Pharmaceuticals, Personal Care Products, and Perfluoroalkyl Substances in Elliott Bay Sediments: 2013 Data Summary

Abstract

Elliott Bay sediment samples, collected in June 2013 at 30 randomly-selected stations, were tested for the presence and concentrations of pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs), identified worldwide as contaminants of emerging concern (CECs).

A small proportion of all PPCP (4.5%) and PFAS (6.9%) measurements generated for this study were found in detectable concentrations. Thirteen of the 119 PPCPs selected for analyses were detected. Most frequently occurring were triclocarban, an antibacterial agent; diphenhydramine, an antihistamine; and triamterene, a diuretic. Ten additional PPCPs, including antibiotics, calcium-channel blockers, an antidepressant, a stimulant, a bronchodilator, and an opiate, were detected less frequently. Of the 13 PFASs measured, perfluorooctane-sulfonate (PFOS), perfluorodecanoate (PFDA), and perfluoroundecanoate (PFUnA) were detected. Data for all detected chemicals are summarized, and concentrations are displayed spatially on maps.

These data contribute to a growing baseline of information about PPCPs and PFASs in Puget Sound and lay a foundation for better understanding of the relationship between their concentrations in marine waters, biota, and various biotic endpoints and their sources, transport patterns, and fate.

Publication No. 14-03-049 December 2014

Publication Information

This report is available on the Department of Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1403049.html

Data for this project are available at Ecology's Environmental Information Management (EIM) website www.ecy.wa.gov/eim/index.htm. Search Study IDs: PSAMP_SP, UWI2013.

Ecology's Activity Tracker Code for this study is 01-900.

Water Resource Inventory Area (WRIA) and 8-digit Hydrologic Unit Code (HUC) numbers for the study area:

WRIA
- 09

HUC numbers
- 17110013
- 17110019

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This project has been funded wholly or in part by the U.S. Environmental Protection Agency (EPA) under Puget Sound Ecosystem Restoration and Protection Cooperative Agreement Grant PC-00J20101 with Washington Department of Ecology. The contents of this document do not necessarily reflect the views and policies of EPA, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Background

In recent years, a diverse group of relatively unmonitored and unregulated chemicals found in consumer and industrial products has been shown to occur at trace levels in wastewater discharges, ambient receiving waters and sediments, drinking water supplies, and biotic tissue samples collected in and near large estuaries. When discharged to the environment, these "contaminants of emerging concern (CECs)" have the potential to cause adverse toxicological, biological, and ecological effects to both wildlife and humans (Diamond et al., 2011).

Two groups of CECs of interest to scientists working in large estuarine and freshwater systems worldwide, including Puget Sound, are pharmaceuticals and personal care products (PPCPs) and perfluoroalkyl substances (PFASs). PPCPs include thousands of prescription and over-the-counter drugs, nutritional supplements, shampoos and lotions used by humans, as well as drugs administered to livestock. PFASs include chemicals with non-stick, water-repellant, and stain-resistant properties and are used in the manufacture of clothing, carpeting, cookware, and other products. After PPCPs are consumed and as products containing PFASs wear, the chemicals find their way into the aquatic environment through both point-source and nonpoint-source pathways (Kummerer, 2004; Buck et al., 2011).

Over the past decade, PPCPs have been found in measurable concentrations in wastewater treatment plant influent and effluent in Washington (Puget Sound and other water bodies) and Oregon (Hope, 2012; Johnson et al., 2004; Lubliner et al., 2010; Morace, 2012), reclaimed water (Johnson and Marti, 2012), and surface water and groundwater (Dougherty et al., 2010). Biological effects due to exposure to estrogenic and endocrine-disrupting chemicals have been observed in Puget Sound fish (Johnson et al., 2008; Peck et al., 2011; daSilva et al., 2013). PPCPs have also been measured in sediments collected in Bellingham Bay and other Puget Sound locations (Long et al., 2013) and in sediments from the Columbia River and selected tributaries (Nilsen et al., 2007).

Perfluoroalkyl substances have been detected in surface water, fish tissue, and osprey eggs in and around Washington rivers and lakes, five of which drain into Puget Sound (Furl and Meredith, 2010). These chemicals have also been found in sediments collected in Bellingham Bay and other Puget Sound locations (Long et al., 2013).

CEC Monitoring Priority for Puget Sound: PPCPs and PFASs in Urban Bay Sediments

Characterization of sources, transport patterns, and fates of CECs in the Puget Sound ecosystem was recently prioritized in the Puget Sound Action Agenda as part of the toxics monitoring strategy for Puget Sound (Puget Sound Partnership, 2012). As part of this strategy, the Washington State Department of Ecology (Ecology) Marine Sediment Monitoring Team (MSMT) received funding through the US Environmental Protection Agency (USEPA) National Estuary Program (NEP) to measure concentrations of PPCPs and PFASs in Elliott Bay sediments.

The measurement of these chemicals in Elliott Bay sediments in June 2013 was conducted as part of the Puget Sound Ecosystem Monitoring Program (PSEMP) Sediment Component and followed a survey conducted in 2010 which successfully established a baseline data set for these CECs in Bellingham Bay and at 10 long-term sediment monitoring stations throughout Puget Sound (Long et al., 2013). Continued establishment of baseline concentrations of these chemicals in Puget Sound is a first step toward understanding the distribution of these chemicals and their potential impact on humans and aquatic biota, and is necessary for future management and regulation of these chemicals in the Puget Sound ecosystem.

Study Objectives

The objectives of this study of Elliott Bay sediments were to:

- Establish a baseline record of concentrations for 119 PPCPs and 13 PFASs in Elliott Bay sediments.
- Provide high quality sediment CEC data to the Puget Sound ecosystem community of scientists, managers, and regulators.
- Compare PPCP and PFAS concentrations among Puget Sound urban bays.

The third objective, comparison of sediment PPCP and PFAS data from this Elliott Bay survey to data from sediment surveys elsewhere in Puget Sound, will be summarized in a separate report upon completion and review of chemical analyses from 2014.

Methods

Sediments were collected from 30 monitoring stations located throughout Elliott Bay (Figure 1). Stations were selected from a stratified random sampling design. Sampling was conducted in June 2013 as part of Ecology's annual Urban Bay sediment monitoring. A double 0.1-m^2 van Veen grab sampler was deployed multiple times to collect sediments from each site, and the top 2-3 cm of sediment was removed and composited for each sample.

Sample preparation, extraction, instrumental analysis, and quantification procedures were conducted by AXYS Analytical Services Ltd., Sidney, BC, Canada. These procedures measured concentrations of 119 PPCPs and 13 PFASs (Table 1) in these sediments in accordance with AXYS Method MLA-075 (an extension of USEPA 1694) for PPCPs and MLA-041 for PFCs. These methods use both acid and basic extractions, spiking with isotopically labeled surrogates or standards, and analysis and quantification with high performance liquid chromatography-tandem mass spectrometry with positive and negative electrospray ionization in multiple reaction monitoring mode.

To ensure comparability of data, all sampling and analysis methods were consistent with those used in 2010 (Long et al., 2013) for samples from Bellingham Bay and long-term stations, and in 2014 for Commencement Bay. Further details about the collection and analysis of these Elliott Bay sediment samples are described in the Quality Assurance Project Plan (QAPP) for the overall status-and-trends sediment monitoring program (Dutch et al., 2009), two QAPP addenda

(Dutch et al., 2010, 2012), a Project Work Plan (Dutch et al., 2013), and data packages from AXYS Analytical Services, all included in the Electronic Appendices.

Data Reporting and Summaries, Quality Review

PPCPs and PFASs data generated for this 2013 Elliott Bay survey are provided in this report. Raw data are available in the Electronic Data Deliverable spreadsheets provided by AXYS Analytical Services, as amended by Ecology's Manchester Environmental Laboratory (MEL) quality assurance officer, Karin Feddersen. Karin examined all raw data for qualitative and quantitative precision and bias. She reviewed quality control conditions flagged by AXYS that may affect the data and added qualifiers, as appropriate, that were consistent with MEL and Ecology Information Management (EIM) guidelines. Her review reports are also included in the Electronic Appendices. These amended data are presented in an Excel spreadsheet in the Electronic Appendices and also have been uploaded to Ecology's EIM database.

Tables and figures summarizing the concentrations and spatial distributions of detected PPCPs and PFASs measured throughout the study area are provided below and will also be posted to Ecology's Marine Sediment Monitoring Website. Comparison of these data to similar data from 10 long-term sediment monitoring stations, and from both Bellingham Bay and Commencement Bay, will be examined in a separate report.

Concentrations of PPCPs and PFASs in Elliott Bay

All measured and estimated chemical concentrations are summarized in Table 2. Station locations are presented in Figure 1 and in the Electronic Appendices. Spatial distributions of PPCP and PFAS detected concentrations and ratios to their reporting limits are mapped in Figures 2 through 9. To provide perspective regarding pathways of contaminants to Elliott Bay, the location of combined sewer overflows (CSOs) and stormwater outfalls are mapped in Figure 10. Percent fines, or the silt plus clay fractions from grain size analysis conducted for this study, are also displayed on this map to indicate areas of fine sediment deposition.

Of the 119 PPCP compounds selected for analysis, 13 (11%) were detected. Only 161 (4.5%) of the total 3570 measurements resulted in measured or estimated concentrations. The three most frequently occurring chemicals were: (1) triclocarban, an antibiotic; (2) diphenhydramine, an antihistamine; and (3) triamterene, a diuretic. They were measured in sediments from 25, 18, and 16 stations, respectively. The remaining 10 PPCPs, detected in sediments from 1 to 5 stations, included antibiotics, calcium-channel blockers, an antidepressant, a stimulant, a bronchodilator, and an opiate (Table 2).

Triclocarban, the most frequently detected PPCP, was found at all but five stations along the northern and southwestern shorelines and mouth of the West Waterway (Figure 2). Diphenhydramine, an antihistamine detected at 18 stations, was similarly distributed but absent from some eastern shoreline and central bay stations (Figure 3). Triamterene, a diuretic detected at 16 stations, was primarily absent from the northern and southwestern shoreline stations (Figure 4).

Five other antibiotics and calcium-channel blockers were detected at two deep stations and one northern shoreline station (Figures 5 and 6), while the remaining five detected PPCPs were found in these and other deep and shallow stations and one station in the Duwamish River (Figures 7 and 8). Concentrations and maximum reporting limits for these 10 chemicals varied greatly. Therefore, they are displayed as both the measured concentrations (Figures 5 and 7) and as a ratio to their reporting limit (RL) (Figures 6 and 8).

Three of the 13 PFASs analyzed (23%) were detected. Only 27 of the 390 total PFAS measurements (6.9%) were detected. The most frequently occurring of these, present in sediments at seven stations, was perfluorooctanesulfonate (PFOS), a breakdown product of chemicals used in stain-proof coatings and film-forming foams. The other two PFASs, measured in sediments from one station, were perfluorodecanoate (PFDA) and perfluoroundecanoate (PFUnA) (Table 2). Both are breakdown products of chemicals used in stain-proof coatings. PFOS was detected at all of the deep and some shallower central stations and in one Duwamish River station (Figure 9), while PFDA and PFUnA were detected only at one deep station in the outer portion of Elliott Bay.

Spatial patterns for these detected PPCPs and PFASs were visually compared with the locations of CSOs and stormwater outfalls and measures of percent fines at the 30 monitoring stations in Elliott Bay (Figure 10). Stormwater outfalls are numerous, located in all but a few locations along the northeastern shoreline, and show no discernible patterns with respect to PPCP and PFAS concentrations. There are fewer CSOs, and although some of the PPCPs were detected at stations located near these, there are again no discernible patterns. Most of the higher chemical concentrations appear to be associated with higher percent fines found in the Duwamish waterways and in the deeper locations in Elliott Bay.

Conclusions and Recommendations

A small proportion of all PPCP (4.5%) and PFAS (6.9%) measurements from sediments collected in Elliott Bay for this study was found in detectable concentrations. These data contribute to and help establish a new and growing baseline of information for these chemicals, laying a foundation for better understanding of their relationship with concentrations in marine waters and biota and various biotic endpoints, and of their sources, transport patterns, and fate in the Puget Sound ecosystem.

Recommendations for future work on PPCPs, PFASs, and CECs in general in Puget Sound include:

- Measurement of an equivalent suite of PPCP and PFAS analytes in sediment collected from three additional urban bays, including Bainbridge Basin, Port Gardner Bay, and Budd Inlet, when they are next sampled as part of the PSEMP sediment component. This will establish a baseline of data for these urban bays.

- Development of a prioritized list of CECs and CEC endpoint analyses for future Puget Sound monitoring and assessment programs.

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Table 1. Personal care products and pharmaceuticals (PPCPs) and perfluoroalkyl substances (PFASs) measured in Elliott Bay sediment samples, 2013.

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-Epianhydrochlor-tetracycline
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)

Table 2. Numbers of samples with detected concentrations[a] and summary statistics for detected and estimated concentrations of personal care products and pharmaceuticals (PPCPs) and perfluoroalkyl substances (PFASs) Elliott Bay sediment samples, 2013.

Detected Chemicals	Function	Incidence		Spatial Extent		Minimum detected concentration (ng/g dry wt)	Maximum detected concentration (ng/g dry wt)	Estimated Mean[b] concentration (ng/g dry wt)	Estimated Median[b] concentration (ng/g dry wt)	Minimum - Maximum RL (ng/g dry wt)	Ratio of Concen-tration (ng/g dry wt)/ RL
		Number of detected values (stations) (n=30)	% detected values (stations) (n=30)	Area (km^2) of Elliott Bay	% of Elliott Bay Study Area						
Personal Care Products and Pharmaceuticals											
Triclocarban	antibacterial	25	83.3	23.2	88.4	2.84	96.50	21.34	23.80	1.63-3.40	1.1-31.0
Diphenhydramine	antihistamine	18	60.0	19.5	74.0	0.63	12.70	2.42	3.38	0.44-1.39	1.1-23.9
Triamterene	diuretic	16	53.3	18.9	71.7	0.37	1.47	0.59	0.83	0.24-1.04	1.2-5.0
Miconazole	antifungal	5	16.7	13.3	50.5	1.56	7.29	n/a	n/a	1.10-12.1	1.1-2.6
Azithromycin	antibiotic	2	6.7	6.6	25.2	3.18	4.53	n/a	n/a	1.10-4.24	2.1-2.9
Albuterol	bronchodilator	2	6.7	6.5	24.8	0.30	0.33	n/a	n/a	0.23-0.46	1.1
Verapamil	calcium channel blocker	1	3.3	5.6	21.2	0.37	0.37	n/a	n/a	0.11-1.56	2.8
Ofloxacin	chemotherapeutic antibiotic	1	3.3	5.6	21.2	3.21	3.21	n/a	n/a	1.48	2.2
Norverapamil	calcium channel blocker	1	3.3	5.6	21.2	0.21	0.21	n/a	n/a	0.08-0.45	1.6
Paroxetine	antidepressant	1	3.3	0.4	1.6	5.33	5.33	n/a	n/a	3.04-4.54	1.4
Diltiazem	calcium channel blockers	1	3.3	0.4	1.6	0.35	0.35	n/a	n/a	0.22-0.31	1.2
Codeine	opiate	1	3.3	0.4	1.6	2.93	2.93	n/a	n/a	2.25-3.98	1.1
Cocaine	stimulant	1	3.3	0.3	1.0	0.47	0.47	n/a	n/a	0.13-0.44	1.1
Perfluoroalkyl Substances											
Perfluorooctanesulfonate (PFOS)	stain-proof coating breakdown product, pre-2002 aqueous film-forming foams	7	23.3	15.4	58.7	0.24	0.48	n/a	n/a	0.19-0.29	0.9
Perfluorodecanoate (PFDA)	stain-proof coating breakdown product	1	3.3	5.6	21.2	0.14	0.14	n/a	n/a	0.09-0.14	1.0
Perfluoroundecanoate (PFUnA)	stain-proof coating breakdown product	1	3.3	5.6	21.2	0.20	0.20	n/a	n/a	0.09-0.14	1.4

[a]Field splits and laboratory duplicates not included; [b]Estimated by regression on order statistics (ROS) when nondetect data were present (Helsel, 2012); n/a = too few detected values (<50% detected) to calculate using ROS. RL = Reporting Limit.

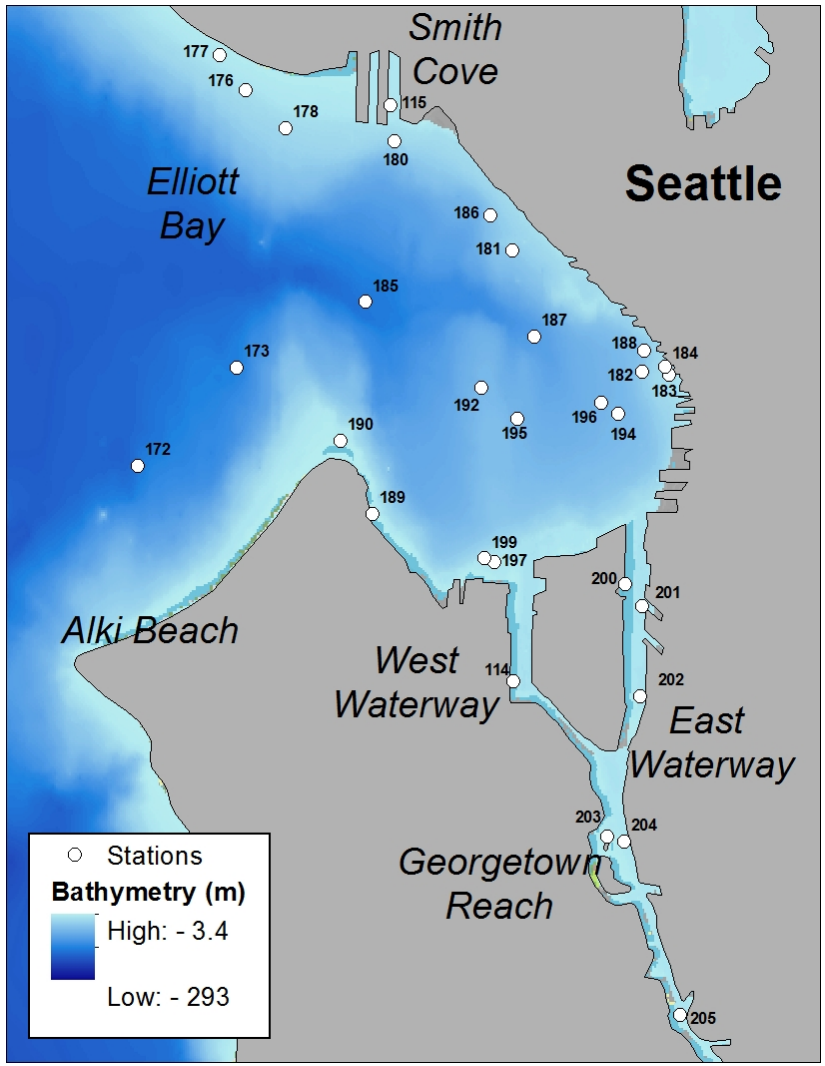

Figure 1. Locations of the 30 stations sampled for PPCPs and PFASs in Elliott Bay in 2013. The numbers on the map are the station identifiers.

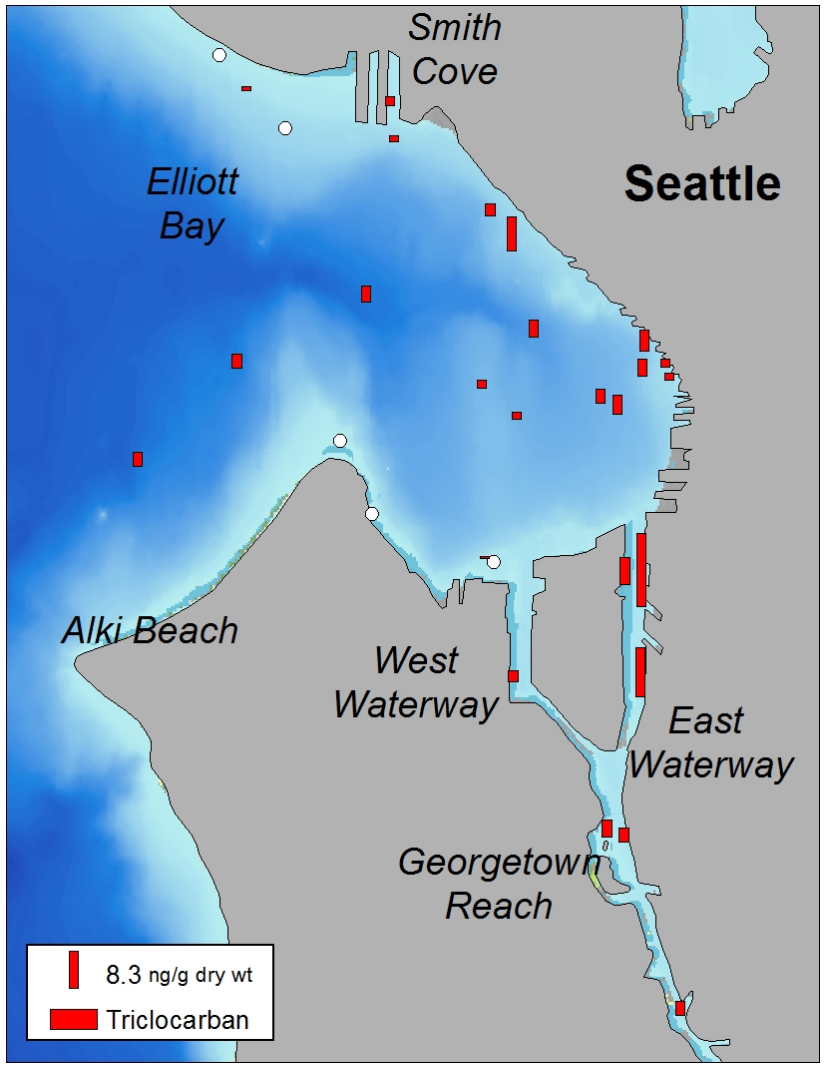

Figure 2. Spatial patterns for Triclocarban concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

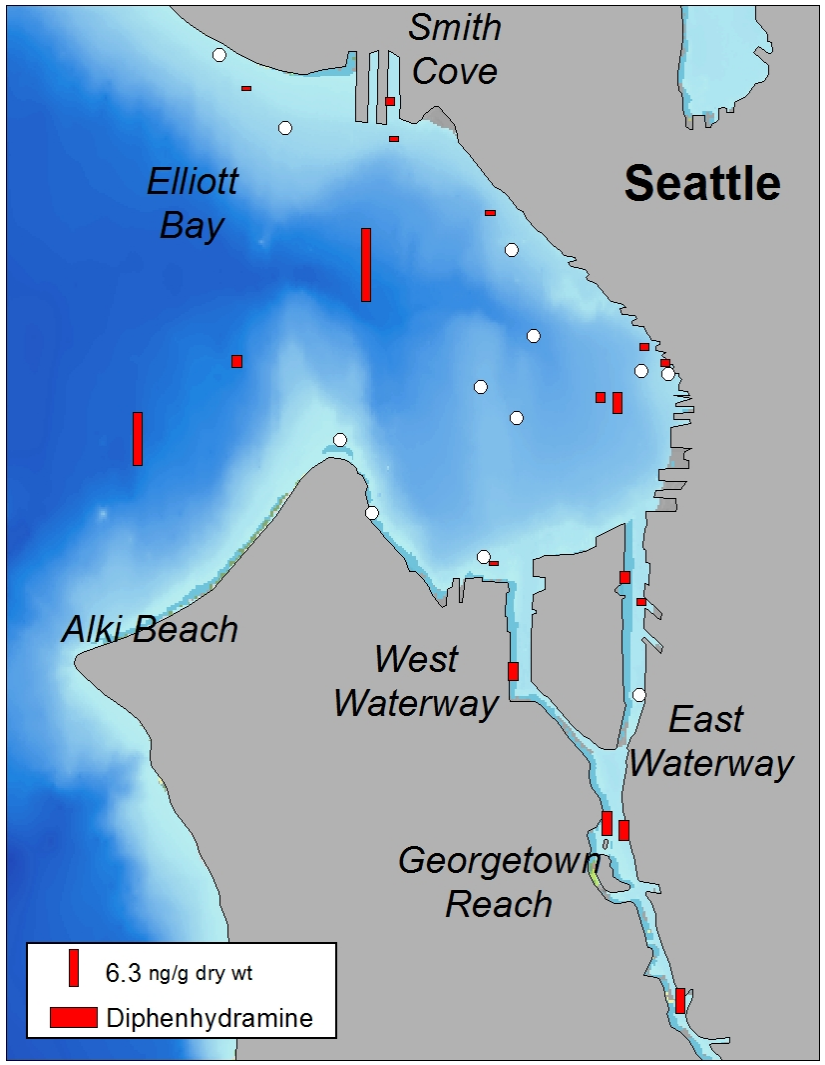

Figure 3. Spatial patterns for Diphenhydramine concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

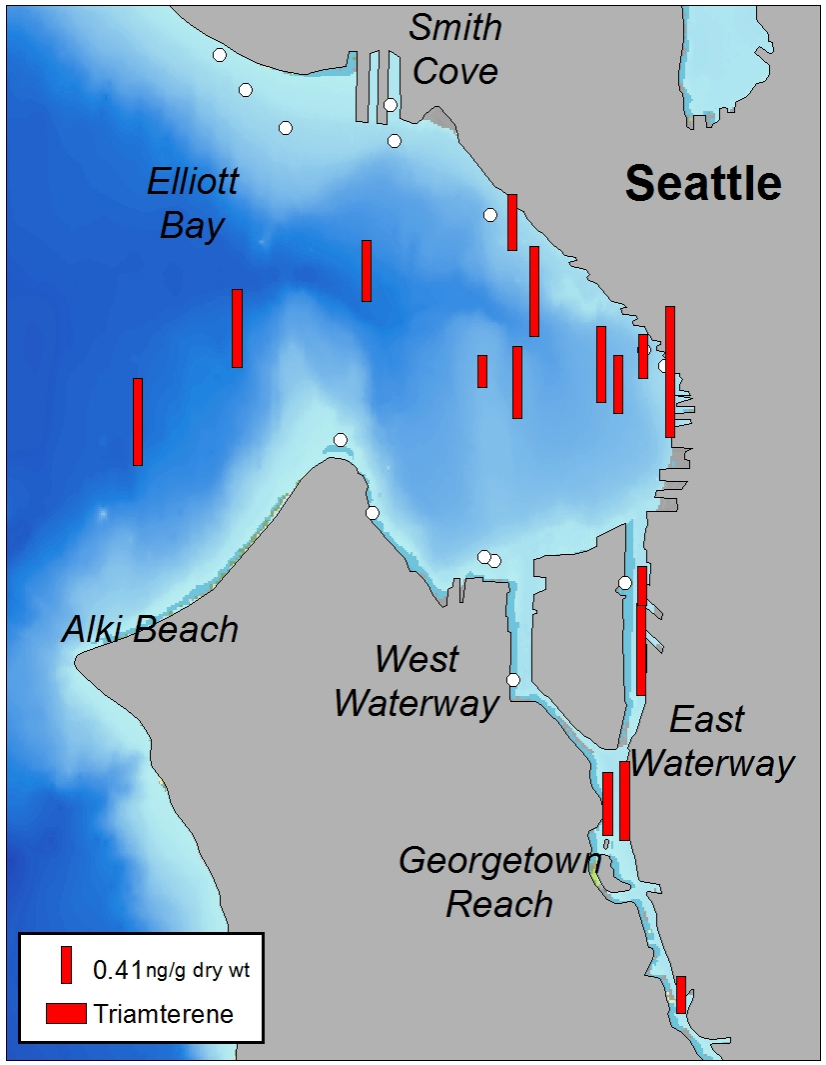

Figure 4. Spatial patterns for Triamterene concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

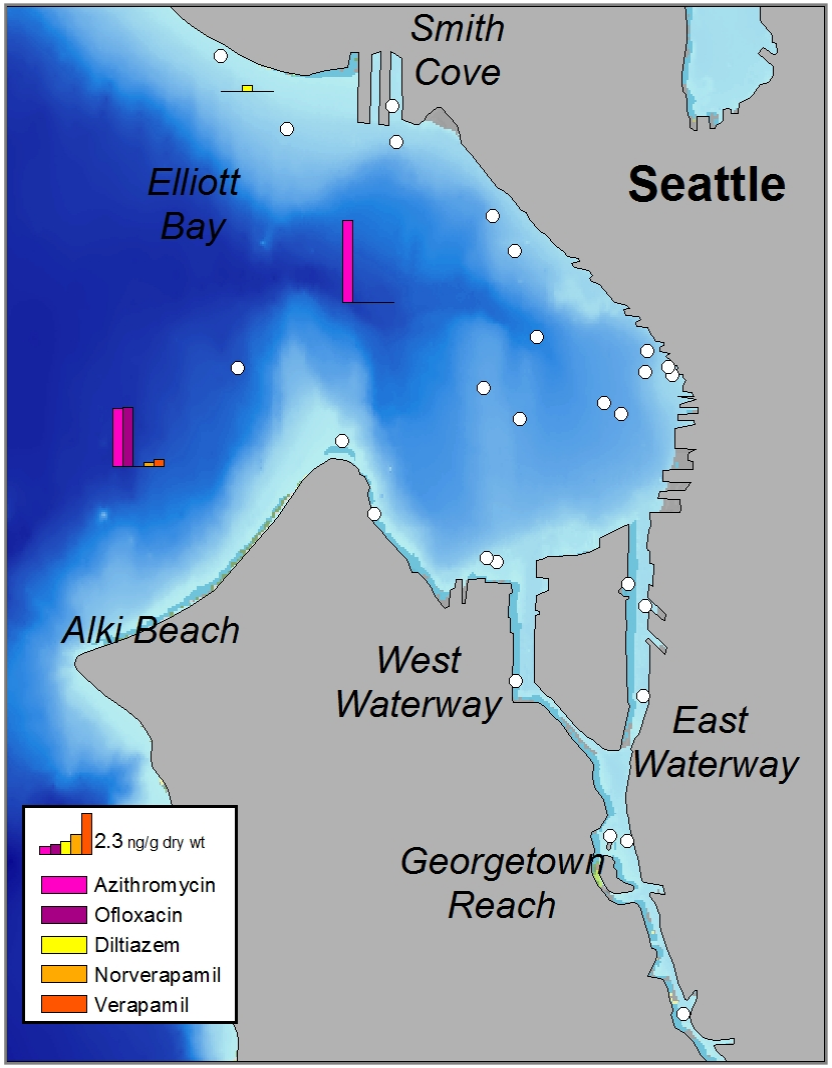

Figure 5. Spatial patterns for 5 antibiotic and calcium channel blocker concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

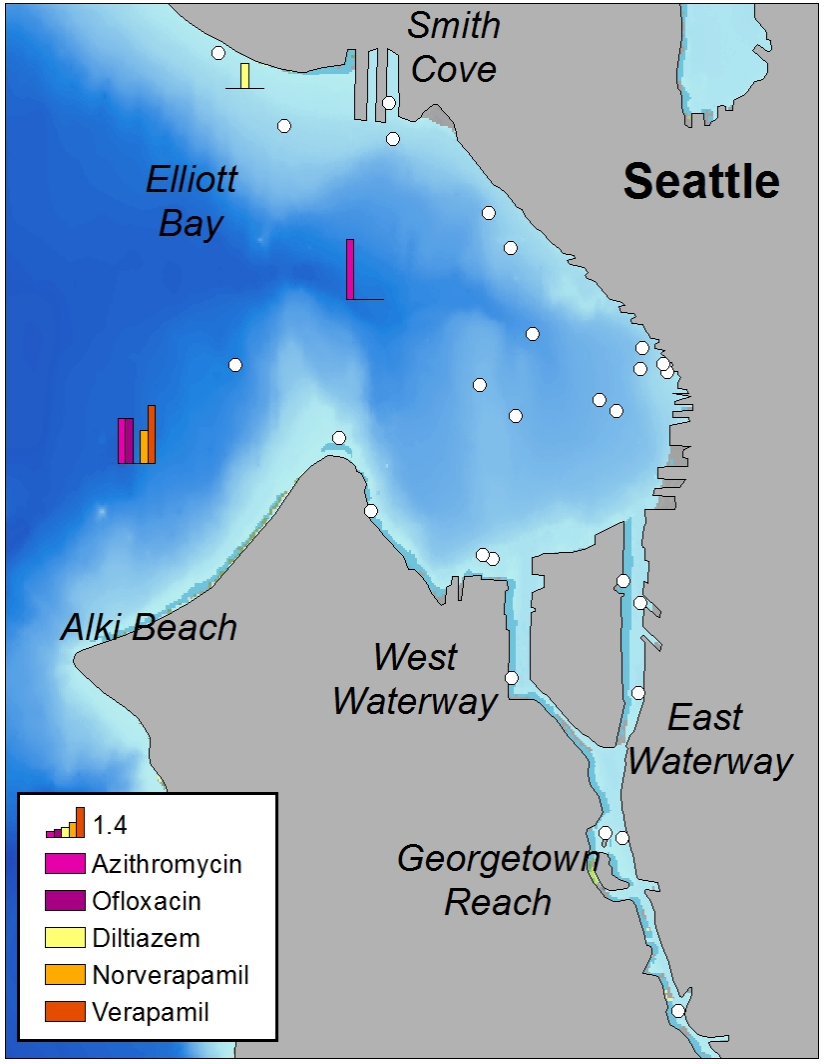

Figure 6. Spatial patterns for 5 antibiotic and calcium channel blocker concentrations detected in Elliott Bay sediments in 2013. Values are reported as the ratio (unitless) of the Result to the Reporting Limit. The white dots indicate nondetects.

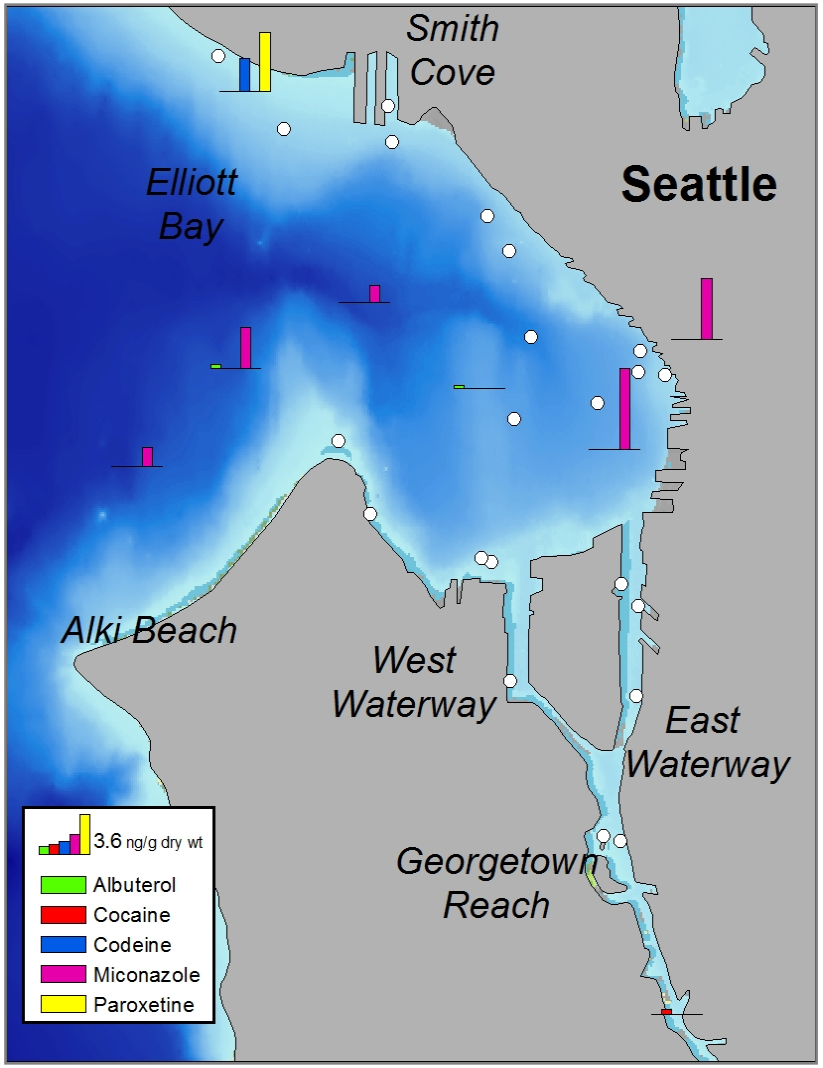

Figure 7. Spatial patterns for other pharmaceutical concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

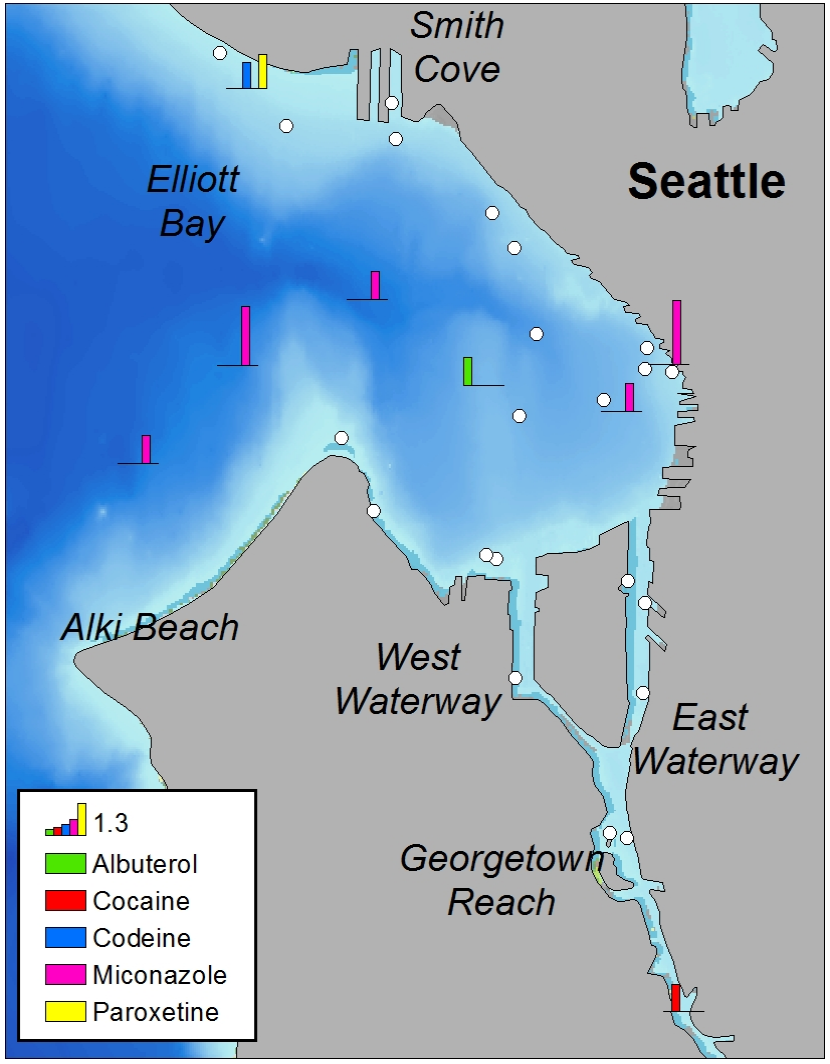

Figure 8. Spatial patterns for other pharmaceutical concentrations detected in Elliott Bay sediments in 2013. Values are reported as the ratio (unitless) of the Result to the Reporting Limit. The white dots indicate nondetects.

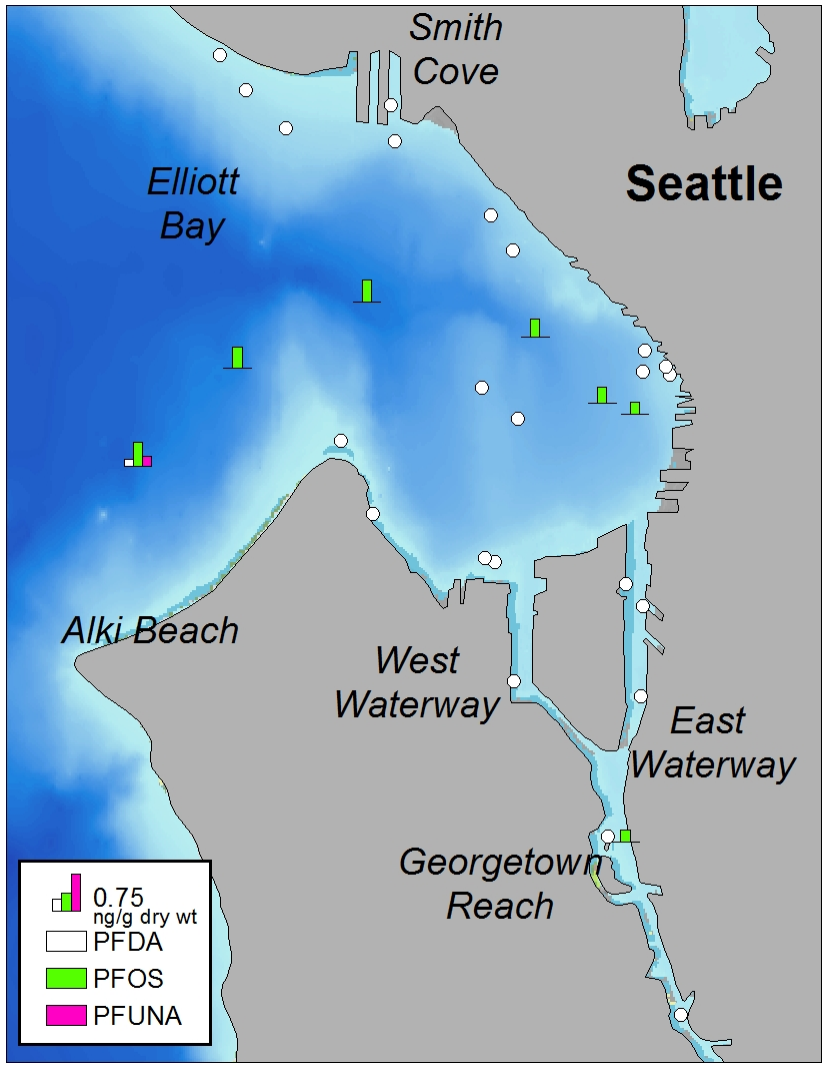

Figure 9. Spatial patterns for perfluoroalkyl substance (PFAS) concentrations detected in Elliott Bay sediments in 2013. The white dots indicate nondetects.

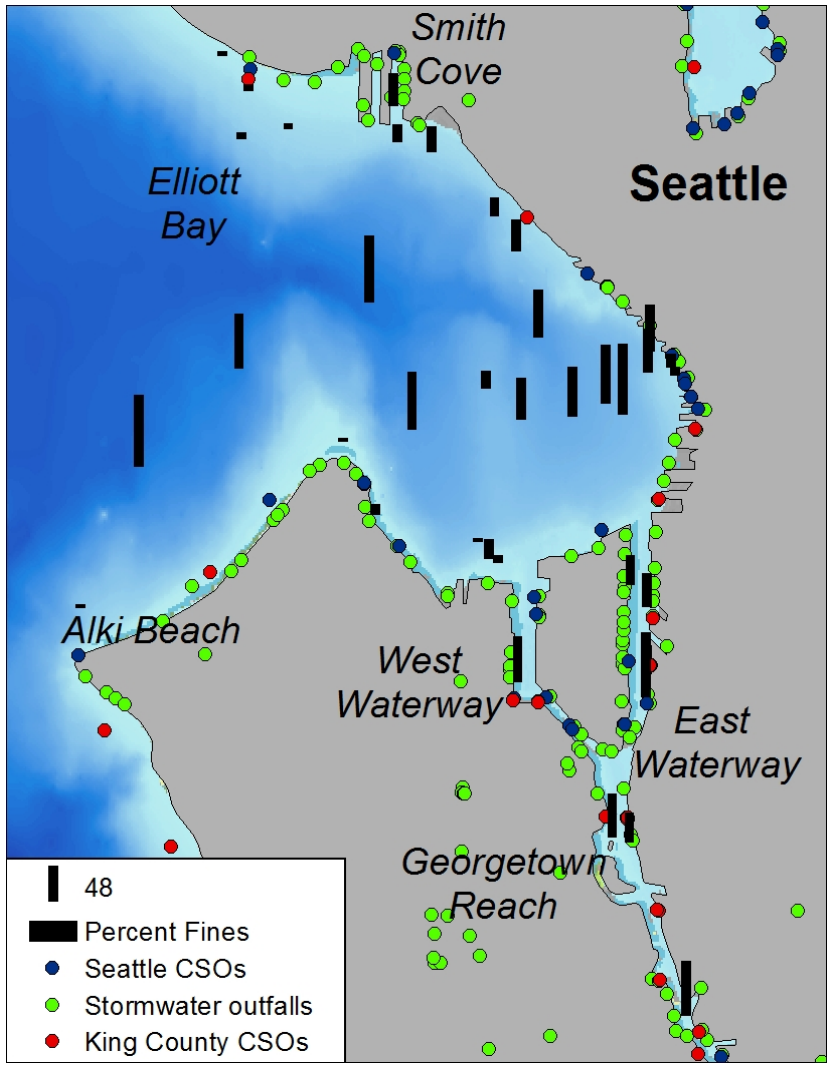

Figure 10. Locations of all Seattle and King County combined sewer overflows (CSOs) and stormwater outfalls in Elliott Bay, and percent fines measured for sediments collected at stations surveyed in 2013.