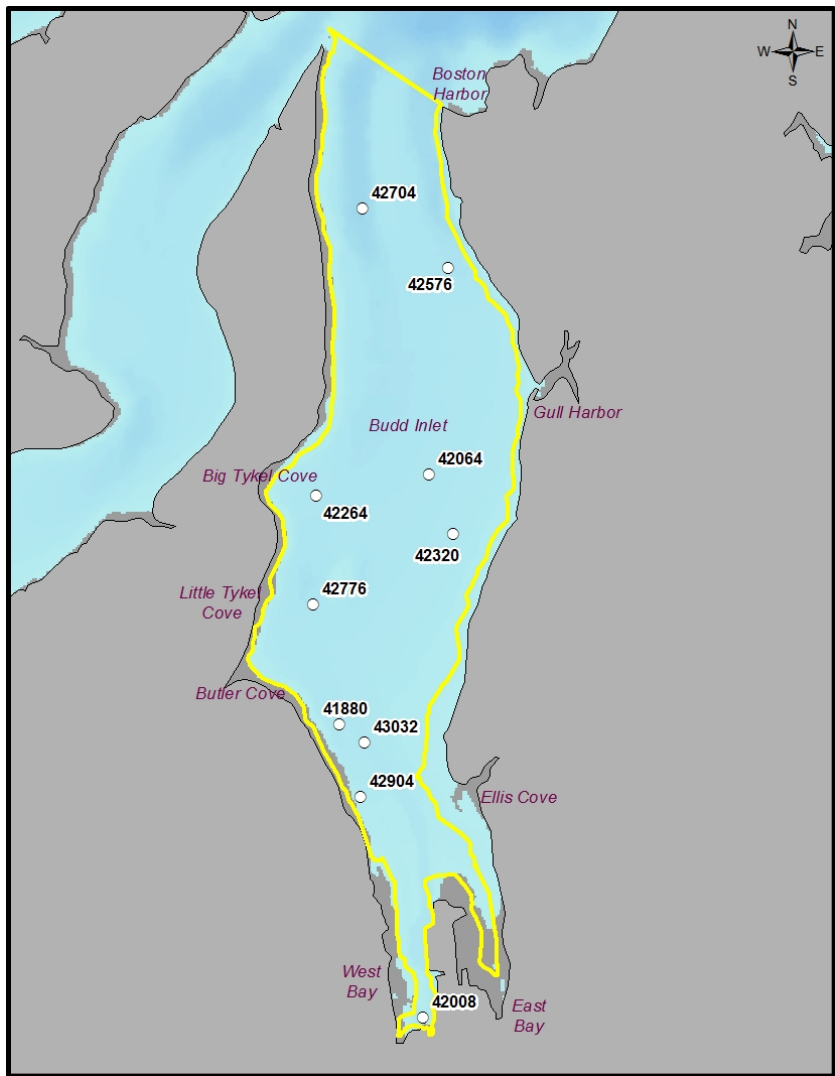


Figure 6. Budd Inlet sampling frame and 10 alternate monitoring station locations.

Table 15. Alternate coordinates for 10 Budd Inlet alternate monitoring stations.

| Station | Target (NAD 83, decimal degrees) | |
|---------|--|------------|
| | Latitude | Longitude |
| 41880 | 47.07653 | -122.92005 |
| 42008 | 47.04648 | -122.90604 |
| 42064 | 47.10261 | -122.90759 |
| 42264 | 47.10007 | -122.92464 |
| 42320 | 47.09653 | -122.90369 |
| 42576 | 47.12388 | -122.90569 |
| 42704 | 47.12982 | -122.91889 |
| 42776 | 47.08882 | -122.92458 |
| 42904 | 47.06903 | -122.91651 |
| 43032 | 47.07466 | -122.91610 |

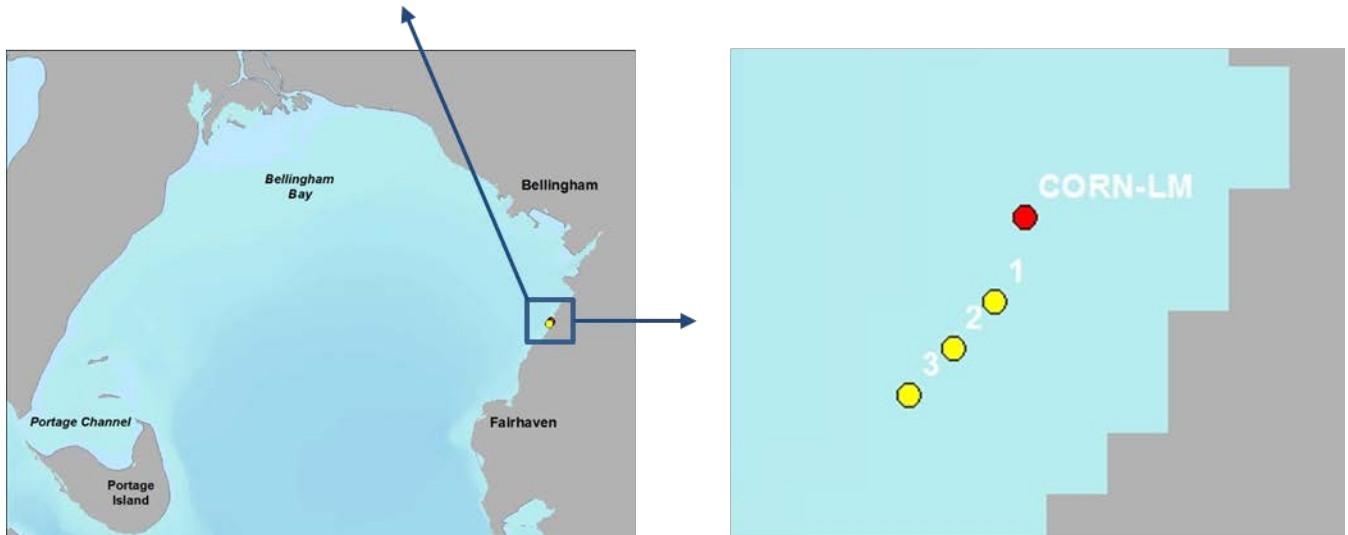


Figure 7. Three intertidal sampling stations adjacent to the Cornwall Avenue Landfill. (CORN-LM is a landmark location that will be used to help relocate sites 1-3)

Table 16. Target coordinates for three intertidal sampling stations adjacent to the Cornwall Avenue Landfill.

| Station | Target (NAD 83, decimal degrees) | |
|---------|-------------------------------------|------------|
| | Latitude | Longitude |
| CORN-LM | 48.73733 | -122.49567 |
| 1 | 48.73708 | -122.49580 |
| 2 | 48.73694 | -122.49598 |
| 3 | 48.73680 | -122.49617 |

7.2.2 Field parameters and laboratory analytes to be measured

Puget Sound Sediment Monitoring Program

For Long-Term and Urban Bays monitoring, all benthos samples and measurements, and all sediment sample field measurements, physical, and biogeochemical parameters will be collected as listed in Table 25 in Dutch et al., 2018. Sediment chemistry parameters will be measured at all 30 Urban Bays stations but at only 10 of the 50 Long-Term stations, including stations 21, 34, 40, 40013, 40015, 4016, 40017, 40018, 40019, and 40020.

PPCPs and PFASs in Urban Bays

The suites of 119 PPCPs and 24 PFASs measured in sediments from the Port Gardner/Everett Harbor and Budd Inlet sampling frames are listed below by method type.

PPCP Lists 1 - 5

| List 1 - Acid Extraction in Positive Ionization | |
|--|-----------------------|
| Acetaminophen | Miconazole |
| Ampicillin 1 | Norfloxacin |
| Azithromycin | Norgestimate |
| Caffeine | Ofloxacin |
| Carbadox | Ormetoprim |
| Carbamazepine | Oxacillin |
| Cefotaxime | Oxolinic acid |
| Ciprofloxacin | Penicillin G |
| Clarithromycin | Penicillin V |
| Clinafloxacin | Roxithromycin |
| Cloxacillin | Sarafloxacin |
| Dehydronifedipine | Sulfachloropyridazine |
| Digoxigenin | Sulfadiazine |
| Digoxin | Sulfadimethoxine |
| Diltiazem | Sulfamerazine |
| 1,7-Dimethylxanthine | Sulfamethazine |
| Diphenhydramine | Sulfamethizole |
| Enrofloxacin | Sulfamethoxazole |
| Erythromycin-H2O | Sulfanilamide |
| Flumequine | Sulfathiazole |
| Fluoxetine | Thiabendazole |
| Lincomycin | Trimethoprim |
| Lomefloxacin | 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
Benzoylcegonine
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

Twenty-four PFASs to be measured by MEL

Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorobutanesulfonate (PFBS)
Perfluorohexanoate (PFHxA)
Perfluoropentanesulfonate (PFPeS)
Perfluoroheptanoate (PFHpA)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanoate (PFOA)
Perfluoroheptanesulfonate (PFHpS)
Perfluorononanoate (PFNA)
Perfluorooctanesulfonate (PFOS)
Perfluorononanesulfonate (PFNS)
Perfluorodecanoate (PFDA)

N-methyl perfluorooctanesulfonamidoacetate (N-MeFOSAA)
Perfluorodecanesulfonate (PFDS)
Perfluoroundecanoate (PFUnA)
N-ethyl perfluorooctanesulfonamidoacetate (N-EtFOSAA)
Perfluorododecanoate (PFDoA)
Perfluorododecanesulfonate (PFDoS)
Perfluorotridecanoate (PFTrDA)
Perfluorotetradecanoate (PFTeDA)
Perfluorohexadecanoate (PFHxDA)
Perfluorooctadecanoate (PFODA)
Perfluorooctanesulfonamide (PFOSA)

Cornwall Avenue Landfill

Parameters measured in intertidal sediments sampled near the Cornwall Avenue Landfill will include those listed in Table 17.

Table 17. Parameters measured in sediments for the Cornwall Avenue Landfill.

| | | |
|--|--------------------------|---|
| <i>Biogeochemistry</i> | Anthracene | Chrysene |
| Total organic carbon (PSEP, 1986) | Biphenyl | Dibenzo(a,h)anthracene |
| | Dibenzothiophene | Fluoranthene |
| | Fluorene | Indeno(1,2,3-c,d)pyrene |
| <i>Organics</i> | Naphthalene | Perylene |
| Polynuclear Aromatic Hydrocarbons | Phenanthrene | Pyrene |
| <i>LPAHs</i> | Retene | Total HPAH ⁺ |
| 1,6,7-Trimethylnaphthalene | Total LPAHs ⁺ | Total Benzofluoranthenes ⁺ |
| 1-Methylnaphthalene | <i>HPAHs</i> | <i>Polychlorinated Biphenyls</i> |
| 1-Methylphenanthrene | Benzo(a)anthracene | PCB congeners 001-209 - |
| 2,6-Dimethylnaphthalene | Benzo(a)pyrene | see Table F-3 in TCP, 2015 |
| 2-Methylnaphthalene | Benzo(b)fluoranthene | (<i>high resolution analyses</i>) |
| 2-Methylphenanthrene | Benzo(e)pyrene | ⁺ <i>calculated values</i> |
| Acenaphthene | Benzo(g,h,i)perylene | |
| Acenaphthylene | Benzo(k)fluoranthene | |

Invertebrate Genomics Initiative

All benthos collected for the Invertebrate Genomics Initiative will be identified to the lowest taxonomic level possible by Ecology taxonomists. DNA barcode sequencing will be conducted for all benthos tissue collected by personnel at the Smithsonian.

8.0 Field Procedures

8.2 Measurement and sampling procedures

Sampling platform and station positioning

Puget Sound Sediment Monitoring Program

Sampling platform and station positioning details for the Long-Term and Urban Bays sampling will be as described in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

Sampling platform and station positioning details for PPCPs and PFASs analysis will be those used for Urban Bays as described in Dutch et al., 2018.

Cornwall Avenue Landfill

Intertidal stations at the Cornwall Avenue Landfill will be located by walking to the beach at low tide and relocating three sets of station coordinates previously selected by Ecology's TCP site manager (Figure 7, Table 16). Stations will be relocated using measured distances from fixed landmarks and a hand-held GPS unit.

Invertebrate Genomics Initiative

Sampling platform and station positioning details will be as described for the Long-Term program in Dutch et al., 2018.

Sampling collection

Puget Sound Sediment Monitoring Program

Procedures for Long-Term and Urban Bays sample collection will be as described in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

Procedures for sample collection and preservation of sediment samples for analyses of PPCPs and PFASs are provided in Table 18.

Table 18. Sample collection and preservation for analyses for PPCPs and PFASs in homogenized sediment.

| Parameter | Size of Sediment Sample | Container | Preservation | Maximum Holding Time* |
|---|-------------------------|--|---|--|
| 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 field freezer immediately after collection. 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 MEL | Freeze | 1 year |

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

Cornwall Avenue Landfill

At the Cornwall Avenue Landfill, a 0.1-m² quadrat will be positioned on the beach at each set of station coordinates, the quadrat will be photographed, and all soft sediment within the quadrat will be removed to a depth of 10-12 cm. These sediments will be homogenized in a decontaminated stainless-steel mixing bucket and samples spooned into appropriate sampling jars (see Table 27 in Dutch et al., 2018). Excess sediment will be returned to the site from which it was collected. All remaining sampling procedures will follow those in Dutch et al., 2018.

Invertebrate Genomics Initiative

A benthos sample will be collected for DNA barcoding from the first station of the day on each day of the Long-Term monitoring cruise. If taxa abundance appears to be low in this sample, another sample may also be collected at the second station of the day, at the discretion of the sampling crew leader. During this 15-day cruise, benthos will be collected from up to 30 stations.

Benthos will be collected with a 0.1-m² double van Veen grab sampler and sieved through a 1-mm mesh screen. All organisms and sediment residue retained on the screen will then be transferred to chemically-clean (i.e., no exposure to formalin), high-density polyethylene leak-proof jars. Large rocks, shells, and woody debris from the sample will be placed in containers separate from fragile organisms so as to not damage the organisms. Fragile organisms may also be picked directly from the screen and placed in separate containers. All known carnivorous organisms and burrowing sea anemones will be placed in separate containers. When sieving is complete, each jar will be partially filled with ambient seawater, labelled following Dutch et al., 2018, and placed in a chemically-clean ice chest filled with ice. No fixatives (i.e., formalin) or preservatives (i.e., ethanol) will be added to these live benthos samples. A 5-gallon carboy of ambient seawater will be collected each morning for use in sample sorting later in the day.

After the benthos samples have been collected, and around midpoint during the sampling day, the research vessel will rendezvous with a sample courier at a prearranged meeting location (marina or boat launch). The benthos samples and carboy of ambient seawater will be transferred to the courier. The courier will drive the samples to either the Ecology Operations Center in Lacey, or to Ecology's Padilla Bay National Estuarine Research Reserve in Mt. Vernon. Ecology staff will receive the samples within hours of their collection and will prepare them for DNA barcoding processing and archiving (see Laboratory Procedures, below and also standard operating procedures in the Appendix).

Sample labeling, chain-of-custody, and field log requirements will be as outlined in Dutch et al., 2018. After processing, remaining seawater will be decanted and washed down the laboratory sink drain, while the sample residue will be transferred to 5-gallon buckets with disposal to the municipal landfill.

9.0 Laboratory Procedures

Puget Sound Sediment Monitoring Program

Laboratory procedures for Long-Term and Urban Bays sample analysis will be as described in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

Laboratory procedures for PPCPs and PFASs analysis are outlined in Table 19.

Table 19. Laboratory analysis and reporting requirements for PPCPs and PFASs in sediments.

| Parameter | Expected Range of Results | Extraction Method | Clean-up Method | Analysis Method | Technique/Instrument | Required Reporting Limit |
|-----------|---------------------------|---|---|-----------------|--|---|
| PPCPs | Unknown | 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. | 1-1,000 µg/kg dry weight |
| PFASs | Unknown | QuEChERS extraction with acetonitrile/ ammonium hydroxide solution (pH9). | Agilent Enhanced Matrix Removal cleanup protocol. Then diluted with ultra-pure LCMS water for analysis. | USEPA 8321B | HPLC/ESI-MS/MS. High performance liquid chromatography with triple quadrupole mass spectrometer in negative electrospray ionization mode using isotopic dilution quantitation. | 0.5 µg/kg dry weight (0.25 ug/Kg wet weight) If the % solids is > 50%, the RL will be > 0.5 ug/Kg. The MEL 100% dry weight RL is 0.25 ug/Kg. |

Cornwall Avenue Landfill

Methods for the Cornwall Avenue Landfill sample analyses are outlined in Table 20. TOC and PAH analyses will be conducted at MEL. High-resolution PCB congener analysis will be conducted by a contract laboratory.

Table 20. Lab procedures for the Cornwall Avenue Landfill project.

| Parameter | Expected Range of Results | Preparation Method | Analysis Method | Technique / Instrument | Practical Quantitation Limit |
|---|---------------------------|---|------------------------------------|---|--|
| Total organic carbon (TOC) | 0.01-15.0% | Drying sediment material | PSEP, 1986 | Drying sediment material, pretreatment and subsequent oxidation of the dried sediment, and determination of CO ₂ by infrared spectroscopy. | 0.1% |
| Polycyclic aromatic hydrocarbons (PAHs) | 0.01 – 50,000 ppb | USEPA 3541 (Me) (Soxtherm with methylene chloride extraction), EPA 3630C (clean-up) | USEPA 8270E with isotopic dilution | MEL modification with capillary GC/MS-SIM isotopic dilution analysis | 0.5-2.0 µg/kg dry weight |
| PCB Congeners (High Resolution) | 0.50-900 ng/kg dry weight | USEPA 1668C | USEPA 1668C | High Resolution Gas Chromatography/High Resolution Mass Spectrophotometry (HRGC/HRMS) | 0.4 ng/kg dry weight (exception: PCB 156/157 = 0.8 ng/kg dry weight) |

Invertebrate Genomics Initiative

Benthos samples will be delivered by land courier to a three-member lab crew by early afternoon on each day of the Long-Term monitoring cruise. Once received, benthos will be sorted and will undergo the following procedures:

- Sorting and morphotyping into various taxonomic groupings.
- Identification to lowest possible level.
- Relaxation (anesthetic added to the seawater).
- Photographing of whole specimens and morphological features.
- Tellinid and ampeliscid specimens preserved for Cosmopolitan Species project.
- Collection and preservation of tissue sample for genetic sequencing for the DNA Barcode Library project.
- Fixation of voucher specimens for long-term archiving.
- Database entry for all field and laboratory information related to each specimen.

Detailed methods are documented in the Appendix.

9.4 Laboratories accredited for methods

Ecology's Manchester Environmental Laboratory (MEL) and contract laboratories will be accredited by Ecology for all 2019 Sediment Program analyses except:

- Stable isotopes of carbon (C^{13}) and nitrogen (N^{15})
- Biogenic silica
- PFAS compounds

Ecology's QA Officer and Environmental Assessment Program (EAP) Manager recently approved three separate waivers from Ecology's accreditation policy:

- C^{13} and N^{15} , accreditation waiver approved for University of California Santa Barbara SIMS Light Stable Isotope Lab, March 19, 2018.
- Biogenic silica, accreditation waiver approved for University of British Columbia's Department of Earth, Ocean, and Atmospheric Sciences Lab, March 19, 2018.
- PFAS in sediment, accreditation waiver approved for MEL, March 2019.

10.0 Quality Control Procedures

Laboratory Analyses

Chemistry

Puget Sound Sediment Monitoring Program

Quality control (QC) procedures for Long-Term and Urban Bays sampling will be as described in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

Analyses conducted by MEL or contract laboratory will adhere to analytical QC methods outlined in published protocols (Table 19) and in each laboratory’s in-house standard operating procedures. The frequency and type of each chemistry QC test is specified in Table 21.

Field duplicate (split) samples – Three will be collected and analyzed for each of 30 PFAS samples from Budd Inlet, 30 PFAS samples from Port Gardner/Everett Harbor, and 30 PPCP samples from Budd Inlet.

Matrix spike and matrix spike duplicate analysis – One of each will be conducted on each of the two sets of 30 PFAS samples and on the 30 PPCP samples.

Cornwall Avenue Landfill

All chemistry analyses conducted by MEL or at contract laboratories will adhere to analytical QC methods outlined in published protocols (Table 20) and in each laboratory’s in-house standard operating procedures. The frequency and type of each chemistry QC test is specified in Table 21.

Table 21. Quality control sample types and frequency for physical and chemistry parameters.

| | Field | Laboratory | | | | | | |
|------------------------------|---------------------------------------|--|-------------------------------|---------------------------------|-----------------------------------|--------------------|---|---------------|
| Quality Control Sample Type: | Field Replicate (Split Sample) | Analytical (Laboratory) Replicate | Matrix Spike Duplicates (MSD) | Laboratory Control Sample (LCS) | Standard Reference Material (SRM) | Matrix Spikes (MS) | Surrogate Spike | Method Blank |
| Measurement Frequency: | Duplicate analysis for 10% of samples | Triplicate analysis/batch of 20 samples for TOC. Duplicate analysis/batch of 20 for organics samples | 1/batch of 30 | 1/batch of 20 | 1/batch of 20 | 1/batch of 30 | Every organics sample, blank, and QC sample | 1/batch of 20 |

Invertebrate Genomics Initiative

Field and lab personnel will be trained in the methods specified in this QAMP addendum for proper sieving, sorting, and processing of benthos and tissue samples collected for the Invertebrate Genomics Initiative (see Appendix). Lead Ecology staff will oversee and strive for consistency in both field and lab operations. Taxonomic identification verification will follow Dutch et al., 2018 and Weakland, 2018.

11.0 Management Procedures

11.1 Data recording and reporting requirements

Puget Sound Sediment Monitoring Program

Procedures for Long-Term and Urban Bays data management will be as described in Dutch et al., 2018.

PPCPs and PFASs in Urban Bays

Procedures for PPCPs and PFASs in Urban Bays data management will be as described in Dutch et al., 2018.

Cornwall Avenue Landfill

Field data for each station will be recorded in a Rite-in-the-Rain notebook and will include:

- Names of field team members.
- Date and time of sample collection.
- Tide height.
- Station coordinates.
- Description of sediment in quadrat.
- All other observations noted by the field crew.

A photograph will be taken of the undisturbed sediment within the quadrat at each station. All logs will be reviewed after each station is sampled to ensure they are complete and correct. All data will be uploaded to Ecology's EIM database where they will be accessible to TCP staff. No other form of reporting will be provided to TCP staff for this project.

Invertebrate Genomics Initiative

All field log and laboratory tracking information generated by Ecology staff will be entered into Excel spreadsheets (see Appendix) archived with the Sediment Program and shared with Dr. Paulay and Smithsonian staff.

All DNA barcoding information generated by the Smithsonian will be shared with Ecology staff and Dr. Paulay and stored in various publically accessible benthos and DNA databases on the world-wide-web (see Appendix).

Sections 12-14 remain the same as described in the QAMP (Dutch et al., 2018).

15.0 References

- Dutch, M., V. Partridge, S. Weakland, D. Burgess, and A. Eagleston. 2018. Quality Assurance Monitoring Plan: The Puget Sound Sediment Monitoring Program. Publication 18-03-109. Washington State Department of Ecology, Olympia.
<https://fortress.wa.gov/ecy/publications/SummaryPages/1803109.html>.
- Martin, R.A., and E.A. Nesbitt. 2015. Foraminiferal evidence of sediment toxicity in anthropogenically influenced embayments of Puget Sound, Washington, U.S.A. *Marine Micropaleontology* 121: 97–106.
- Martin, R.A., and E.A. Nesbitt. 2017. Good news and bad news in two highly industrialized Puget Sound, Washington (U.S.A.) embayments. *Journal of Foraminiferal Research* 47(4): 372–388.
- Nesbitt, E.A., R.A. Martin, D.E. Martin, and J. Apple. 2015. Rapid deterioration of sediment surface habitats in Bellingham Bay, Washington State, as indicated by benthic foraminifera. *Marine Pollution Bulletin* 97(1-2): 273–284.
- PSEMP Marine Waters Workgroup. 2018. Puget Sound marine waters: 2017 overview. S. K. Moore, R. Wold, K. Stark, J. Bos, P. Williams, N. Hamel, S. Kim, A. Brown, C. Krembs, and J. Newton (editors).
www.psp.wa.gov/PSmarinewatersoverview.php.
- Washington State Department of Ecology. 2014. Cleanup Action Plan, Cornwall Avenue Landfill, Bellingham, WA.
<https://fortress.wa.gov/ecy/gsp/DocViewer.ashx?did=41550>.
- Ecology [Washington State Department of Ecology]. 2015. Bellingham Bay Regional Background Sediment Characterization: Final Data Evaluation and Summary Report. Toxics Cleanup Program, Washington State Department of Ecology, Olympia. Publication 15-09-044. <https://fortress.wa.gov/ecy/publications/SummaryPages/1509044.html>.

16.0 Appendix.

Standard Operating Procedures

West Coast Invertebrate Genomics Initiative

An **Overview** and the following 9 sections of this appendix are available electronically at <https://fortress.wa.gov/ecy/publications/SummaryPages/2003101.html>

Ecology protocols

A-1. Burgess, D. and A. Eagleston. 2019. Protocol for Shipping Scientific Specimens.docx

A-2. Weakland, S. 2018a. Standard Operating Procedures for Obtaining Marine Sediment Samples EAP039 v1.3.

A-3. Weakland, S. 2018b. Standard Operating Procedures for Marine Macrobenthic Sample Analysis. EAP043 v1.2.

Dr. Gustov Paulay's protocols

A-4. Paulay Field Template.xlsx

A-5. Paulay Field Methods.docx

A-6. Paulay Photo Processing.docx

A-7. Paulay Specimen Processing.docx

Smithsonian Institution protocols

A-8. SI Invertebrate Zoology Collection Info Spreadsheet.xlsx

A-9. SI Invertebrate Zoology Collection Procedures.pdf