2012 Ocean Bioaccumulation Survey

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Executive Summary:

Oregon State University collected and analyzed coastal marine species for concentrations of heavy metals and organic pollutants August to October 2012. Target animals included flatfish (speckled sanddab), crustaceans (Dungeness crab & *Crangon* shrimp), and molluscs (*Mytilus* mussels & olive snails). An additional collection of subtidal rock scallops (*Crassadoma gigantean*) was made in fall 2013. Animals were collected from stations near the Georgia Pacific (G-P) outfall pipe adjacent to Nye Beach, OR (mixing zone stations) as well as stations north of Yaquina Head and south of Yaquina Bay. These offshore survey areas and adjacent beaches were stipulated in the RFP as the study sites and correspond to those sites sampled by CH2M Hill in 2010. Physis Environmental Laboratories, Inc. was contracted to conduct the chemical analyses. Organisms were processed for trace metals, PCBs & congeners, phenolics, and PBDEs.

Of 137 possible contaminants tested, 38 were detected in animals from one or more sites. There was little evidence for bioaccumulation of contaminants of concern associated with the G-P outfall pipe. Specifically, **there were no elevated levels of PCBs, phenolic compounds, or PBDEs in any organisms tested**. Some detected metals were found in concentration exceeding the dose found to have no observable effects on the organisms itself. Chemicals with concentrations higher than published toxicity reference values (TRVs) for effects on surrogate aquatic organisms were further investigated by comparing levels in animals collected in the mixing zone to those from reference sites. Tissue concentrations of 22 chemicals either exceeded conservative TRVs for surrogate species or had no TRV for comparison. Among those, 20 were trace metals and two were organic compounds (2,4'-DDD and Oxychlordane).

We could not relate accumulated concentrations to the G-P outfall. **Fish, crabs, and shrimp collected from subtidal sites had higher concentrations of metals at reference locations than the Mixing Zone** (where the G-P outfall discharges). Rock scallops collected near the mixing zone showed higher concentrations of 2 metals as compared to the one reference location near Seal Rock, while 3 metals showed higher concentrations from the Seal Rock reference site. Mussels and olive snails collected from Nye Beach (near the mixing zone as well as the City of Newport Waste Water Treatent Plant output and Nye Creek) had higher concentrations of metals as compared to reference beaches. **None of the detected chemicals approached concentrations for human health concern by seafood consumption**

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Abbreviations

AET – Apparent Effects Threshold

CTD – conductivity, temperature, depth sensor
CVAFS – Cold Vapor Atomic Fluorescence Spectrometry

DO – dissolved oxygen

ERED – Environmental Residue Effects Database

GCMS – Gas Chromatography/Mass Spectrometry
G-P – Georgia Pacific

HMSC – Hatfield Marine Science Center

ICPMS – Inductively Coupled Plasma Mass Spectrometry

LC – Lost Creek

MB – Moolack Beach
MZ – Mixing Zone
MDL – minimum detection limit

NB – Nye Beach
NBN – Nye Beach North
NBS – Nye Beach South
NMZ – North Mixing Zone
NOED – No Observable Effects Dose
NYR – North Yaquina Reference

ODFW – Oregon Department of Fish and Wildlife
OSU – Oregon State University

PBDE – polybrominated diphenyl ethers
PISCO – Partnership for Interdisciplinary Studies of Coastal Oceans
POP – persistent organic pollutant

RL – reporting limit

SBR – South Beach Reference
SMZ – South Mixing Zone
SR – Seal Rock

TRV – toxic reference value

U.S. ACE – United States Army Corp of Engineers
U.S. EPA – United States Environmental Protection Agency

Introduction and Background

The Georgia-Pacific (G-P) Pulp and Paper Recycling Mill in Toledo, Oregon discharges treated wastewater through an ocean outfall (Outfall 001) approximately 4,000 feet offshore Nye Beach in Newport. On March 15, 2010, the Newport City Council adopted Resolution No. 3502 which directed that the fees paid by G-P under the G-P agreement for the years 2008, 2009, and 2010, totaling approximately $170,000, be used for the testing of potential contaminants in ocean waters, beaches, and animals near the G-P ocean outfall. Previous analysis of effluent concentrations in water samples collected at the G-P Toledo Mill, prior to discharge through the marine outfall diffuser, indicated that most total recoverable metals and cyanide concentrations were below the acute and chronic water quality criteria for the protection of aquatic life. However, copper levels were measured above acute and chronic criteria and lead was measured above chronic criteria. After the Outfall 001 effluent is mixed with the seawater it is expected (based on application of the dilution factors as outlined in G-P's NPDES Permit), that copper and lead concentrations are reduced to levels that are much less than the acute and chronic criteria for the protection of aquatic organisms. In May and September 2010, G-P contracted a firm to conduct comprehensive Aquatic Surveys to evaluate the physical, chemical and biological characteristics of the area surrounding the Outfall and at references locations off South Beach (SBR) and north of Yaquina Head (NYR). Seabed bathymetry and sediment and water chemistry data were collected and analyzed and benthic infauna (invertebrates living in the sediment) were identified and enumerated. Phenolics were detected in sediment samples from the mixing zone but were below the screening criteria for sediment, as was the case at the NYR location. At the SBR site, phenolic levels were many times greater than those identified in the mixing zone and NYR; however, only one sample exceeded the U.S. Environmental Protection Agency (USEPA) established Apparent Effects Threshold (AET), and it did not exceed the U.S. Army Corps of Engineers (USACE) screening criterion. Sediment metal concentrations at the mixing zone and both reference locations were below the screening criterion for chemicals of concern. Although there was some variation in the benthic invertebrates among sampling sites, the results showed that the benthic infaunal community within the Outfall 001 mixing zone (MZ) did not differ statistically from the communities outside the MZ or at the reference sites.

While the previous surveys established a snapshot of sediment quality and organism distributions at the mixing zone and reference locations, no tissue samples from benthic organisms were chemically analyzed, neither were any analyses of bioaccumulation of chemicals of concern carried out. Bioaccumulation is defined as the accumulation of chemicals in the tissue of organisms through any route, including respiration, ingestion, or direct contact with contaminated water, sediment, and pore water in the sediment (USEPA 2000). As most metals are not metabolized, bioaccumulation is of particular value as an exposure indicator by providing a longer-term, integrative measure (Luoma & Rainbow 2005). Because of their low solubility in water and their resistances to chemical and metabolic degradation, most Persistent Organic Pollutants (POPs) are eliminated from organisms very slowly; as a consequence, POPs can accumulate to relatively high levels in organisms even at low environmental exposures (Vallack et al. 1998). Thus, although values for both metals and organic compounds were low in tested sediment, there is potential for higher concentrations to exist within the tissues of

benthic organisms that are in contact with, and in many cases ingest, the sediment.

The goal of this study was to determine if accumulation of effluent-borne pollutants could be detected in resident coastal species and related to discharge from the G-P outfall. In August 2012, pursuant to Oregon State University's (OSU) proposal for the City of Newport Ocean Bioaccumulation Survey, OSU conducted a 'Tier 1' analysis of concentrations of heavy metals and organic pollutants in a suite of coastal Oregon marine species. Tier 1 is part of a three-tiered sampling plan recommended by the USEPA (USEPA 2000) where the first stage is to determine if bioaccumulation of effluent-borne pollutants can be detected in organisms of interest (Characterization of Problem). If warranted, Tier 2 studies would identify the specific effluent-related parameters responsible for contamination (Diagnosis of Causes) and suggest a plan for assessing overall risks to ecosystem and public health. Tier 3 would begin intensive monitoring and/or cause and effect research (Diagnosis of Interaction and Forecasting).

Physis Environmental Laboratories, Inc. (Physis) was contracted by Oregon State University to conduct the chemical analyses. Organisms were processed for trace metals, PCBs and congeners, and phenolics, as called for in the RFP. Additionally, organisms were analyzed for an additional class of compounds, the polybrominated diphenyl ethers (PBDEs). PBDEs are a group of globally distributed contaminants similar in structure to PCBs (Ueno et al. 2004). Often used in flame-retardants, PBDEs have greater potential to bioaccumulate than PCBs and therefore may pose more of a risk to wildlife and human populations (Burreau et al. 2006). In Canada, PBDEs associated with urbanization and run off near pulp and paper mills were found in Dungeness crab (*Metacarcinus magister*) as well as English sole (*Parophrys vetulus*) (Ikonomou et al. 2006). In the 2008 Southern California Bight Survey, 100 % of the sediment samples collected from Pt. Conception, CA, to the Mexican border contained PBDEs (Bay et al. 2011). It was concluded that the sediment-associated PBDEs were a potential pathway to bioaccumulation into higher marine organisms such as mussels and marine mammals (Schiff et al. 2011); thus, the researchers felt these additional compounds should be included in the analysis.

Oregon coastal species selected for this study are representative of local populations of demersal finfish, epibenthic crustaceans, and sessile and infaunal molluscs and constitute commercially important species as well as functional groups with a diverse range of ecological niches and feeding strategies, including transient scavengers and infaunal, epifaunal, and pelagic filter feeders. In total, we collected and analyzed six types of organisms: flatfish, shrimp, crab, scallops, mussels, and snails.

Methods

SAMPLING

Prior to the collection of organisms, an Oregon Department of Fish and Wildlife (ODFW) Scientific Taking Permit for marine fish and invertebrates was obtained, along with the approval of an Animal Care and Use Protocol by the Institutional Animal Care and Use Committee (IACUC) at OSU for the handling of live vertebrate animals. Offshore sampling was conducted at the three primary sampling locations used in the Comprehensive Aquatic Surveys: the North Yaquina Reference Area (NYR), South Beach Reference Area (SBR), and the G-P Outfall Primary Survey Area (Figure 1). The G-P Outfall Primary Survey Area was subdivided into three distinct sampling regions; the North Mixing Zone (NMZ), Central Mixing Zone (MZ), and South Mixing Zone (SMZ). Subdivision of the G-P Outfall Primary Survey Area into regions was done to aid in characterizing the extent of any possible contamination stemming from the Outfall. Onshore sampling sites varied depending on the species collected, identified in each section below. Coordinates and collection numbers for all samplings are in Appendix 2, Table 1. Undergraduate and graduate student volunteers served as sampling crew throughout the project.

Sampling was first conducted August 2nd to October 17th, 2012. Flatfish (speckled sanddab, *Citharichthys stigmaeus*) and shrimp (*Crangon* sp.) were collected using a 5 m otter trawl with 20 mm wall netting and 3 mm liner netting that was towed for 5 minutes at each sampling location. *Crangon* shrimp were collected in place of mysid shrimp as originally proposed because mysids were encountered in densities too low to constitute meaningful samples. Dungeness crabs were collected using 3 weighted crab pots on 8 hour baited soaks. Once collected, all samples were flash frozen onboard the R/V Kalipi using dry ice and later stored in a -20 °C freezer at Hatfield Marine Science Center (HMSC). CTD casts were done during crabbing and trawling. All offshore sampling was performed aboard the R/V Kalipi, a 29’ cabin cruiser operated by the Partnership for Interdisciplinary Studies of Coastal Oceans.

AAUS-certified scientific SCUBA divers from OSU attempted to collect mussels from hard, sub-tidal structures at the sampling locations, but densities were too low for meaningful collection. As a substitute, mussels (*Mytilus* sp.) were collected from the rocky intertidal near three of the sampling locations. Because the three Outfall Survey Areas are located directly adjacent to one another offshore and three distinct subsets of rocky structure could not be identified onshore, one sample site of mussels (Nye Beach) was collected to represent the overall region of the Outfall Primary Survey Area. The rock outcrop from which the mussels were collected is located over 3000 ft from the offshore discharge point of the G-P outfall. The north reference site for intertidal mussels was Moolack Beach (MB) and the south reference site was Seal Rock (SR).

Because of the desire to have a subtidal filter-feeding organism assessed, in fall 2013 OSU divers collected rock scallops (*Crassadoma gigantean*) from a subtidal rock reef near the Mixing Zone and from a reference location to the south between South Beach and Seal Rock. We were not able to collect rock scallops from a northern reference location.

OSU staff and undergraduate volunteers from the OSU Marine Team attempted to collect razor clams from intertidal sand flats on beaches adjacent to the coastal sampling locations, but razor

clams were found in densities too low to constitute meaningful samples. As a substitute, olive snails (*Callianax biplicata*) were collected from the intertidal sand flats at locations directly onshore (Nye Beach) of the offshore collection sites. The north reference beach for olive snails also was Moolack Beach and the south reference beach was Lost Creek.

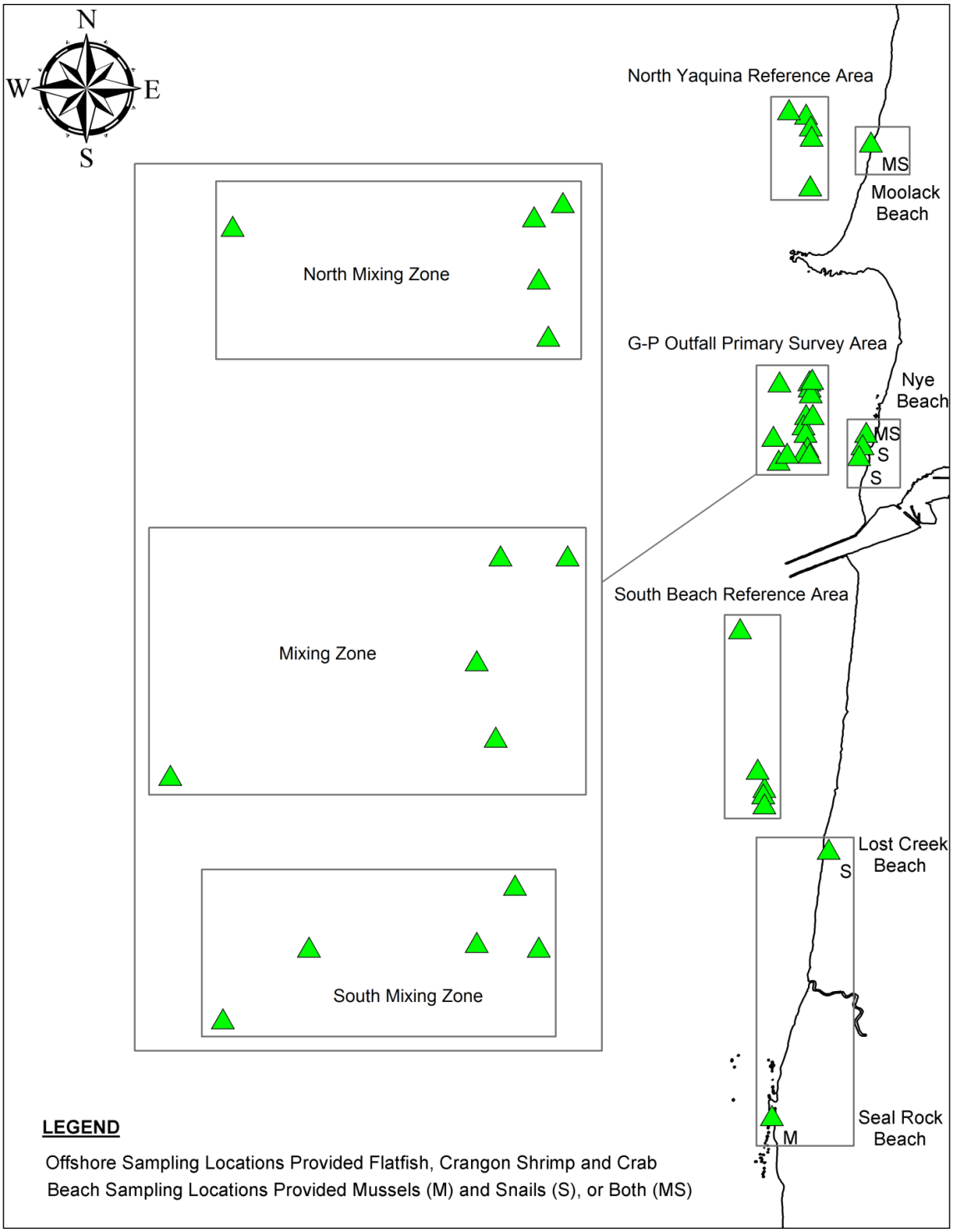

Figure 1 – Locations of coastal sampling sites and adjacent beach sampling sites. Triangles represent site coordinates recorded via GPS while carrying out organism collections.

TISSUE PROCESSING AND ANALYSIS (TESTING)

Physis Environmental Laboratories, Inc. (Physis), located in Anaheim, California, carried out the tissue processing and analysis. Samples were shipped overnight from HMSC to Physis for compositing and analysis. Samples were received by Physis and maintained at -20 °C until processed for analysis. At Physis, morphometrics including wet weight, crab carapace width, flatfish total length, and mussel, snail, and *Crangon* composite weights were recorded. In order to sufficiently capture variation in highly mobile target organisms that may travel in and out of the impact zone around the Outfall diffuser, five replicate composites of Dungeness crab, *Crangon*, *Mytilus*, and olive snail from each of the sampling sites were assembled. Ten individuals comprised a single composite sample for Dungeness crab, *Mytilus*, and olive snails. An effort was made to obtain a similar size distribution of organisms in composite samples. For *Crangon*, an approximate volume of 250 ml of shrimp was used to make composite samples. Each individual speckled sanddab was considered a single sample. At the request of the City, five additional fish were processed in each of the three outfall mixing zones for a total of ten fish from each of those areas. Prior to compositing and analysis, all organisms were removed from their shell and, because sediments retained in the gut may bias whole body analyses (Sample et al. 1998), stomachs of flatfish and crab were removed. Whole body analyses are preferable to individual tissue or organ sampling as differences in internal distribution rates and physiological functions affect rates of uptake and may cause accumulation to be more pronounced in organs such as the liver, gills, and kidneys (Karuppasamy 2004; Fabris et al. 2006; Murugan et al. 2008).

Tissue samples were analyzed for trace metals, PCBs, congeners, and phenolics and PBDEs. For a full list of samples per location see Appendix 2, Table 1. A full list of individual analytes, Minimum Detection Limits (MDL), and Reporting Limits (RL) can be found in Appendix 2, Tables 2 and 3. All dissected tissues were homogenized in a Class 100 Laminar-flow clean hood with a Teflon-coated bench and class 100 cleaned air supply. Physical measurement data was collected and animal tissue was homogenized using a pre-cleaned tissue grinder. Contact with plastic and metals was minimized, or avoided as much as practical, to minimize contamination of the samples during homogenization. Quality control processes can be found in Appendix 3.

Trace Organics Analysis

Physis uses a Soxhlet extraction procedure with methylene chloride. All solvents used were of pesticide grade solvent quality and all glassware was cleaned by heating at 1000 °F for 4 hours. Sample cleanup was performed on all samples using Alumina/Silica Gel chromatography to remove interfering lipids and fatty acids from the animal tissues. Sample extracts were concentrated using roto-evaporation followed by gentle blow-down using nitrogen.

All tissue extracts were analyzed using Gas Chromatography/Mass Spectrometry (GCMS) Quadrupole systems in the “full-scan” mode. Chromatographic separation was achieved using a DB-5, 60-meter, 0.25 mm ID, 0.25 μm film thickness column temperature programmed at a maximum rate of 2.5 °C per minute. Using 60-meter narrow-bore columns and a slow

temperature program rate assures maximum separation of all peaks in the chromatogram and enhances the qualitative identification and quantitation of the target compounds. The target ion and a minimum of 2 or more qualifier ions (when possible) were evaluated for confirming peak identification. Quantitation was based on a 5-point calibration curve using standards purchased from a commercial supplier traceable to NIST. For data quality objectives see Appendix 2, Table 4.

Trace Metals Analysis

Samples were digested using a Milestone microwave digestion system containing sealed Teflon vessels. Microwave time and temperature conformed to Laboratory Method parameters. Samples received a nitric/hydrochloric acid digestion. All acids were of Optima Grade and all labware was constructed of Teflon or plastic.

All metals except mercury were analyzed using Inductively Coupled Plasma Mass Spectrometry (ICPMS) by EPA Method 6020. Quantitation was performed using an internal standard of Rhodium or Thulium and interference corrections were applied where needed. The Physis ICPMS system was calibrated using a 5-point calibration curve on a daily basis with calibration standards purchased from a commercial supplier and traceable to NIST.

Mercury samples were analyzed using Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) by EPA Method 245.7. Samples were digested for 2 minutes using aqua regia at 95 °C. The digestate was preserved by adding a bromine monochloride (BrCl) solution followed by oxidation with potassium permanganate. After oxidation, the sample was reduced with NH_2OH-HCl to destroy the free halogens. The sample was then reduced with stannous chloride ($SnCl_2$) to convert Hg (II) to volatile Hg (0). The Hg (0) is separated from solution by purging with high purity argon gas through a semi-permeable dryer tube. The Hg (0) passes into an inert gas stream that carries the released mercury into the cell of a CVAFS for detection.

The Physis CVAFS system was calibrated using a 5-point calibration curve using calibration standards purchased from a commercial supplier traceable to NIST. For data quality objectives see Appendix 2, Table 5.

TOXICITY REFERENCE VALUE SELECTION

Toxicity reference values (TRV) refer to tissue concentrations of a contaminant of concern that have been shown to have a toxic effect **on the animal of interest or a surrogate species**. ***These values do not represent toxicity to humans*** that might result from consuming the organism of interest. Reported TRVs are highly dependent on both the type of tissue tested and the effects measured. To be consistent with the analyses conducted in this survey, TRVs obtained utilizing whole body organisms to test toxicity were chosen preferentially over those that were developed using only a portion of a study animal. For example, if two TRVs were available, one from a study utilizing flatfish digestive tract and the other utilizing the entire fish, the TRV from the whole body study was chosen. In addition, two sets of effect measures were compiled to help determine the level of biological risk to the organisms. First, a 'conservative' set of TRVs from studies using No Observable Effect Dose (NOED) measures was compiled. These values represent the most conservative measure of risk available and it is reasonable to assume tissue

concentrations below these levels pose little risk to the species of interest because it is the dose at which no observable effects were seen on the organism, while concentrations above that threshold were shown to have some effect on the experimental organism. A second set of 'less-conservative' TRVs from studies utilizing measures such as LD50s (the concentration required to reduce the survival of a study population by 50%) to judge toxicity was compiled. These values may represent a more realistic measure of tissue toxicity when chemical concentrations not only show measureable effects but also result in organism death.

Toxicity Reference Values were researched for each chemical detected in each of the five study organisms; however, because TRVs are not available for every organism, appropriate biological surrogates were used where available (for example a freshwater snail if no marine snail had a TRV). For a few chemicals, no TRVs could be identified. Information on the specific measures used to define both types of TRVs for exceeding analytes is presented in Tables 5 and 6.

Toxicity reference values were compiled using the Jarvinen and Ankley toxicity/residue database, available at the website http://www.epa.gov/med/Prods_Pubs/tox_residue.htm, and the USACE/USEPA Environmental Residue-Effects Database (ERED; http://el.erdc.usace.army.mil/ered/).

DATA PROCESSING AND STATISTICAL ANALYSIS

Several levels of screening were applied to the raw data received from Physis. First, all non-detected chemicals were precluded from further analysis. Tissue concentrations from detected chemicals were then compared to the conservative TRVs determined from the USEPA and USACE databases. At this stage, if the concentration in any sample from any site was found to exceed the conservative TRV, the chemical and organism were carried on for further evaluation. For instances where a chemical was detected but no TRV could be found, the species and chemical was also carried on for further analysis as a precautionary measure.

At the next stage of analysis, tissues concentrations from composite samples were averaged for each site to compare samples from the G-P mixing zone with samples obtained from reference sites to the north and south. There were several instances where a chemical was detected and exceeded a conservative TRV in only one or two of the composites, so when averaging the data, zeros were put in the place of any non-detect so that n = 5 (10 in the case of fish from the mixing zone) for each site. Data were then analyzed using non-parametric Kruskal-Wallis to determine if there were differences among sites and Wilcoxon Each Pair comparisons to determine the rank order of sites. For those chemicals that showed differences in concentrations among sites and where at least one MZ site was higher than at least one of the reference sites, concentrations were plotted and compared to FDA or other human health thresholds where available.

Kruskal-Wallis and Wilcoxon Each Pair comparisons were also run on average morphometric data to determine if there were significant differences in the size of organisms collected at each sampling location.

Results

OCEANOGRAPHIC AND SAMPLING CONDITIONS

CTD casts were not conducted during the first crab sampling event on 8/2/2012, but oceanographic conditions were sampled on all subsequent crabbing and trawling events. During the time of sample collection, dissolved oxygen (DO) averaged 5.75 ml/L, temperature 9.74 ° C, and salinity 33.55 PSU at the bottom of the CTD cast (Table **1**). In general, temperatures were typical for the season. Dissolved oxygen levels were not approaching hypoxia, and pH conditions were favorable. Collection depths for Dungeness, *Crangon*, and sanddabs ranged from 12.9 to 17.6 m (42.3 - 57.7 ft).

Table 1: Summary of CTD data from crabbing and trawling events. Values are from the deepest point of the CTD casts and represent conditions closest to the benthic organisms collected.

Activity (target sp.)	Date	Site	Depth (m)	Temp (deg. C)	DO (ml/L)	Salinity (PSU)	Fluorescence [mg/m^3]	pH	Turbidity
Crabbing (Dungeness crab)	8/18/12	NYR	15.41	9.84	5.28	33.63	2.09	8.07	1.68
	8/18/12	NMZ	13.44	10.10	6.06	33.61	7.45	8.09	1.68
	8/18/12	MZ	13.52	9.55	5.08	33.65	3.25	8.03	1.68
	8/18/12	SMZ	13.62	9.79	5.67	33.63	4.54	8.09	1.68
	8/18/12	SBR	16.85	9.79	6.16	33.48	7.72	7.88	1.68
	8/19/12	NMZ	15.84	9.90	6.14	33.61	6.81	8.21	1.67
	8/19/12	MZ	13.53	10.35	6.89	33.53	9.23	8.12	1.67
	8/19/12	SBR	16.90	9.46	5.63	33.53	7.30	7.87	1.69
Trawling (Speckled sanddab & *Crangon* shrimp)	9/19/12	NYR	16.52	9.19	4.62	33.51	1.35	8.04	1.69
	9/19/12	NMZ	16.30	9.46	5.30	33.53	5.76	8.11	1.68
	9/19/12	MZ	12.94	9.54	5.70	33.50	6.89	8.18	1.68
	9/19/12	SMZ	14.48	9.58	5.79	33.53	8.18	8.18	1.68
	9/19/12	SBR	17.65	10.04	6.49	33.36	10.84	8.03	1.67

TISSUE ANALYSIS

In total, 38 of the 127 tested chemicals were detected in animal tissues (Table **2**). Mean concentrations of analytes across all sampling stations are presented to demonstrate that concentrations of any particular metal vary widely across species and that no species always carries the highest burden of metals. Thirty-five chemicals were present in Dungeness tissue, 21 in olive snail tissue, 20 each in both *Crangon* shrimp and rock scallop tissues, and 19 each in both sanddab and *Mytilus* tissues. PBDEs were not detected in any animal tissues; Dungeness crabs were the only organisms in which organic compounds were detected. Tissue concentrations of twenty-two chemicals either exceeded the conservative toxicity reference value (TRV) or had no TRV available for comparison and were carried on for statistical analysis. Among those 22 chemicals, 20 were trace metals and two were organic compounds (2,4'-DDD and Oxychlordane), neither of which had a TRV.

Dungeness Crab (collected via crab pots)

In Dungeness crab, all organic compounds were below published TRVs. The two organics that were carried forth for further analysis had no TRV for comparison and were found in only one composite sample each. Oxychlordane was detected in one sample from the mixing zone and 2,4'-DDD was detected in one sample from the north Yaquina reference (NYR).

Concentrations of 6 metals exceeded the conservative TRVs and 6 metals had no TRV for comparison; thus, these 12 metals were carried forth for further analysis. Based on the Kruskal-Wallis and Wilcoxon tests, only one metal (vanadium) showed differences in tissue chemical concentrations of Dungeness crabs among sites (Table **3**; Figure **1**). Concentrations of vanadium were higher in organisms collected from the NMZ and SMZ sites, as well as the SBR, than in animals collected from the NYR. While vanadium concentrations from two individual SBR samples did exceed the conservative TRV, averaged concentrations did not exceed the conservative TRV of finding no observable effect on mortality when analyzing whole body tissue of the surrogate organism – shore crab (Table **5**). Dungeness crabs collected from both reference locations were significantly larger than those collected from the mixing zone (Table **8**). Because the highest and lowest concentrations of vanadium were found in the largest crabs, it does not appear that the size of the crabs influenced the observed pattern of accumulation.

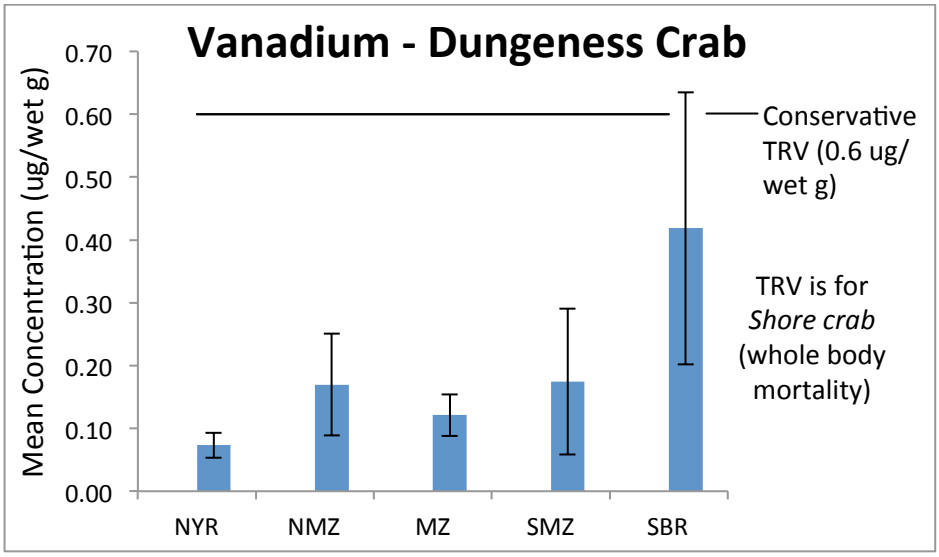

Figure 1: Average concentrations of vanadium in Dungeness crab across sites.

Crangon Shrimp (collected via trawl)

Crangon shrimp tissues were found to contain 3 metals in concentrations that exceeded the conservative TRVs and 4 detected metals that had no TRV for comparison; thus these 7 metals were carried forth for further analysis (Table **3**). Two metals (vanadium and zinc) were found in higher concentrations in animals from at least one of the mixing zone sites than from the NYR; however, concentrations of both were still highest in animals collected from the SBR (Figures 2 & 3). For both vanadium and zinc, concentrations in the animals collected from the mixing zone and SBR exceeded the NOED for mortality, based on studies on other crustacean species (Table **5**). For *Crangon*, the highest concentrations of vanadium and zinc were found in the largest animals (SBR; Table **7**); however, the smallest shrimp samples had concentrations that were not different from the other sites, again suggesting that organism size is not a factor.

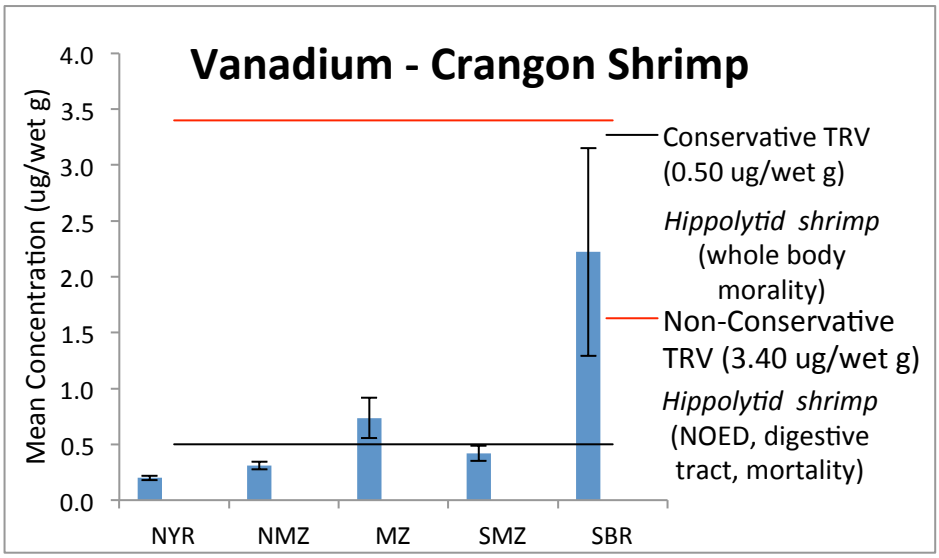

Figure 2: Average concentrations of vanadium in Crangon shrimp across sites

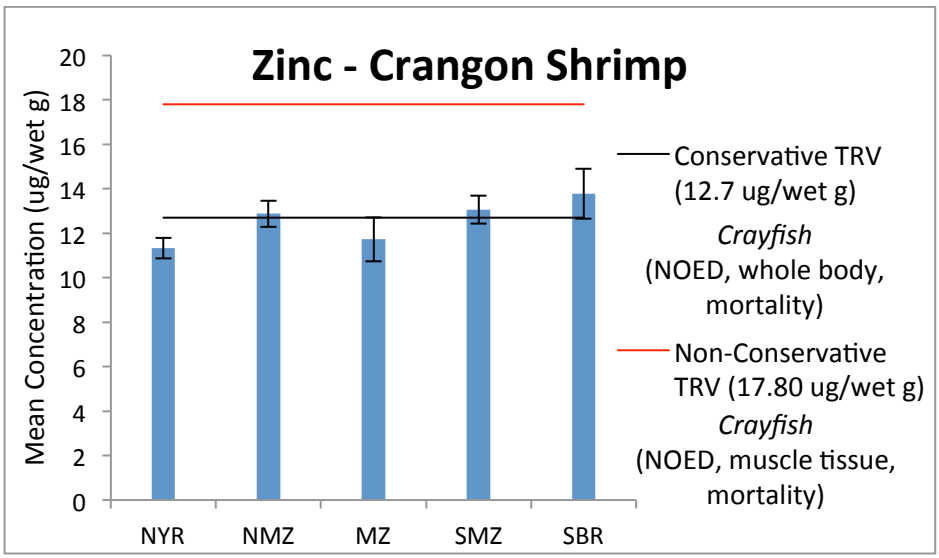

Figure 3: Average concentrations of zinc in Crangon shrimp across sites

Speckled Sanddab (collected via trawl)

In speckled sanddabs, 5 metals were detected in concentrations that exceeded conservative TRVs and 3 detected metals had no TRV for comparison; thus these 8 metals were carried forth for further analysis (Table **3**). For those metals that varied among collection sites (iron, selenium, strontium, and tin), values were typically highest in fish collected from the NYR (Table 3). Tin concentrations were significantly higher in fish from the mixing zone than from SBR; however values were higher still at the NYR (Table **5**; Figure **4**). No differences in sanddab sizes were detected (Table **8**).

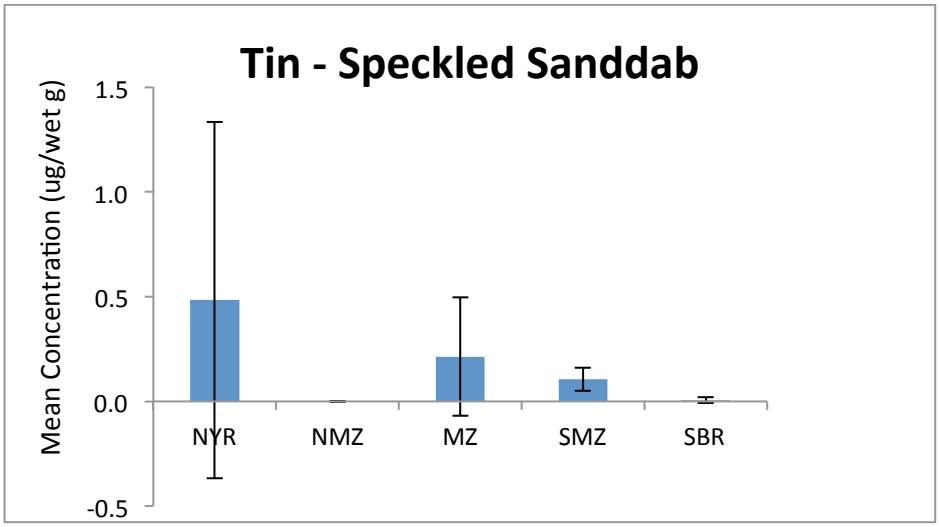

Figure 4: Average concentrations of tin in speckled sanddab across sites.

Rock Scallops (collected via diving)

Rock scallop tissues were found to contain 2 metals in concentrations that exceeded the conservative TRVs and 7 metals that had no TRV for comparison; thus these 9 analytes were carried forth for further analysis (Table **3**). **Two metals (aluminum and barium) were found in higher concentrations in scallops from the mixing zone site than from reference area near Seal Rock** (Table **5**; Figures 5 & 6).

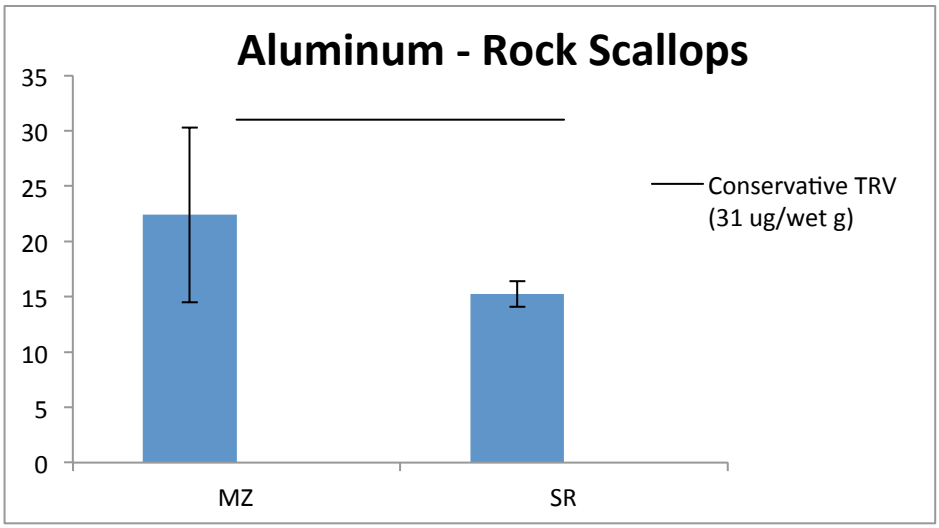

Figure 5: Average aluminum concentrations in scallops collected from near the mixing zone and a reference site to the south.

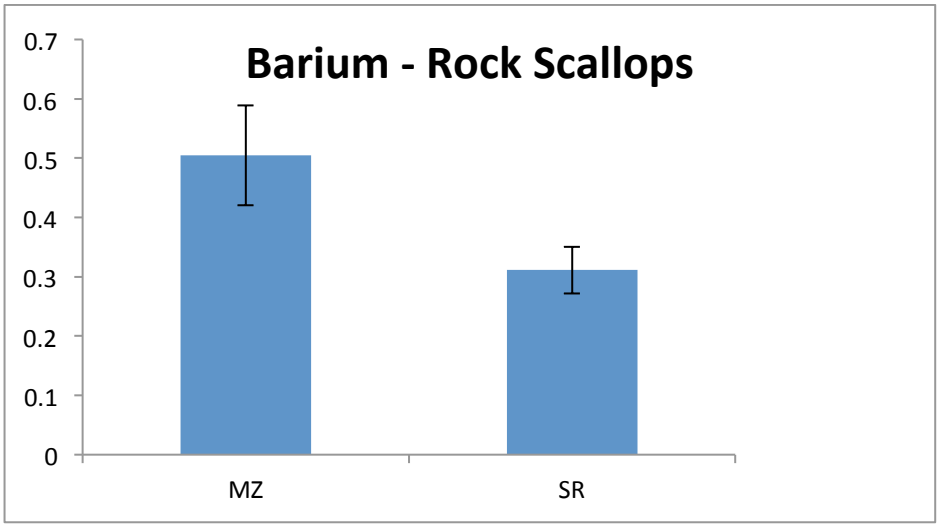

Figure 6: Average barium concentrations in scallops collected from near the mixing zone and a reference site to the south.

Mytilus Mussels (collected from the intertidal)

Mytilus mussels collected from rocky outcrops at Nye Beach and north and south reference locations (3 sample sites total) contained 4 metals in concentrations higher than the conservative TRVs and 5 metals without TRVs; thus these 9 metals were carried forth for further analysis (Table **3**). **Only arsenic concentrations in mussels showed differences among sites**. Concentrations of arsenic in mussels from the three collection locations exceeded the no observable effects dose for mortality with mussels collected from Nye Beach having the highest arsenic concentrations (Table **5**; Figure **7**). The largest collected mussels were from onshore rocks near Nye Beach (Table **7**), which also had the highest arsenic concentrations; however, these larger mussels also had the lower concentrations of aluminum, iron, molybdenum, tin, and vanadium.

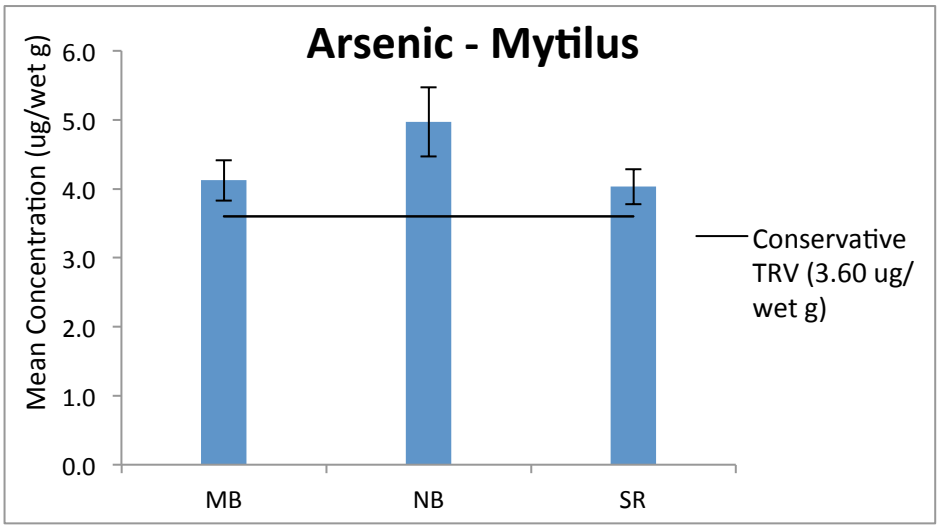

Figure 7: Average concentration of arsenic in mussels across rocky intertidal collection sites.

Olive Snails (collected from intertidal)

Concentrations of 14 metals in olive snail tissue exceeded conservative TRVs or had no TRV for comparison. In some cases (lead, vanadium), TRVs were based on echinoderm studies rather than snails due to lack of references for molluscs. **Olive snails had the greatest number of chemicals (11) with tissue concentrations that were significantly higher in the Nye Beach area** (Table **4**; Figures 8-19). Selenium concentrations in snail tissues from both Nye Beach and Moolack Beach (north reference) were found to exceed both the conservative and less-conservative TRVs (Table **6**). Generally, snails from Lost Creek (south reference) were heavier than Moolack Beach and North Nye Beach samples, but LC samples were not different from the other Nye Beach samples (Table 8).

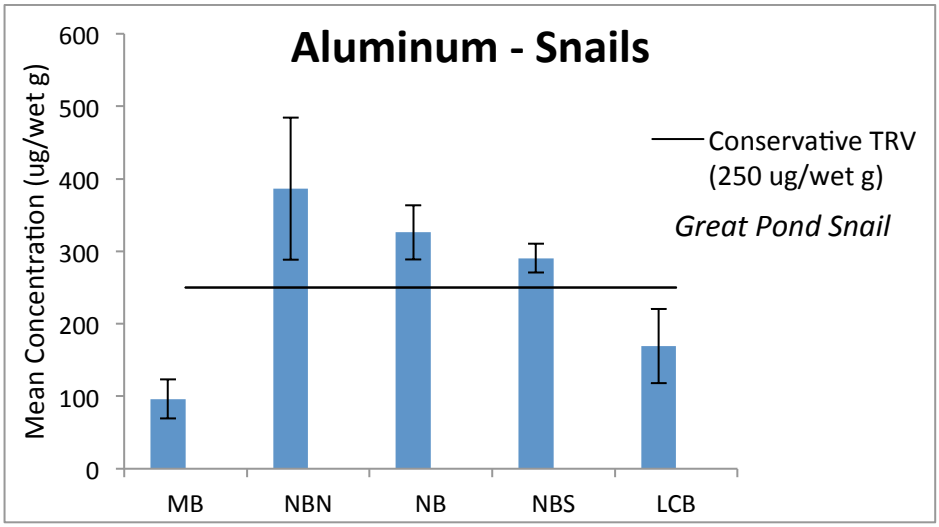

Figure 8: Average concentrations of aluminum in olive snails across beaches

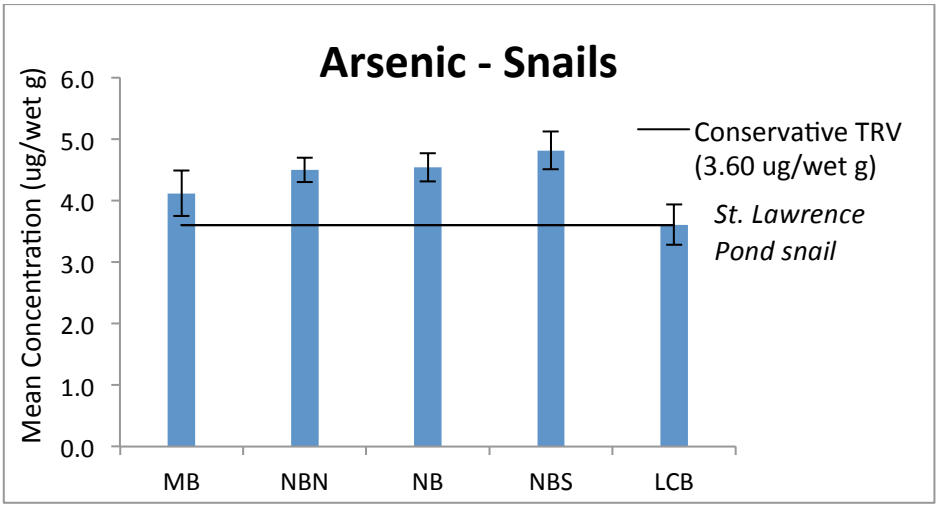

Figure 9: Average concentrations of arsenic in olive snails across beaches

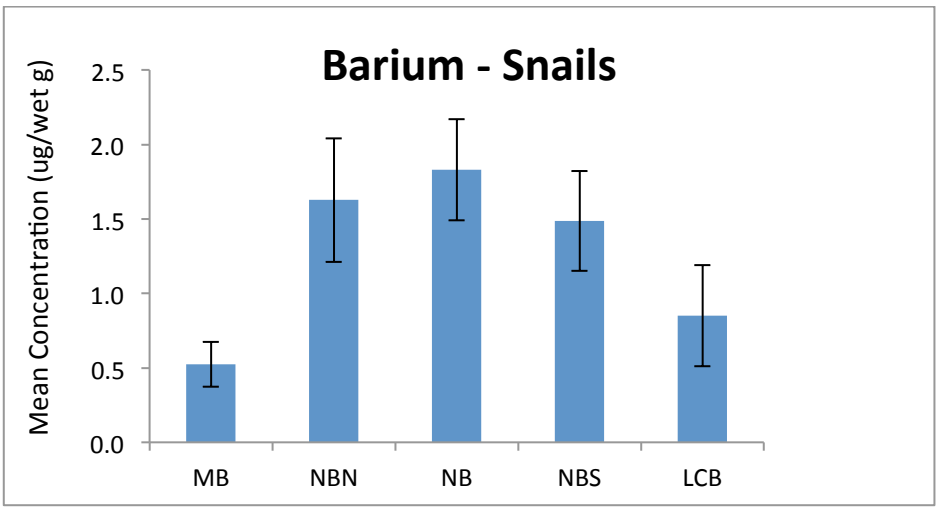

Figure 10: Average concentrations of barium in olive snails across beaches

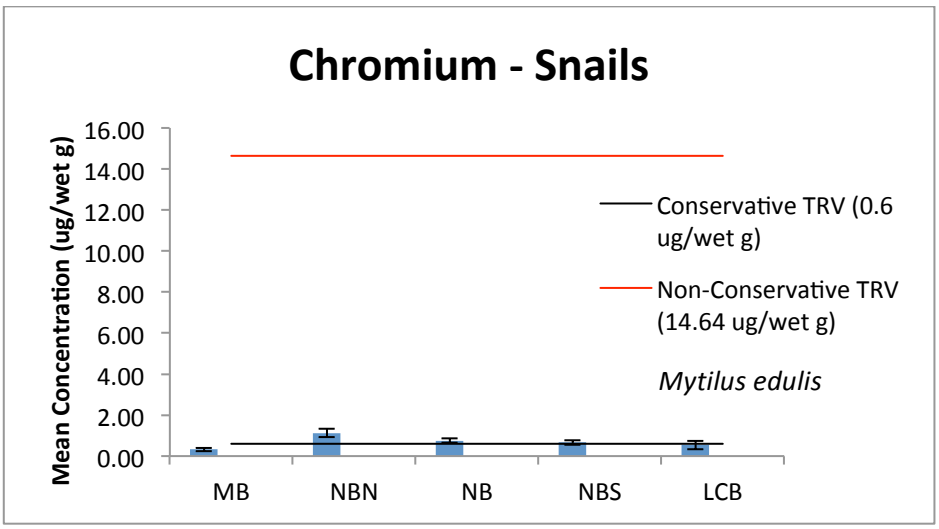

Figure 11: Average concentrations of chromium in olive snails across beaches

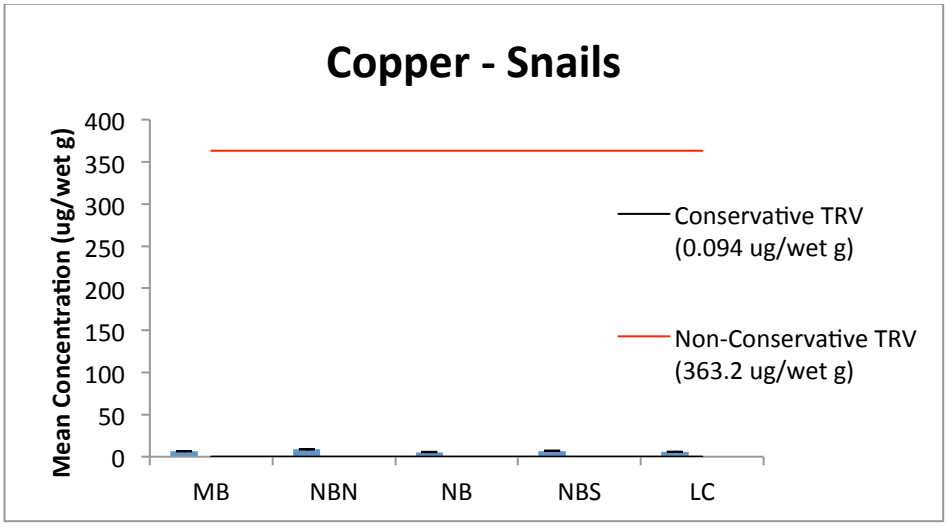

Figure 12: Average concentrations of copper in olive snails across beaches

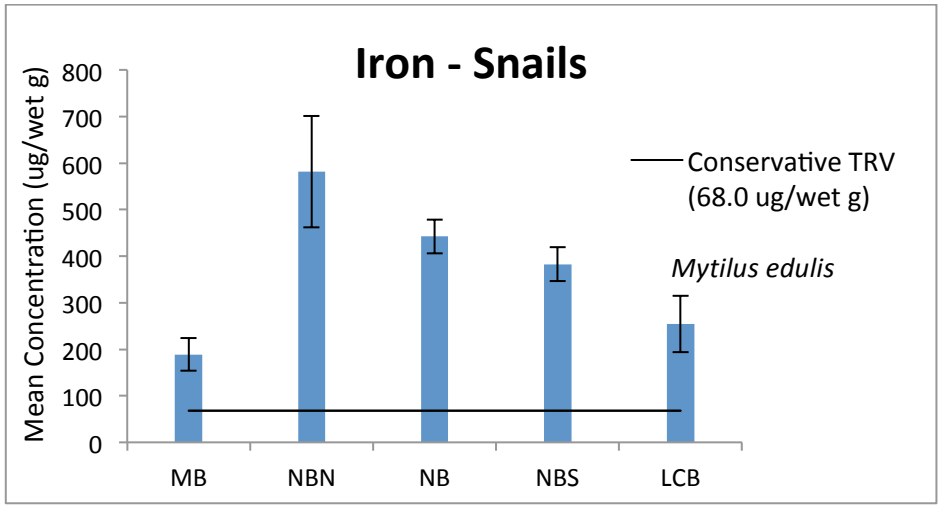

Figure 13: Average concentrations of iron in olive snails across beaches

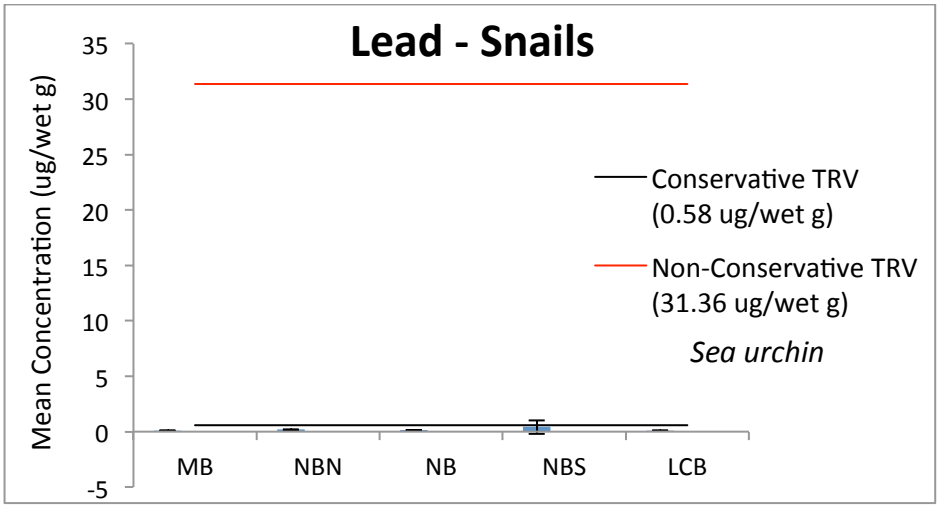

Figure 14: Average concentrations of lead in olive snails across beaches

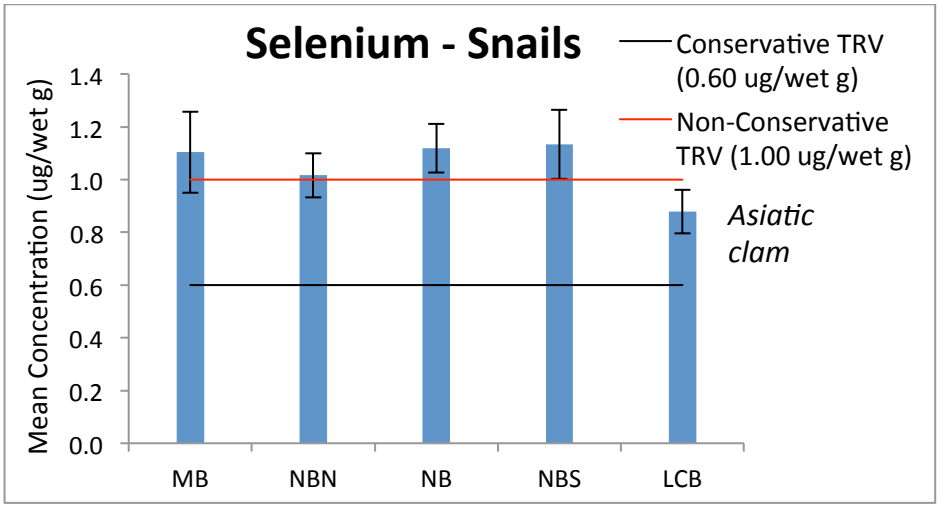

Figure 15: Average concentrations of selenium in olive snails across beaches

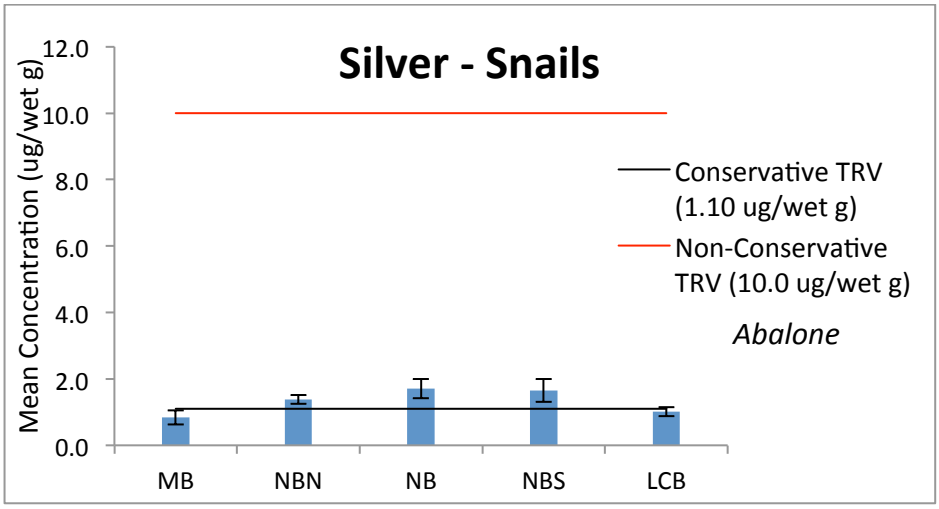

Figure 16: Average concentrations of silver in olive snails across beaches

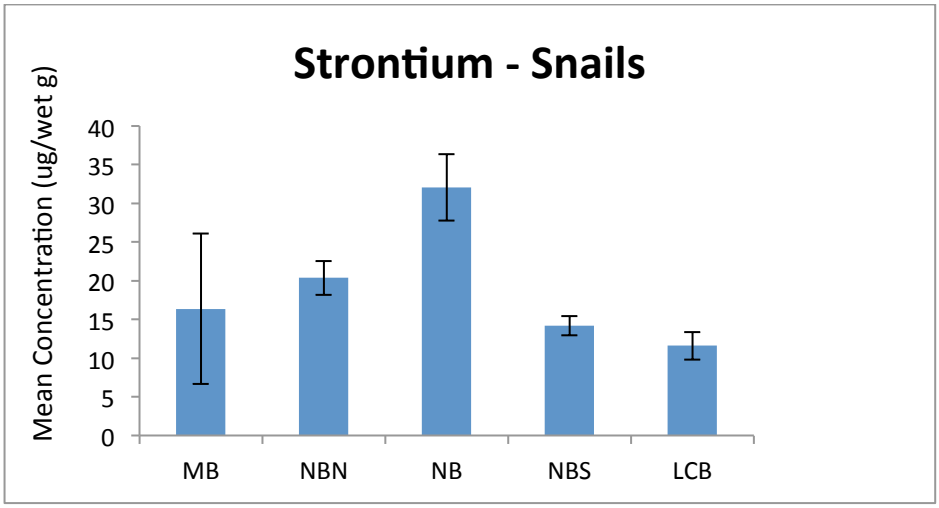

Figure 17: Average concentrations of strontium in olive snails across beaches

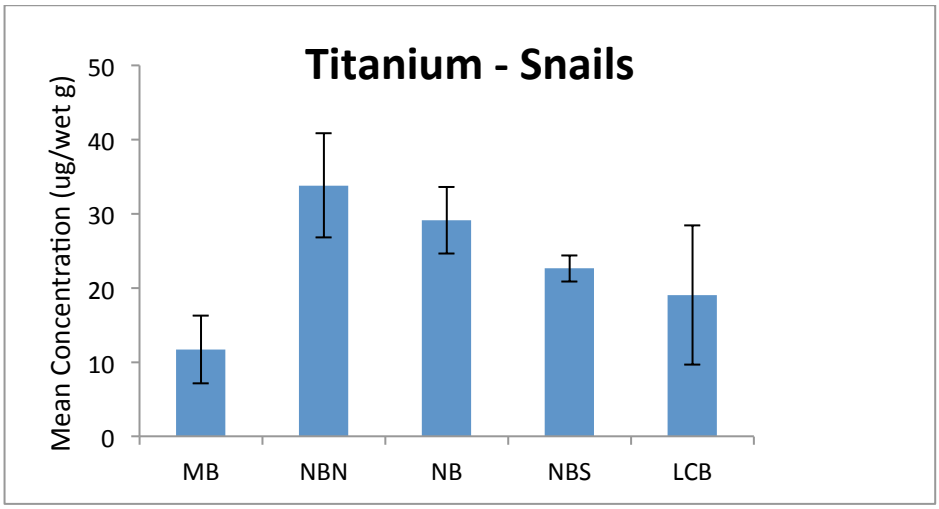

Figure 18: Average concentrations of titanium in olive snails across beaches

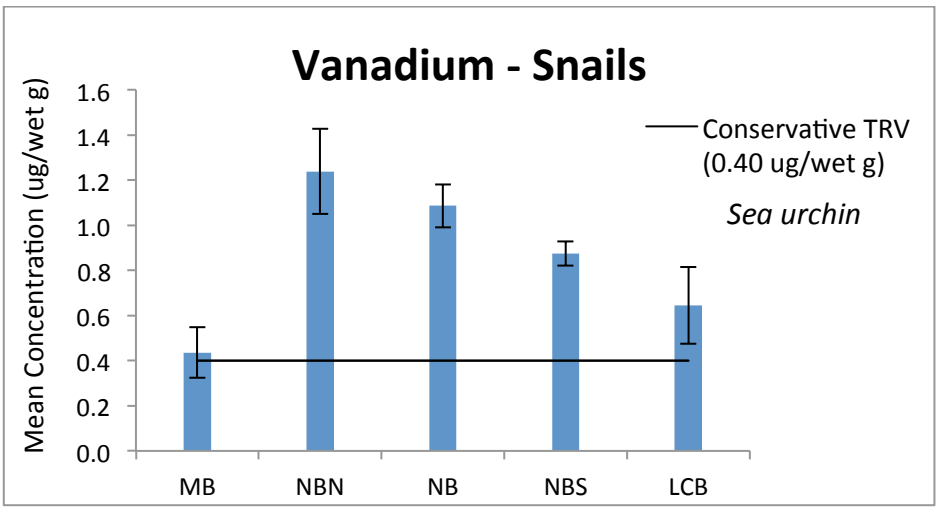

Figure 19: Average concentrations of vanadium in olive snails across beaches

Table 2: Average (across all samples) concentration (µg/wet g) of 38 chemicals detected in the tissues of animals from the Oregon coast. "Present in GP Effluent/Newport Effluent/MZ Sediment" columns indicate if the chemical was detected or estimated in previous comprehensive Aquatic Surveys (CH2M Hill 2010). Blank cells indicate chemicals that were not tested; "<< SSC" indicates concentrations in sediment that are less than 1/10 the Sediment Screening Criteria, based on US ACE and WA Department of Ecology Standards. "DEQ PPS" column indicates chemicals that are listed as compounds for reasonable potential analysis by Oregon Department of Environmental Quality for aquatic organisms.

	Subtidal				Intertidal					
Analyte	Dungeness crab	*Crangon* shrimp	Speckled sanddab	Rock scallop	*Mytilus* mussels	Olive snails	Present in GP Effluent	Present in City of Newport WWTP Effluent	Present in MZ Sediment	DEQ PPS
METALS										
Aluminum	6.052	20.992	3.229	18.527	85.107	253.56				Y
Antimony	n.d.	n.d.	n.d.	n.d.	n.d.	0.001				Y
Arsenic	10.006	0.897	0.706	1.276	4.374	4.314	Below RL	Below RL	Yes << SSC	Y
Barium	0.234	1.417	0.606	0.399	0.798	1.263				Y
Cadmium	1.081	0.686	0.006	16.164	0.94	2.02	Yes	Below RL	Yes << SSC	Y
Chromium	0.017	0.094	0.001	0.346	0.242	0.68	Yes	Yes	Yes << SSC	Y
Cobalt	0.185	0.004	n.d.	0.044	0.091	0.144				
Copper	17.27	7.919	0.313	0.990	0.327	6.746	Below RL	Below RL	Yes << SSC	Y
Iron	16.048	24.724	5.737	29.482	87.8	369.82				Y
Lead	n.d.	0.064	0.003	0.039	0.052	0.179	Yes	Below RL	Yes << SSC	Y
Manganese	0.639	0.855	2.843	0.714	1.592	5.976				Y
Mercury	0.134	0.015	0.005	0.018	0.01	0.011	Yes		Yes << SSC	Y
Molybdenum	0.059	0.038	0.038	0.064	0.083	0.261				
Nickel	0.132	0.106	0.002	0.142	0.376	0.624	Below RL	Below RL	Yes << SSC	Y
Selenium	1.165	0.212	0.189	0.507	0.524	1.05	Below RL	Yes	Estimated	Y
Silver	0.389	0.096	n.d.	n.d.	n.d.	1.319	Below RL	Below RL	Estimated	Y
Strontium	25.976	226.076	27.799	5.201	7.67	18.92				
Thallium	n.d	n.d.	0.002	n.d.	n.d.	n.d.	Below RL	Below RL		Y

Titanium	2.252	17.234	9.904	2.136	6.241	23.27				
Vanadium	0.191	0.778	0.172	0.350	2.595	0.856				
Zinc	34.972	12.554	9.794	16.036	18.855	14.07	Below RL	Yes	Yes << SSC	Y
ORGANICS										
2,4'-DDD	0.00006	n.d	n.d.	n.d	n.d	n.d.				
4,4'-DDD	0.0005	n.d	n.d.	n.d	n.d	n.d.				Y
4,4'-DDE	0.007	n.d	n.d.	n.d	n.d	n.d.				Y
Endosulfan-I	0.011	n.d	n.d.	n.d	n.d	n.d.				Y
Endosulfan-II	0.007	n.d	n.d.	n.d	n.d	n.d.				Y
Hexachloro-benzene	0.00009	n.d	n.d.	n.d	n.d	n.d.				Y
Oxychlordane	0.0007	n.d	n.d.	n.d	n.d	n.d.				Chlordane
PCB003	0.0002	n.d	n.d.	n.d	n.d	n.d.				
PCB018	0.0003	n.d	n.d.	n.d	n.d	n.d.				
PCB028	0.00006	n.d	n.d.	n.d	n.d	n.d.				
PCB037	0.00007	n.d	n.d.	n.d	n.d	n.d.				
PCB105	0.0005	n.d	n.d.	n.d	n.d	n.d.				
PCB119	0.002	n.d	n.d.	n.d	n.d	n.d.				
PCB138	0.00006	n.d	n.d.	n.d	n.d	n.d.				
PCB153	0.00008	n.d	n.d.	n.d	n.d	n.d.				
PCB170	0.00008	n.d	n.d.	n.d	n.d	n.d.				

Table 3: Subtidal species comparisons (Kruskal-Wallis and Wilcoxon Each Pair) of mean chemical concentration by sampling location for chemicals for which at least 1 sample exceeded the conservative TRVs or for which no TRVs could be determined. Highlighted chemicals showed significantly higher mean tissue concentrations in the mixing zone areas relative to at least one of the reference locations.

Species	Chemical	TRV?	Prob>ChiSq	Significant Relationships
Dungeness crab	2,4'-DDD	None	0.406	
	Aluminum	Exceeded Conservative	0.127	
	Arsenic	Exceeded Conservative	0.817	
	Barium	None	0.576	
	Cadmium	Exceeded Conservative	0.186	
	Molybdenum	None	0.608	
	Oxychlordane	None	0.406	
	Selenium	Exceeded Conservative	0.658	
	Silver	None	0.991	
	Strontium	None	0.170	
	Tin	None	0.676	
	Titanium	None	0.624	
	Vanadium	Exceeded Conservative	*0.011*	*(NMZ, SMZ, SBR) > NYR*
	Zinc	Exceeded Conservative	0.530	
Crangon shrimp	Aluminum	Exceeded Conservative	0.039	(NYR, MZ, SMZ, SBR) > NMZ
	Barium	None	0.201	
	Strontium	None	0.052	
	Tin	None	0.124	
	Titanium	None	0.047	(MZ, SMZ) > NMZ
	Vanadium	Exceeded Conservative	*0.0001*	*SBR > NMZ > MZ> SMZ > NYR*
	Zinc	Exceeded Conservative	*0.005*	*(NMZ, SMZ, SBR) > (NYR, MZ)*
Speckled sanddab	Arsenic	Exceeded Conservative	0.073	
	Barium	Exceeded Conservative	0.192	
	Iron	Exceeded Conservative	0.022	(NYR, NMZ, SMZ) > MZ; NYR > SBR
	Manganese	Exceeded Conservative	0.189	

	Selenium	Exceeded Conservative	0.008	NYR > (NMZ, SMZ); MZ > SMZ
	Strontium	None	0.040	MZ > NMZ
	Tin	None	*<0.001*	*(NYR, MZ, SMZ) > (NMZ, SBR)*
	Titanium	None	0.081	
Rock scallop	Aluminum	Exceeded Conservative	0.022	MZ > SR
	Barium	None	0.009	MZ > SR
	Cadmium	Exceeded Conservative	0.251	
	Cobalt	None	0.009	SR > MZ
	Molybdenum	None	0.009	SR > MZ
	Nickel	None	0.009	SR > MZ
	Strontium	None	0.251	
	Tin	None	0.602	
	Titanium	None	0.754	

Table 4: Intertidal species comparisons (Kruskal-Wallis and Wilcoxon Each Pair) of mean chemical concentration by sampling location for chemicals that exceeded the conservative TRVs or for which no TRVs could be determined. Highlighted chemicals showed significantly higher mean tissue concentrations in at least one mixing zone area relative to at least one of the reference locations.

Species	Chemical	TRV?	Prob>ChiSq	Significant Relationships
Mytilus mussels	Aluminum	Exceeded Conservative	0.0255	MB > NB
	Arsenic	Exceeded Conservative	0.0132	NB > (MB, SR)
	Barium	None	0.5655	
	Iron	Exceeded Conservative	0.0143	MB > NB
	Molybdenum	None	0.0221	MB > NB
	Strontium	None	0.0805	
	Tin	None	0.007	MB > NB, SR
	Titanium	None	0.0935	
	Vanadium	Exceeded Conservative	0.0081	MB, (SR > NB)
Olive snails	Aluminum	Exceeded Conservative	0.0005	(NBN, NB, NBS) > LC > MB
	Antimony	None	0.4060	
	Arsenic	Exceeded Conservative	0.0038	(MB, NBN, NB, NBS) > LC
	Barium	None	0.0020	(NBN, NB, NBS) > (MB, LC)
	Chromium	Exceeded Conservative	0.0012	NBN > (NB, NBS, LC, MB)
	Copper	Exceeded Conservative	0.0030	NMZ > (NYR, SBR, SMZ); NYR > MZ
	Iron	Exceeded Conservative	0.0003	(NBN, NB, NBS) > (MB, LC); NBN > NBS
	Lead	Exceeded Conservative	0.0006	(NBN, NB, NBS) > (MB, LC); NBN > NB
	Molybdenum	None	0.1067	
	Selenium	Exceeded Less-Conservative	0.0181	(MB, NBN, NB, NBS) > LC
	Silver	Exceeded Conservative	0.0008	(NBN, NB, NB) > (MB, LC)
	Strontium	None	0.0023	NB > NBN > (MB, NBS, LC)
	Tin	None	0.3975	
	Titanium	None	0.0022	(NBN, NB) > (NBS, LC) > MB
	Vanadium	Exceeded Conservative	0.0003	(NBN, NB, NBS) > (MB, LC); (NBN, NB) > NBS

Table 5: Subtidal organism mean tissue concentrations for chemicals that were significantly higher in animals from the G-P Primary Outfall Area than at least one reference area. Gray highlights indicate locations where average tissue chemical concentrations exceeded a conservative TRV. Effect Type indicates tissues and biological endpoints used for the TRV. Conservative TRVs were mostly NOED (No Observable Effect Dose) studies. Sites where individual samples had concentrations higher than the TRV but average values did not exceed are marked with an asterisk. "Present in Effluents?" column reports on sampling results from the GP-Toledo Mill Effluent, the City of Newport Waste Water Treatment Plant, and Nye Creek from the previous comprehensive Aquatic Surveys (CH2M Hill 2010).

					Conservative Measure		Less Conservative Measure		
Species	**Chemical**	**Site**	**Mean Concentration (µg/wet g)**	**ST DEV**	**TRV (µg/wet g)**	**NOED Effect Type**	**TRV (µg/wet g)**	**Effect Type**	**Present in Effluents?**
Dungeness crab	Vanadium	NYR	0.07	0.02	0.6	whole body, mortality shore crab Miramand et al. 1981	NO TRV	--	Not Tested
		NMZ	0.17	0.08	0.6		NO TRV		
		MZ	0.12	0.03	0.6		NO TRV		
		SMZ	0.17	0.12	0.6		NO TRV		
		SBR*	0.42	0.22	0.6		NO TRV		
Crangon shrimp	Vanadium	NYR	0.20	0.02	0.50	whole body, mortality shrimp Miramand et al. 1981	3.40	digestive tract, mortality shrimp Miramand et al. 1981	Not Tested
		NMZ	0.31	0.03	0.50		3.40		
		MZ	*0.74*	*0.18*	*0.50*		3.40		
		SMZ	0.42	0.07	0.50		3.40		
		SBR	*2.22*	*0.93*	*0.50*		3.40		
	Zinc	NYR	11.33	0.46	12.7	whole body, mortality crayfish Mirenda 1986	17.80	muscle tissue, mortality	GP: < RL WWTP: Yes Nye: < RL
		NMZ	*12.88*	*0.59*	*12.7*		17.80		
		MZ	11.72	0.98	12.7		17.80		
		SMZ	*13.07*	*0.63*	*12.7*		17.80		
		SBR	*13.77*	*1.13*	*12.7*		17.80		
Speckled sanddab	Tin (Sn)	NYR	0.48	0.85	NO TRV	--	NO TRV	--	Reported at MDL for all
		NMZ	0.00	0.00	NO TRV		NO TRV		
		MZ	0.21	0.28	NO TRV		NO TRV		
		SMZ	0.11	0.05	NO TRV		NO TRV		
		SBR	0.01	0.01	NO TRV		NO TRV		

Rock Scallops	Aluminum	MZ*	22.40	7.91	31	whole body, mortality mussels St. Jean et al. 2003	NO TRV	--	Not Tested
		SR	15.24	1.18	31		NO TRV		
	Barium	MZ	0.505	0.08	NO TRV	--	NO TRV	--	Not Tested
		SR	0.311	0.04	NO TRV		NO TRV		

Table 6: Intertidal organism mean tissue concentrations for chemicals that were significantly higher in animals from the Nye Beach Area than at least one reference area. Gray highlights indicate sampling locations where tissue chemical concentrations exceeded a conservative TRV. Bold text in highlighted cells indicates sampling locations where tissue chemical concentrations also exceeded a less conservative TRV. Effect Type indicates tissues and biological endpoints for the TRV. All conservative TRVs were NOED (No Observable Effect Dose) studies. Stations where individual samples had analyte concentrations higher than the TRV but averaged values did not exceed are marked with an asterisk. "Present in Effluents?" column reports on sampling results from the GP-Toledo Mill Effluent, the City of Newport Waste Water Treatment Plant, and Nye Creek from the previous comprehensive Aquatic Surveys (CH2M Hill 2010).

					Conservative Measure		Less Conservative Measure		
Species	**Chemical**	**Site**	**Mean Concentration (µg/wet g)**	**STDEV**	**TRV (µg/wet g)**	**NOED Effect Type**	**TRV (µg/wet g)**	**Effect Type**	**Present in Effluents?**
Mytilus mussels	Arsenic	*MB*	*4.12*	*0.30*	*3.60*	whole body mortality *Mytilus*; St Jean et al. 2003	NO TRV	--	Below the Reporting Limit for all
		NB	*4.97*	*0.50*	*3.60*		NO TRV		
		SR	*4.03*	*0.25*	*3.60*		NO TRV		
Olive snails	Aluminum	MB	95.92	26.94	250.00	digestive gland mortality pond snail Desouky 2006	NO TRV	--	Not Tested
		NBN	*386.14*	*98.03*	*250.00*		NO TRV		
		NB	*326.06*	*37.32*	*250.00*		NO TRV		
		NBS	*291.33*	*19.78*	*250.00*		NO TRV		
		LC	169.36	51.24	250.00		NO TRV		
	Arsenic	*MB*	*4.12*	*0.37*	*3.60*	whole body, mortality pond snail Spehar et al. 1980	NO TRV	--	Below the Reporting Limit for all
		NBN	*4.50*	*0.20*	*3.60*		NO TRV		
		NB	*4.54*	*0.23*	*3.60*		NO TRV		
		NBS	*4.81*	*0.31*	*3.60*		NO TRV		
		LC	*3.61*	*0.33*	*3.60*		NO TRV		
	Barium (Ba)	MB	0.52	0.15	NO TRV	--	NO TRV	--	Not Tested
		NBN	1.63	0.42	NO TRV		NO TRV		
		NB	1.83	0.34	NO TRV		NO TRV		
		NBS	1.49	0.34	NO TRV		NO TRV		
		LC	0.85	0.34	NO TRV		NO TRV		

Species	Chemical	Site	Mean Concentration (µg/wet g)	STDEV	Conservative Measure TRV (µg/wet g)	Conservative Measure NOED Effect Type	Less Conservative Measure TRV (µg/wet g)	Less Conservative Measure Effect Type	Present in Effluents?
Olive snails	Chromium	MB	0.32	0.09	0.6	digestive tract biochemical *Mytilus edulis* Barmo et al. 2011	14.64	muscle, biochemical *Mytilus edulis* Emmanouil, et al. 2007	GP: Yes WWTP: at RL Nye: < RL
		NBN	*1.12*	*0.20*	*0.6*		3.20		
		NB	*0.75*	*0.10*	*0.6*		3.20		
		NBS	*0.66*	*0.12*	*0.6*		3.20		
		LC	0.54	0.21	0.6		3.20		
	Copper	*MB*	*6.61*	*0.44*	0.094	whole body mortality (LD20) pond snail Das & Khangarot 2011	363.2	soft tissue mortality apple snail Hoang et al. 2011	Below the Reporting Limit for all
		NBN	*9.00*	*1.57*	0.094		363.2		
		NB	*5.38*	*0.87*	0.094		363.2		
		NBS	*6.88*	*1.43*	0.094		363.2		
		LC	*5.86*	*0.51*	0.094		363.2		
	Iron	*MB*	*188.82*	*35.43*	*68.00*	whole body, mortality mussel St Jean et al. 2003	NO TRV	--	Not Tested
		NBN	*581.04*	*119.65*	*68.00*		NO TRV		
		NB	*442.50*	*36.46*	*68.00*		NO TRV		
		NBS	*382.66*	*36.16*	*68.00*		NO TRV		
		LC	*254.08*	*60.45*	*68.00*		NO TRV		
	Lead	MB	0.08	0.01	0.58	soft tissue, mortality sea urchin Radenac et al. 2001	31.36	LOED, whole body, survival sea urchin Radenac et al. 2001	GP: Yes WWTP: < RL Nye: < RL
		NBN	0.18	0.03	0.58		31.36		
		NB	0.14	0.01	0.58		31.36		
		NBS*	0.41	0.62	0.58		31.36		
		LC	0.09	0.01	0.58		31.36		
	Selenium	***MB***	***1.10***	***0.15***	***0.60***	soft tissues, physiological clam Fournier et al. 2006	***1.00***	NOED soft tissues, physiological clam Fournier et al. 2006	GP: < RL WWTP: Yes Nye: Yes
		NBN	***1.02***	***0.08***	***0.60***		***1.00***		
		NB	***1.12***	***0.09***	***0.60***		***1.00***		
		NBS	***1.13***	***0.13***	***0.60***		***1.00***		
		LC	*0.88*	*0.08*	*0.60*		1.00		

Species	Chemical	Site	Mean Concentration (µg/wet g)	STDEV	Conservative Measure TRV (µg/wet g)	Conservative Measure NOED Effect Type	Less Conservative Measure TRV (µg/wet g)	Less Conservative Measure Effect Type	Present in Effluents?
Olive snails	Silver	MB	0.84	0.22	1.10	soft tissues, growth abalone Huang et al. 2010	10.00	soft tissue, reduced reproduction limpet Nelson et al. 1983	Below the Reporting Limit for all
		NBN	*1.38*	*0.13*	*1.10*		10.00		
		NB	*1.71*	*0.29*	*1.10*		10.00		
		NBS	*1.66*	*0.35*	*1.10*		10.00		
		LC	1.01	0.14	1.10		10.00		
	Strontium (Sr)	MB	16.37	9.71	NO TRV	--	NO TRV	--	Not Tested
		NBN	20.37	2.20	NO TRV		NO TRV		
		NB	32.09	4.31	NO TRV		NO TRV		
		NBS	14.19	1.24	NO TRV		NO TRV		
		LC	11.59	1.80	NO TRV		NO TRV		
	Titanium (Ti)	MB	11.69	4.55	NO TRV	--	NO TRV	--	Not Tested
		NBN	33.85	7.00	NO TRV		NO TRV		
		NB	29.13	4.48	NO TRV		NO TRV		
		NBS	22.62	1.75	NO TRV		NO TRV		
		LC	19.06	9.40	NO TRV		NO TRV		
	Vanadium	*MB*	*0.44*	*0.11*	*0.40*	whole body mortality echinoderms Miramand et al. 1982	NO TRV	--	Not Tested
		NBN	*1.24*	*0.19*	*0.40*		NO TRV		
		NB	*1.09*	*0.10*	*0.40*		NO TRV		
		NBS	*0.88*	*0.05*	*0.40*		NO TRV		
		LC	*0.65*	*0.17*	*0.40*		NO TRV		

MORPHOMETRICS

Morphometric data, averaged over the composite samples for each species and site, are presented in Table **7**. Grand average carapace width for Dungeness was 156.9 mm; speckled sanddab had an average length of 64.96 mm and an average weight of 2.17 g; *Mytilus* averaged 33.74 mm and weighed an average of 4.13 g; *Crangon* composite weight averaged 40.96 g; and olive snail composites averaged 5.89 g. There were significant differences in size among sites for all invertebrate species, but there were no significant differences in sanddab sizes among sites (Table **8**). Dungeness crabs collected from both reference locations were larger than those collected from the mixing zone. Within the mixing zone, NMZ crabs were larger than SMZ crabs. Larger mussels were collected from Nye Beach than from the reference areas, with Seal Rock mussels larger than Moolack Beach mussels. Similarly, olive snails collected from Nye Beach and Lost Creek were larger than those collected from Moolack Beach.

Table 7: Morphometric summary. Values are the average of all samples per site. Mean size represents carapace width for Dungeness and total length for sanddabs and *Mytilus*. Mean weight represents composite weights for *Crangon* and olive snails and mean individual weights for sanddab and *Mytilus*.

Species	Site	Mean size (mm)	STDEV	Mean Weight (g)	STDEV
Dungeness crab	NYR	161.00	13.94	.	.
	NMZ	155.60	13.35	.	.
	MZ	153.20	14.72	.	.
	SMZ	149.30	12.70	.	.
	SBR	165.40	13.13	.	.
Crangon shrimp	NYR	.	.	39.38	1.19
	NMZ	.	.	48.72	0.63
	MZ	.	.	18.08	2.01
	SMZ	.	.	40.00	1.08
	SBR	.	.	58.64	1.66
Speckled sanddab	NYR	62.44	6.84	1.80	0.73
	NMZ	64.50	8.31	2.16	1.04
	MZ	64.85	8.10	2.24	1.06
	SMZ	62.63	5.00	1.84	0.44
	SBR	71.10	7.40	2.88	1.15
Mytilus mussels	MB	22.68	4.79	1.25	1.07
	NB	39.92	9.81	7.15	5.93
	SR	38.62	6.99	3.99	2.06
Olive snails	MB	.	.	3.90	0.59
	NBN	.	.	5.45	0.52
	NB	.	.	6.79	0.71
	NBS	.	.	6.23	0.60
	LC	.	.	7.07	0.62

Table 8: Results of Kruskal-Wallis and Wilcoxon Each Pair Comparisons investigating differences in size or weight of composite samples between sites. Significant relationship column indicates significant differences in size or weight between sites.

Species	Mean size (mm)	Significant Relationships	Mean Weight (g)	Significant Relationships
Dungeness crab	<0.0001	NYR > MZ, SMZ SBR > NMZ, MZ, SMZ NMZ > SMZ	.	.
Crangon shrimp	.	.	0.0002	SBR > NMZ > NYR, SMZ > MZ
Speckled sanddab	0.0826	.	0.0771	.
Mytilus mussels	<0.0001	NB, SR > FC	<0.0001	NB > SR > MB
Olive snails	.	.	0.001	NB, LC > NBN, NBS, > MB

Discussion

The goal of this study was to determine if accumulation of effluent-borne pollutants could be detected in resident coastal species and be related to discharge from the G-P outfall. A second objective was to evaluate the potential organismal and human health impacts related to elevated levels (if present).

In most subtidal organisms (fish, crabs, shrimp) there were few compounds elevated to levels that might approach concern, and there was no relationship between elevated concentrations of any contaminants and proximity to the G-P outfall. In metals that showed different concentrations among collection sites, animals from either the north (fish) or south (crabs and shrimp) reference site exhibited the highest concentrations. Within the mixing zone, in only one case (shrimp, vanadium) the central collection station (closest to the outfall) had higher concentrations than the north or south mixing zone stations; but still, the South Beach Reference station had even higher concentrations. For scallops, only 5 metals showed differences between sites. For three metals, scallops from Seal Rock had higher concentrations, and for two metals (aluminum and barium) scallops from the Mixing Zone had higher concentrations.

In mussels collected from intertidal locations only one contaminant (arsenic) exhibited elevated concentrations from the central (Nye Beach) collection site relative to the reference sites; however concentrations at all three sites exceeded NOED levels (St. Jean et al. 2003). For olive snails collected from local beaches there were several metals for which the Nye Beach area had the highest concentrations relative to reference areas north and south. However, for the intertidal organisms we are not able to conclusively relate higher concentrations to the G-P outfall as other factors (City of Newport WWTP outfall, Nye Creek discharge, and non-point-source runoff) could be contributing to contaminant loads in these on-shore organisms.

The original goal of this work was to determine whether there were contaminant accumulation issues associated with the G-P outfall site. As noted in the proposal and introduction to this report, if warranted, Tier 2 studies would identify the specific effluent-related parameters responsible for contamination (Diagnosis of Causes) and suggest a plan for assessing overall risks to ecosystem and public health. While we did not detect evidence of any trends in accumulation of contaminants of concern related to the G-P outfall, the authors acknowledge the City of Newport's and the public interest in understanding how general contaminant concentrations in organisms collected for this study compare to human health concerns and relative to historical data (where available). As such, we have provided a "Broader Context" appendix (Appendix 1) that discusses the findings for mussels as compared to Mussel Watch data (citation?) and for crabs as compared to FDA limits for shellfish consumption.

Below we discuss the findings for each group of contaminants and organisms that showed differences among sites, although few show any relation to the G-P Outfall.

ORGANICS

Organic compounds were only detected in Dungeness crabs. Of the 19 organic compounds detected in crabs, only two were found in more than one composite sample.

The compound 4,4'-DDE, found in samples from all sites, is a breakdown product of the pesticide DDT (ATSDR 2002). Although it was broadly detected, it was measured in concentrations far below levels of concern. Values of 4,4'-DDE ranged from 0.0026 to 0.0254 µg/g wet weight. The toxicity reference value for effects on crabs is 0.75 µg/g wet weight, and the FDA regulatory level for human ingestion is 5 µg/g (FDA 2001a). This means that 4,4'-DDE levels in crab are 200-2000 times lower than FDA regulatory levels.

Two other DDT breakdown products (2,4'-DDE and 4,4'-DDD) were each found in a single crab composite sample. The concentration of 4,4'-DDD did not exceed the TRV for that derivative. There is no TRV for 2,4'-DDE, but it did not exceed the TRV for other DDT derivatives.

Hexochloro-benzene, detected in only two crab samples, was used as a seed-treatment fungicide with use voluntarily cancelled in 1984 (ATSDR 2002). It is reasonably anticipated to be a human carcinogen (NIH 2011); however, the detected concentrations in this study were 10,000 times less than the toxicity reference value for effects on crabs themselves.

PBDEs

PBDEs were not detected in any of the collected organisms. This is in contrast to findings offshore of urban areas in California (Bay et al. 2011) and in Canada, associated with both urbanization and run-off near pulp and paper mills (Ikonomou et al. 2006).

METALS

The 22 detected metals varied widely in tissue concentrations across organisms, and there was little discernable pattern related to the G-P outfall pipe or mixing zone. Thus it appears these compounds are broadly distributed offshore of Newport, Oregon, and are differentially accumulating in various animal tissues. In many cases, a metal that was continued through the screening process because it was higher in the mixing zone than at one reference location was found in even higher concentrations in organisms from the other reference location.

In the subtidal-collected mobile organisms (crabs, shrimp, and sanddabs), where differences in metal concentrations were detected among sites, aluminum, vanadium and zinc concentrations were highest in crustaceans collected off South Beach (SBR) and iron, selenium, and tin were highest in fish from north of Yaquina Head (NYR). In subtidal-collected scallops, aluminum and barium were higher in scallops collected from the Mixing Zone and cobalt, molybdenum, and nickel were higher in scallops collected near Seal Rock. Other detected metals did not show differences among sites.

Mussels and snails (both collected onshore) showed higher concentration of certain metals from the central collection site relative to both reference locations. While it is possible that the higher metal loads carried by these mussels and snails from the 'mixing zone' area is because of the G-P outfall, it is also quite possible that these elevated levels are due to their proximity to the developed Newport Beach area, including the City of Newport Waste Water Treatment Plant effluent and Nye Creek. Animals from the north and south reference locations are likely less impacted by urban point and non-point sources of contamination. Some metals exceeded published TRVs in mussels and snails collected from all on-shore locations, namely arsenic, iron, and selenium. In mussels, arsenic was found in higher concentration from the central collection

location, but average concentrations in mussels from all sites exceeded the conservative TRV (3.6 µg/g as published in the ERED database). However, detected concentrations of 3.641 to 5.543 µg/g in mussels collected near Newport are well within the range of concentrations found in mussels exposed to treated lumber (4.74 – 11.78 µg/g) where no significant difference in condition index was found between experimental and control mussels (Adler-Ivanbrook & Breslin 1999). For snails, arsenic concentrations were within the range of snail species tested by Spehar et al. (1980) who found that accumulation of arsenic 99 times greater than the water concentration had no significant effect on the survival of snails after 28 days of exposure. Thus, although these 'elevated' concentrations are found broadly across sites in the area, they may not be having significant adverse effects on the mussel and snail populations.

Recommendations for Future Studies

Mussels and snails collected from the beach near the mixing zone were found to contain significantly higher concentrations of some trace metals relative to reference locations; thus, OSU recommends follow-up studies on these organisms to clarify the potential source of contamination (Tier 2; Diagnosis of Causes). In this case, it first needs to be determined whether the trace metals are likely coming from the G-P outfall or from other point or non-point sources associated with urbanization. We recommend that if this pattern is of further interest to the City of Newport, that additional sampling and analysis of olive snails and mussels be conducted at the previously investigated sites, plus additional urbanized and non-urbanized locations along the Oregon coast. Additional areas might include Lincoln City and Florence, two moderately large urban areas in reasonable proximity to Newport. It is recommended that study be undertaken in the same season as the 2012 study (August – October) as researchers attempted a second snail collection in February 2013, but olive snails were absent from the beaches where they had been collected in the summer. This could likely be accomplished with a single sampling event, with composite samples again sent to Physis for analytical processing.

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Appendix 1

Broader Context

MUSSELS

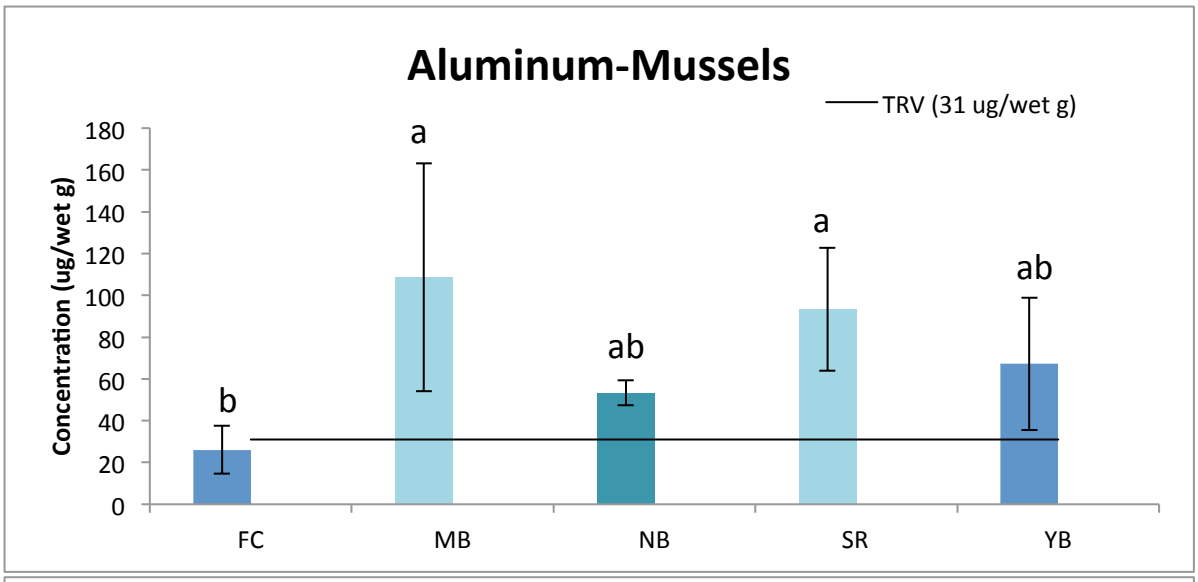
Mussel Watch (http://ccma.nos.noaa.gov/about/coast/nsandt/musselwatch.aspx) is a contaminant monitoring program developed to analyze chemical and biological contaminant trends in bivalve tissues collected at over 300 coastal sites from 1986 to present. Mussel Watch data are available for download at the NS&T Program Download Page: http://ccma.nos.noaa.gov/about/coast/nsandt/download.aspx.

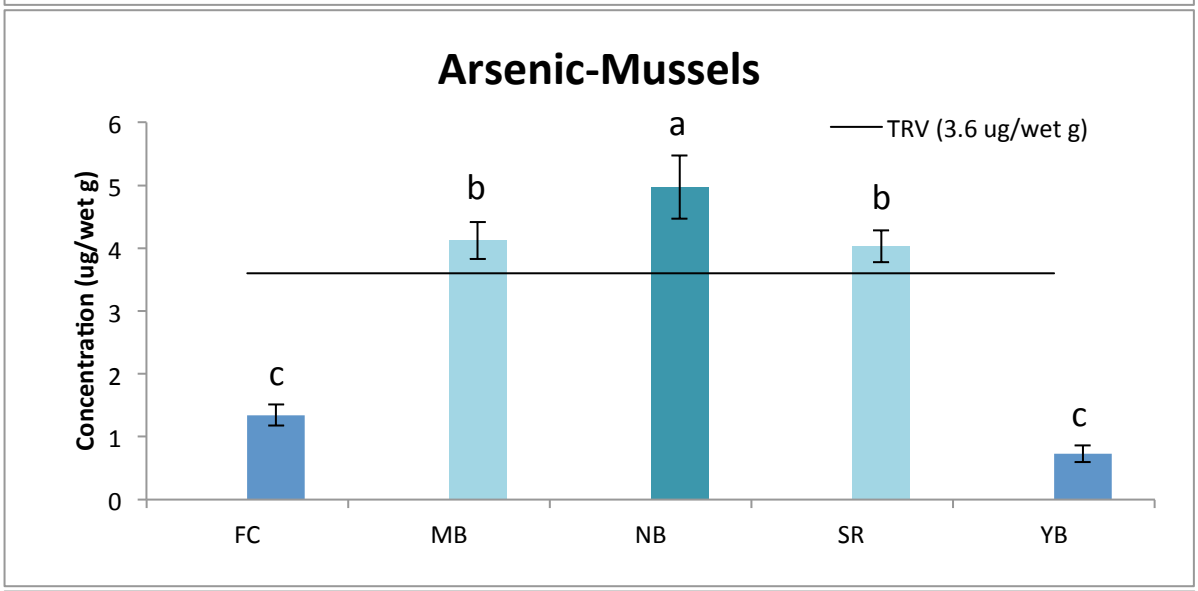
Mussel Watch data for the west coast were downloaded from the NS&T site and data from relevant Oregon sites were extracted from the dataset. These sites were YBFC (Yaquina Bay, Fogarty Creek), YBYH (Yaquina Bay, Yaquina Head), and YBOP (Yaquina Bay, Onnetta Point). The most recent sampling event for any of these sites was 2007 (or at least that is the most recent data available online). This creates a substantial caveat for these comparisons; there is a five-year gap in collection dates between the Mussel Watch data and our recent collections. Starting in 2001, ICPMS was used as the analytical method; thus, we only used data from the 2000s as comparisons to data collected for the City of Newport project so that we were using comparably collected and analyzed data. The Yaquina Head site was not sampled after 1991, so we did not use YH as a comparison site. Using non-parametric Kruskal-Wallis tests, we compared the means of samples collected at a site in this study (multiple replicate in a single sampling event) to the average of 4 years of data from 2001 to 2007 (samples were collected every other year in the Mussel Watch program) at Fogerty Creek (FC) and Onnetta Point in Yaquina Bay (YB). Although the data are not normally distributed, we also conducted one-way ANOVA tests to determine if there were differences between any sample sites, and Tukey's comparisons to determine where the differences exist, as ANOVA is generally robust to non-normality and we were more interested in specific site differences than the ranks provided by the Kruskal-Wallis tests. Although significant differences existed between any pair of sites for almost every analyte, overall only arsenic, cadmium, and selenium exhibited higher concentrations in samples from this study than the historical Mussel Watch data (Table A1-1). As previously noted in the body of this report, only arsenic showed higher concentrations in mussels from the central collecting location (Nye Beach) relative to the north and south reference areas. Also included in Table A1-1 are the conservative TRV values previously defined and FDA limits for molluscan shellfish. **Tissue concentrations of metals of concern in mussels were not approaching levels for human health concern from molluscan shellfish consumption.** The following graph series plots the City of Newport data (teal bars; Moolack Beach, Nye Beach, Seal Rock) against historic mussel watch data (blue bars; Fogerty Creek, Yaquina Bay-Onnetta Point). Letters over the bars indicate results from the Tukey statistical tests. Bars that have the same letter over them are statistically the same; bars with different letters are statistically different. Bars that have 2 letters are not different from either group. No letters indicates no significant differences among sites. Where tissue concentrations exceeded TRV values, the TRV concentration is plotted on the graph. The figure legend shows the experimental organism on which the TRV is based. FDA limits were of such higher value than any tissue concentrations that including those levels on the graph would have made it impossible to see the data.

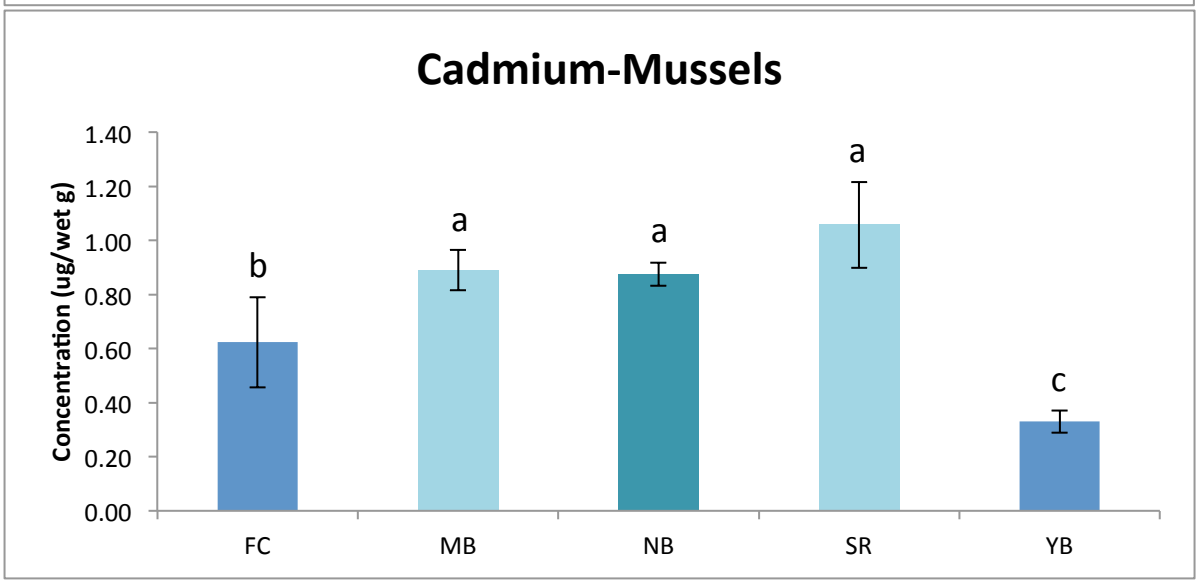
Table A1-1: Comparison of metal concentrations detected in mussels collected for this study as compared to recent Mussel Watch collections near the North Reference site and in Yaquina Bay. Concentrations in red text exceeded the conservative toxicity reference vale. No concentrations detected in mussels exceeded U.S. Food and Drug Administration limits.

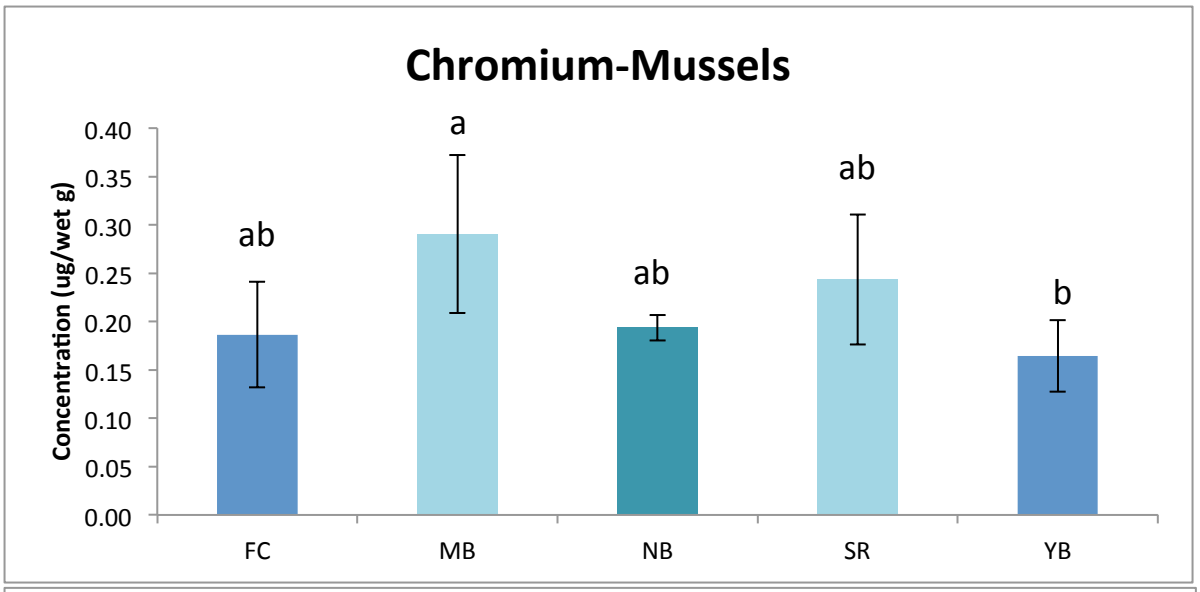
Chemical	Site	Average Conc. (ug/g)	StDev	TRV (ug/g)	FDA (ug/g)	K-W p-value (adj)	ANOVA p-value	Site Differences (Based on Tukey Comparisons)
Aluminum (Al)	*FC*	26.06	11.39	31	None	p = 0.007	p = 0.010	No differences within this study, not different from Mussel Watch, most exceed TRV
	MB	108.64	54.55					
	NB	53.30	5.90					
	SR	93.38	29.49					
	YB	67.16	31.62					
Arsenic (As)	*FC*	1.34	0.17	3.6	86	p = 0.001	p < 0.0001	Nye Beach higher than references, all exceed TRV and higher than Mussel Watch
	MB	4.12	0.30					
	NB	4.97	0.50					
	SR	4.03	0.25					
	YB	0.72	0.13					
Cadmium (Cd)	*FC*	0.62	0.17	6.45	4	p = 0.002	p < 0.0001	No differences within this study, all higher than Mussel Watch
	MB	0.89	0.07					
	NB	0.87	0.04					
	SR	1.06	0.16					
	YB	0.33	0.04					
Chromium (Cr)	*FC*	0.19	0.05	0.8	None	p = 0.030	p = 0.023	No differences among sites for this study; this study similar to Mussel Watch sites
	MB	0.29	0.08					
	NB	0.19	0.01					
	SR	0.24	0.07					
	YB	0.16	0.04					
Copper (Cu)	*FC*	1.25	0.25	3.4	None	p = 0.016	p = 0.001	Nye Beach higher than reference sites for this study; overall this study similar to Mussel Watch sites
	MB	1.21	0.08					
	NB	1.56	0.18					
	SR	1.20	0.12					
	YB	1.01	0.13					
Iron (Fe)	*FC*	57.44	22.73	68	None	p = 0.016 (p = 0.016)	p = 0.031	No differences between this study and Mussel Watch, most exceed TRV
	MB	113.66	45.53					
	NB	57.42	3.25					
	SR	92.32	23.05					
	YB	98.40	35.77					
Lead (Pb)	*FC*	0.08	0.02	*4.4*	1.7	p = 0.094 (p = 0.093)	p = 0.096	No differences among 2012 sites or compared to Mussel Watch
	MB	0.05	0.01					
	NB	0.06	0.00					
	SR	0.05	0.01					
	YB	0.06	0.02					

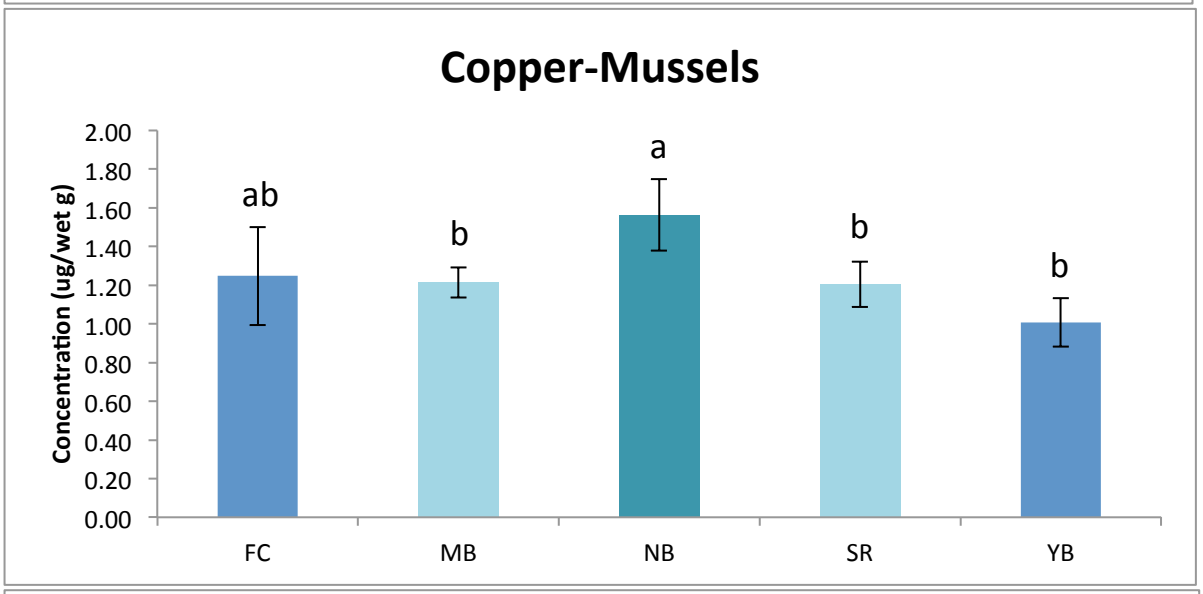
Chemical	Site	Average Conc. (ug/g)	StDev	TRV (ug/g)	FDA (ug/g)	K-W p-value (adj)	ANOVA p-value	Site Differences (Based on Tukey Comparisons)
Manganese (Mn)	*FC*	0.99	0.20	9.6	None	p = 0.002	p < 0.0001	Yaquina Bay Mussel Watch values highest, no differences among other sites
	MB	2.23	0.61					
	NB	1.19	0.08					
	SR	1.36	0.21					
	YB	4.15	1.94					
Mercury (Hg)	*FC*	0.02	0.01	1.12	1	p = 0.037 (p = 0.024)	p = 0.062	No differences among 2012 sites or compared to Mussel Watch
	MB	0.01	0.00					
	NB	0.01	0.00					
	SR	0.01	0.00					
	YB	0.01	0.00					
Nickel (Ni)	*FC*	0.35	0.06	*79**	80	p = 0.019 (p = 0.019)	p = 0.001	No differences among 2012 sites or FC Mussel Watch; 2012 sites higher than Yaquina Bay
	MB	0.42	0.05					
	NB	0.37	0.04					
	SR	0.34	0.05					
	YB	0.25	0.04					
Selenium (Se)	*FC*	0.39	0.10	*0.6**	None	p = 0.005	p < 0.0001	No differences within this study, all higher than Mussel Watch
	MB	0.54	0.04					
	NB	0.51	0.04					
	SR	0.52	0.06					
	YB	0.32	0.03					
Silver (Ag)	*FC*	0.01	0.02	None	None	p = 0.051 (p = 0.003)	p = 0.112	No differences among 2012 sites or compared to Mussel Watch
	MB	0.00	0.00					
	NB	0.00	0.00					
	SR	0.00	0.00					
	YB	0.01	0.01					
Tin (Sn)	*FC*	0.00	0.01	None	None	p = 0.001	p < 0.0001	Nye Beach not different from Mussel Watch, reference sites for this study highest
	MB	0.12	0.02					
	NB	0.05	0.04					
	SR	0.09	0.01					
	YB	0.01	0.01					
Zinc (Zn)	*FC*	21.01	4.02	26	None	p = 0.015	p = 0.016	Moolack Beach higher than Seal Rock; Nye Beach site intermediate. Overall similar to Mussel Watch
	MB	22.01	2.86					
	NB	18.10	1.24					
	SR	16.45	1.08					
	YB	16.01	3.91					

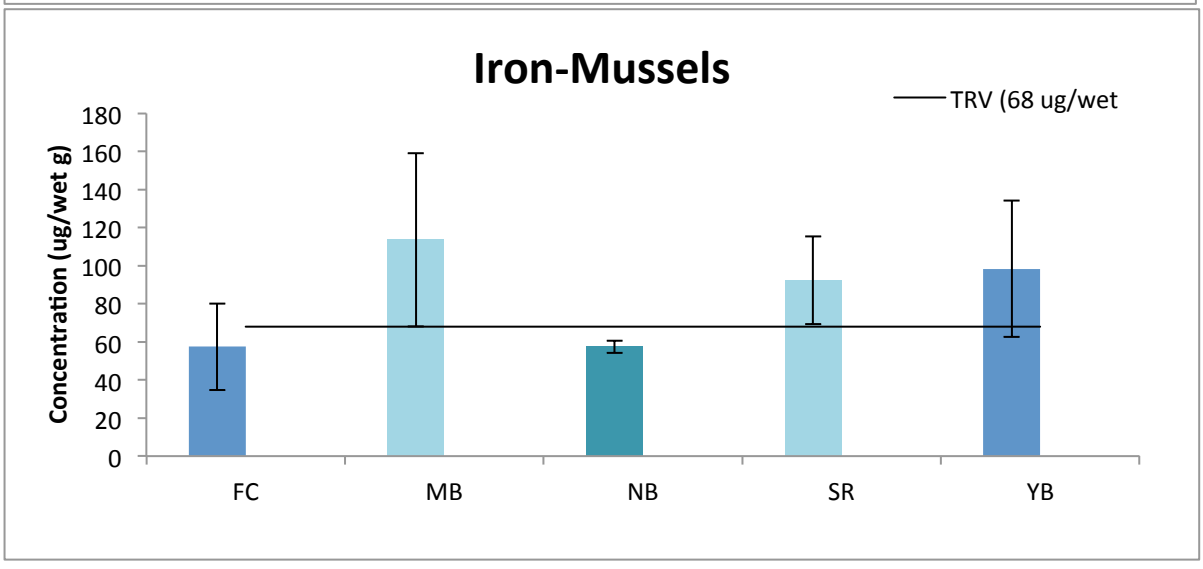

Aluminum-Mussels
TRV (31 ug/wet g)
Concentration (ug/wet g)
180
160
140
120
100
80
60
40
20
0
a
ab
a
ab
b
FC
MB
NB
SR
YB

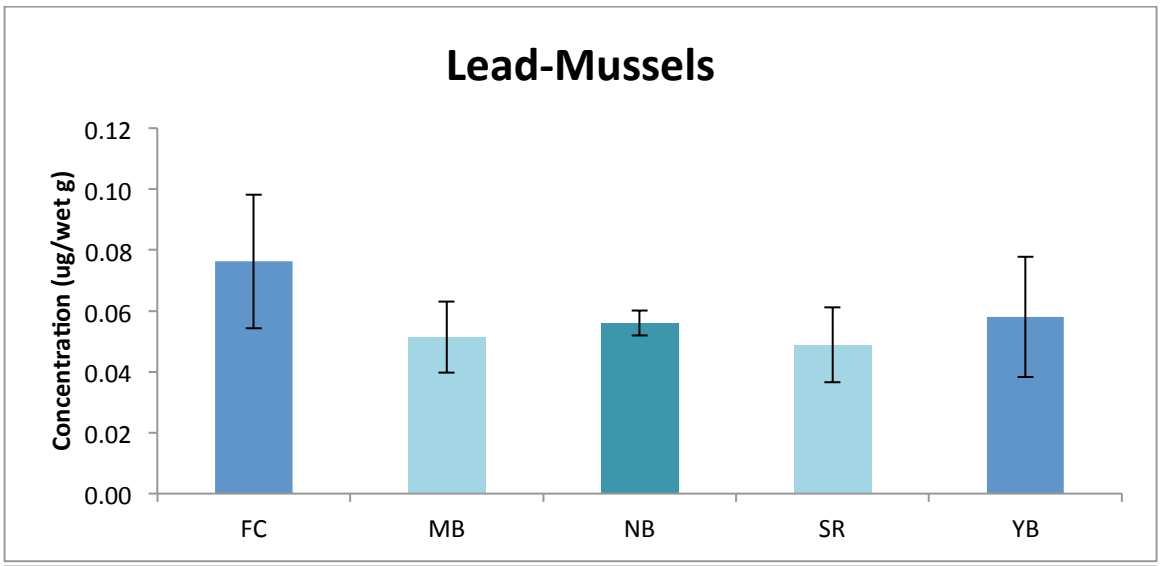

Arsenic-Mussels
TRV (3.6 ug/wet g)
Concentration (ug/wet g)
6
5
4
3
2
1
0
a
b
b
c
c
FC
MB
NB
SR
YB

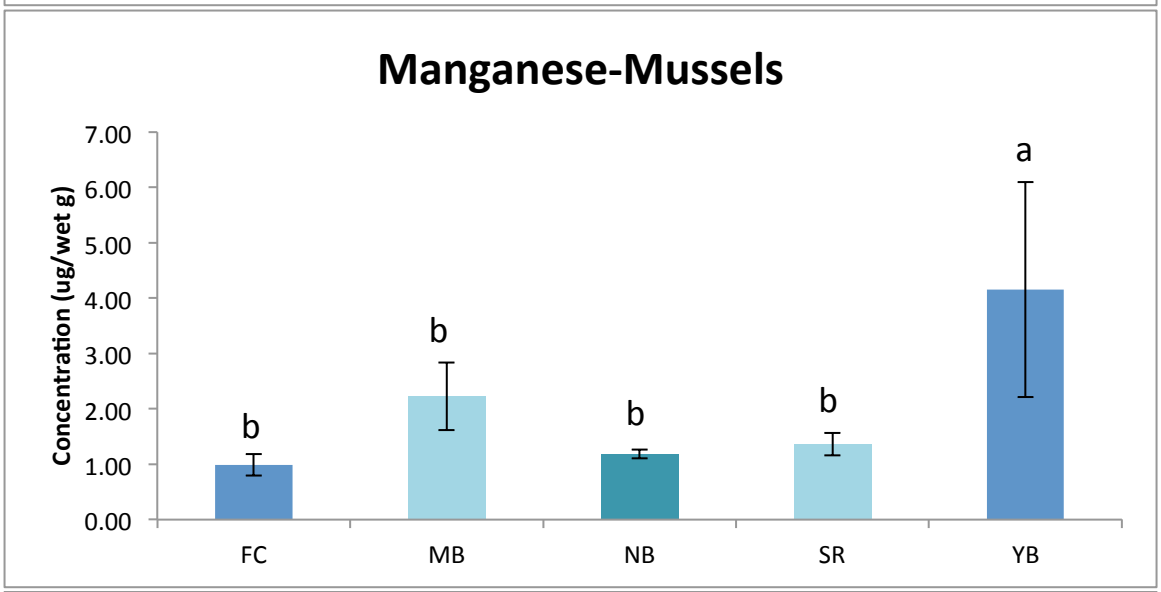

Cadmium-Mussels
Concentration (ug/wet g)
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0.00
a
a
a
b
c
FC
MB
NB
SR
YB

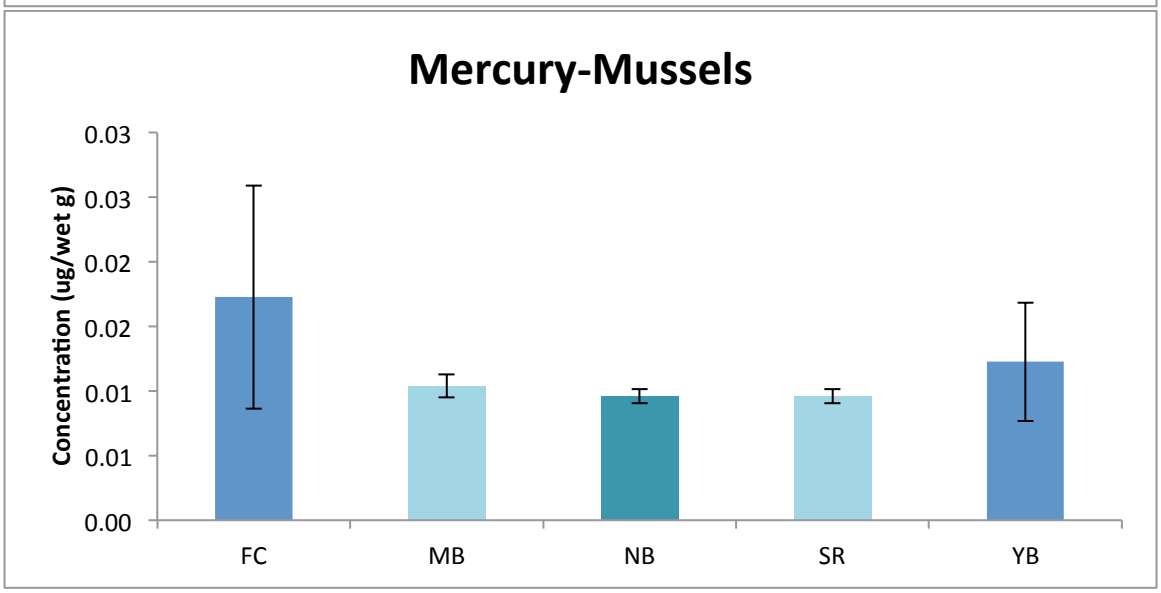

Chromium-Mussels
Concentration (ug/wet g)
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
ab
a
ab
ab
b
FC
MB
NB
SR
YB

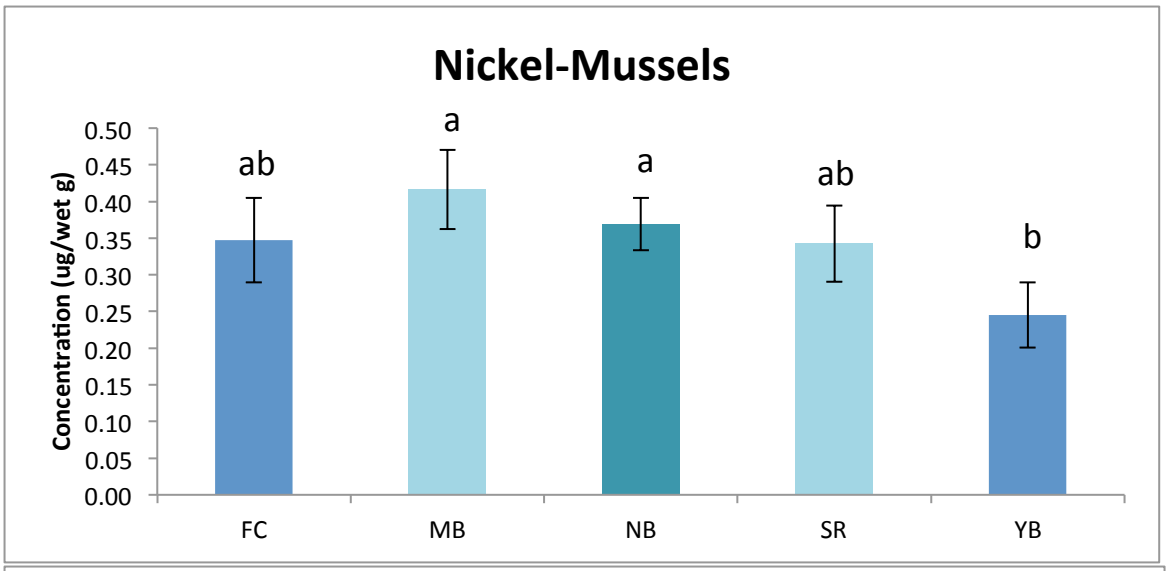

Copper-Mussels
Concentration (ug/wet g)
2.00
1.80
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0.00
ab
b
a
b
b
FC
MB
NB
SR
YB

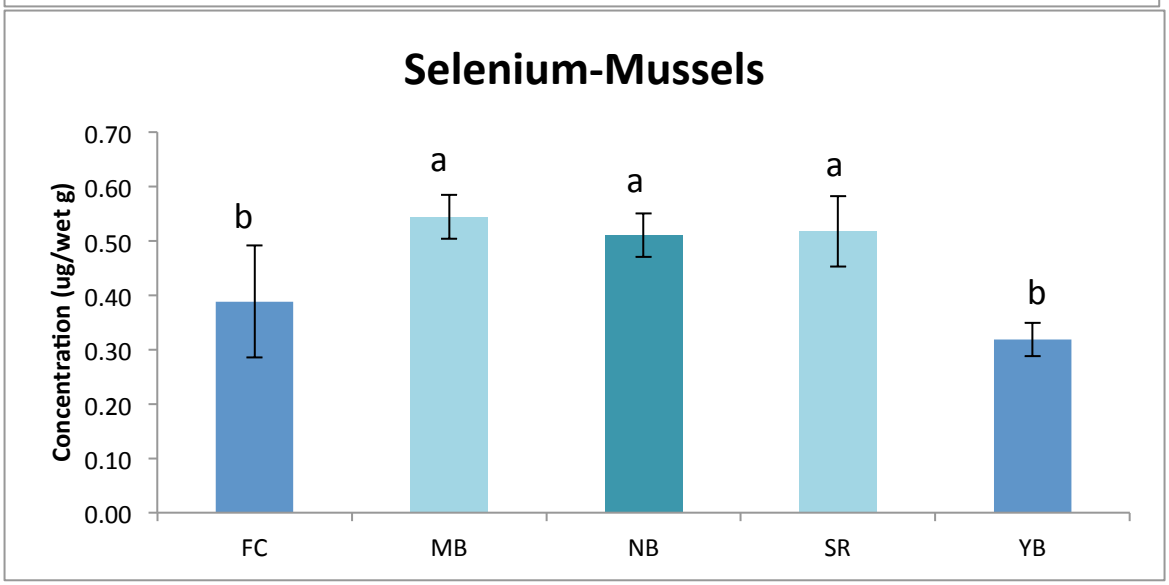

Iron-Mussels
TRV (68 ug/wet
Concentration (ug/wet g)
180
160
140
120
100
80
60
40
20
0
FC
MB
NB
SR
YB

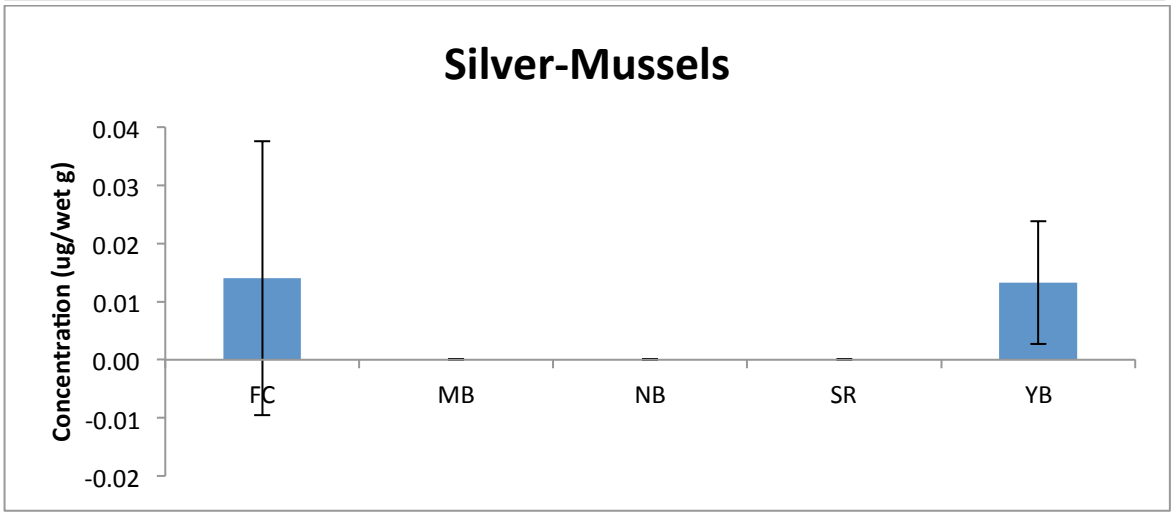

Lead-Mussels
Concentration (ug/wet g)
0.12
0.10
0.08
0.06
0.04
0.02
0.00
FC
MB
NB
SR
YB

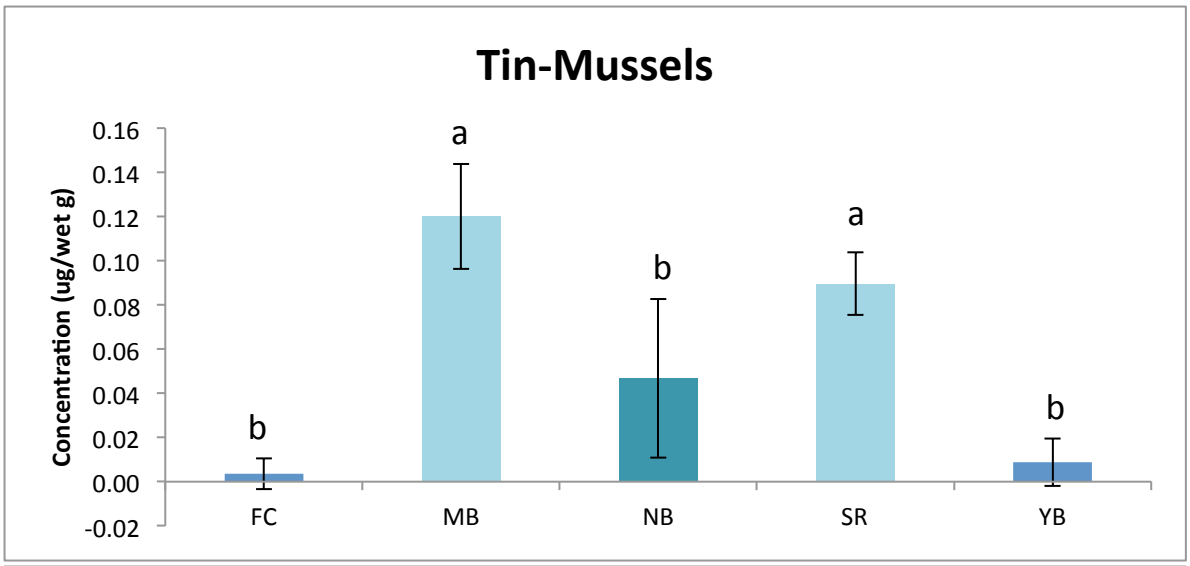

Manganese-Mussels
Concentration (ug/wet g)
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
b
b
b
b
a
FC
MB
NB
SR
YB

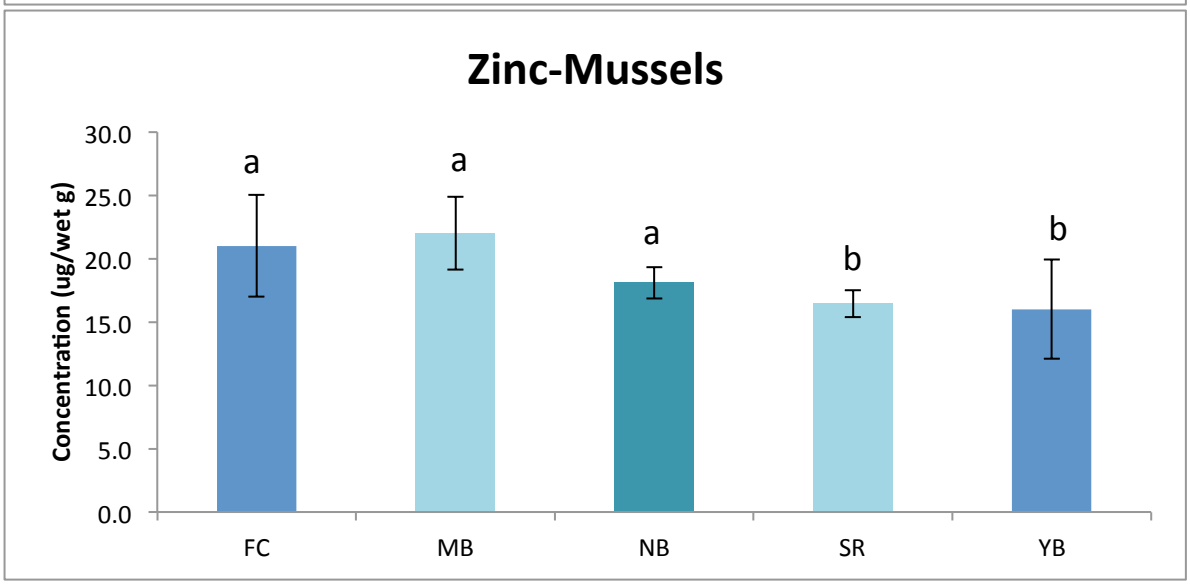

Mercury-Mussels
Concentration (ug/wet g)
0.03
0.03
0.02
0.02
0.01
0.01
0.00
FC
MB
NB
SR
YB


Nickel-Mussels
Concentration (ug/wet g)
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
ab
a
a
ab
b
FC
MB
NB
SR
YB


Selenium-Mussels
Concentration (ug/wet g)
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
b
a
a
a
b
FC
MB
NB
SR
YB


Silver-Mussels
Concentration (ug/wet g)
0.04
0.03
0.02
0.01
0.00
-0.01
-0.02
FC
MB
NB
SR
YB


Tin-Mussels
Concentration (ug/wet g)
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
a
b
a
b
b
FC
MB
NB
SR
YB

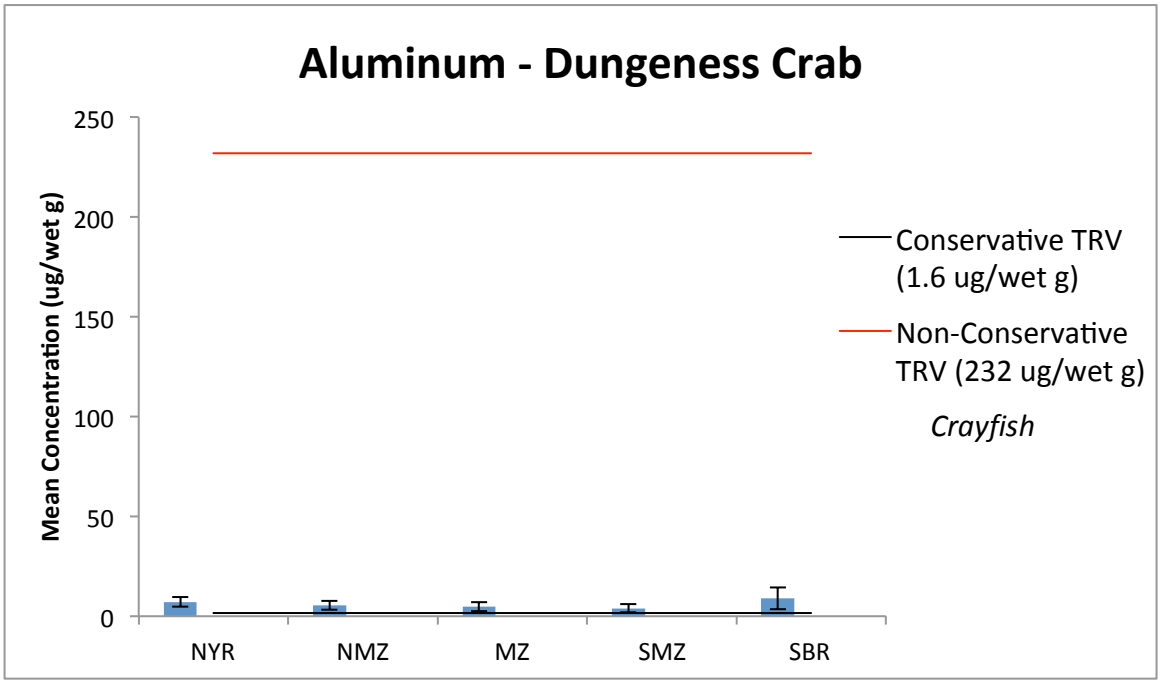

Zinc-Mussels
Concentration (ug/wet g)
30.0
25.0
20.0
15.0
10.0
5.0
0.0
a
a
a
b
b
FC
MB
NB
SR
YB

CRABS

While certain metals exceed a reference value for toxicity to the marine species itself (in most cases it is a conservative value below which there is "no observable effect"), **concentrations were not approaching levels for human health concern for crustacean consumption**. Crabs likely have accumulated the widest variety of metals and organics because they eat both live infaunal invertebrates and scavenge on various dead organisms. They likely did not show differences among sites because they are highly mobile. Although crabs did not show accumulation patterns that appear to be attributable to the G-P outfall, we briefly discuss the seven metals that exceeded TRVs for crabs since these organisms are most likely to be consumed by humans. Figures for each metal show the average concentrations of the metal in crab samples from each site. The conservative and, where available, the less conservative TRVs are plotted. The figure legend shows the experimental organism on which the TRV is based. Where available, the FDA (or other human health) regulatory levels also are plotted.

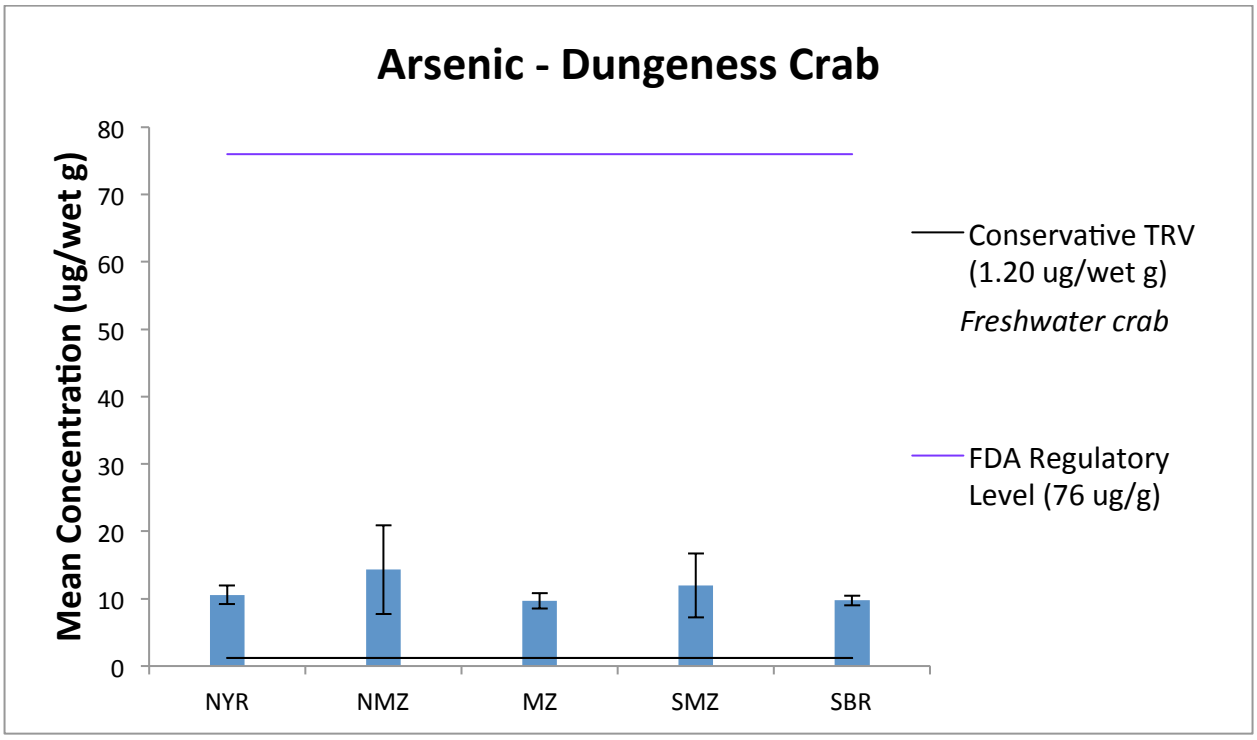
Aluminum

Aluminum is considered a metal with minor potential for toxicity (IOM 2001). Concentrations in crabs collected for this study ranged from 1.6 to 18.4 µg/g; thus, nearly all samples exceeded the conservative TRV of 1.6 µg/g; however, they did not approach the less conservative TRV of 232 µg/g.



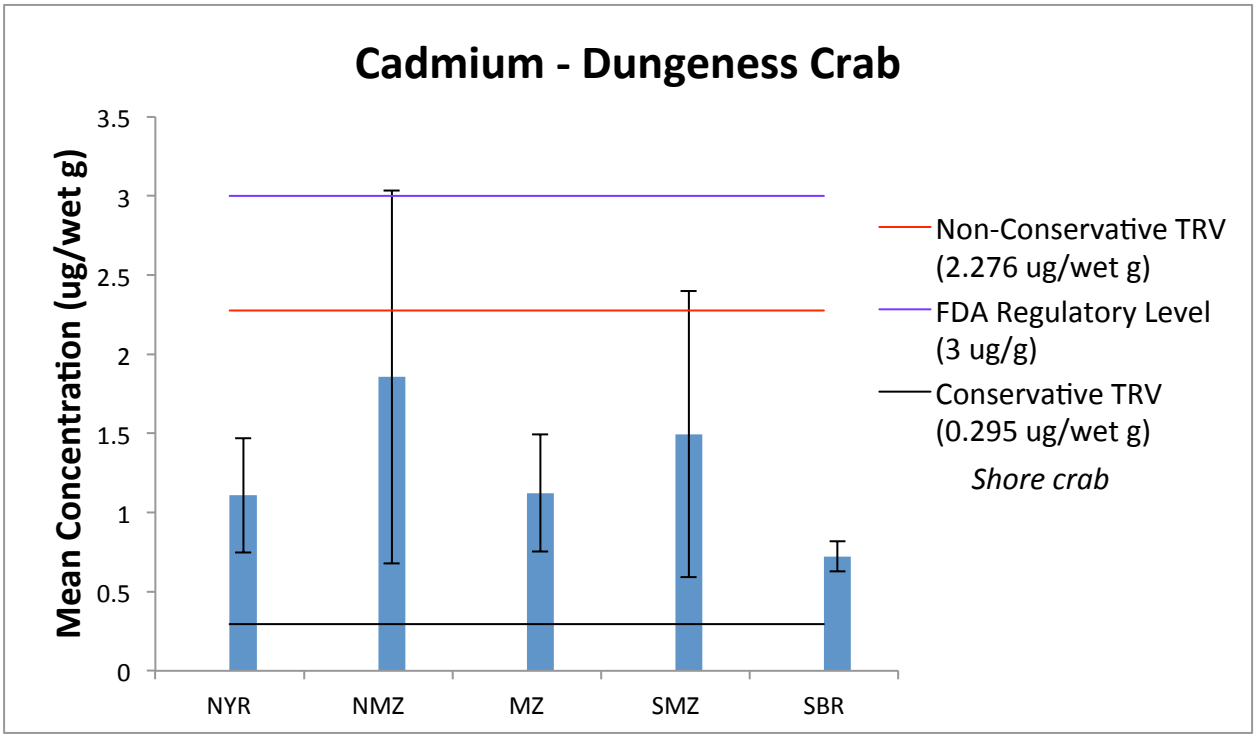
Arsenic

Arsenic is usually found in the environment in both organic and inorganic forms. Most of the arsenic found in seafood is the less harmful organic form. Based on various studies, the FDA proposes that 10 % of total detected arsenic be estimated as the more harmful inorganic form. Thus, estimated values of inorganic arsenic for crabs in this study would range from 0.77 to 1.23 µg/g, which is ~100 times less than the FDA Regulatory Level of 76 µg/g for crustaceans (FDA 1993).



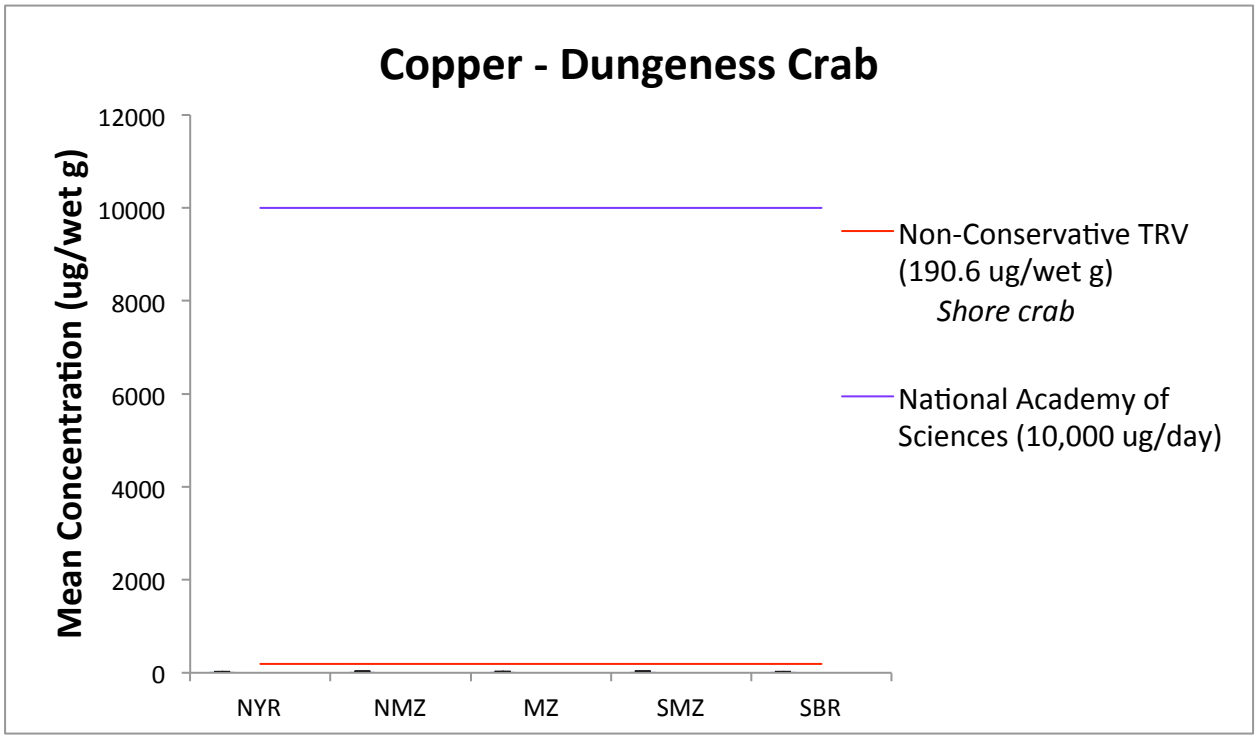
Cadmium

Cadmium is considered to be a relatively recent (50 years) contaminant of the aquatic environment. Its sources are solid waste dumping (pigment in paint) and cadmium-containing sewage sludge due to the use of phosphatic fertilizers, electroplating and galvanizing manufacture, and mining wastewater (Sherlock 1986; Sloan and Karcher 1985). While the cadmium levels detected in crabs for this study (0.569 – 1.897 µg/g) exceeded the conservative TRV of 0.295 µg/g, they are well below the FDA Regulatory level of 3 µg/g for crustaceans (FDA 2001a). There is a differential affinity between crustacean muscle and hepatopancreas, the latter organ containing 10-20 times the concentration of the former (IOM 2001). Since the whole body (minus the gut) of the crabs was analyzed for this study, the amount of cadmium potentially accumulated in the musculature and consumed by human would be considerably lower than the values reported here.



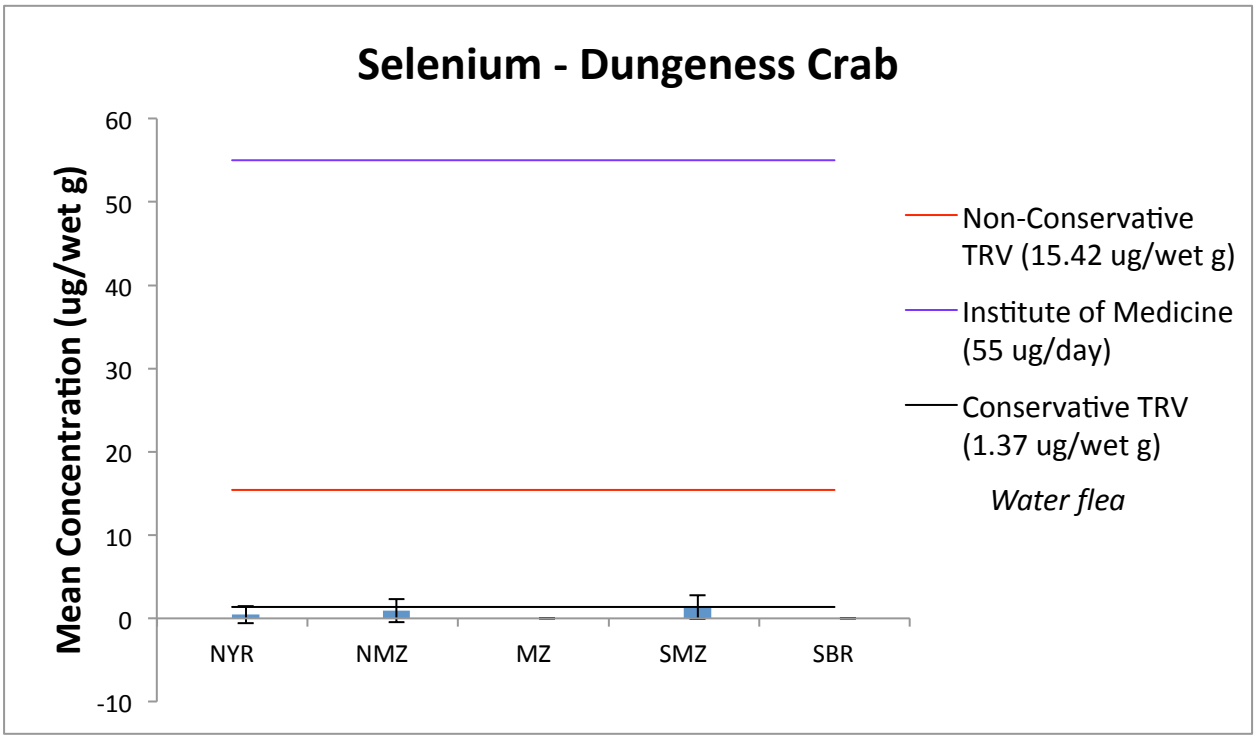
Copper

Copper is a necessary nutrient for humans; thus there are few toxicological and epidemiological studies available. There is no FDA Regulatory Level for copper, but the level determined by the National Academy of Sciences to be safe (no long-term liver damage) is 10,000 µg copper/day (NAS 2000). Copper concentrations in crabs collected for this study ranged from 11.8 to 23.3 µg/g of whole body crab tissue.



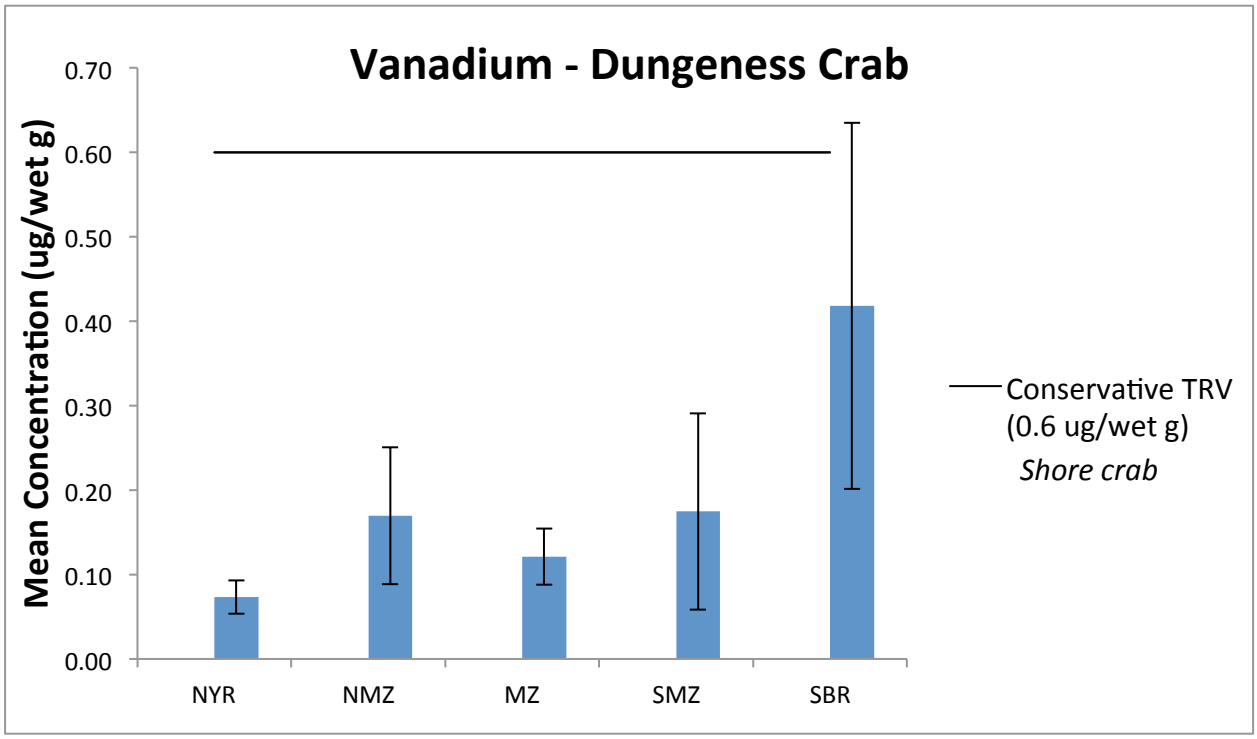
Selenium

Selenium likewise is an essential nutrient but at slightly higher levels functions as a poison; it has both potential protective (from mercury toxicity) and deleterious effects (IOM 2001). The recommended daily value for adults is 55 µg/day with a tolerable upper limit of 400 µg/day. Anthropogenic contamination is the product of fossil fuel combustion (fly ash) and of paint, alloy, photoelectric battery, and rectifier manufacture (Fishbein 1983; Sorensen et al. 1984). Selenium concentrations in crabs collected for this study ranged from 0.6 to 2.3 µg/g, similar to the range of concentrations observed in large, oceanic fish such as swordfish and tuna (Kaneko and Ralston 2007). On average, samples were slightly lower in concentration than the conservative TRV of 1.37 µg/g and well below the less conservative TRV of 15.42 µg/g.



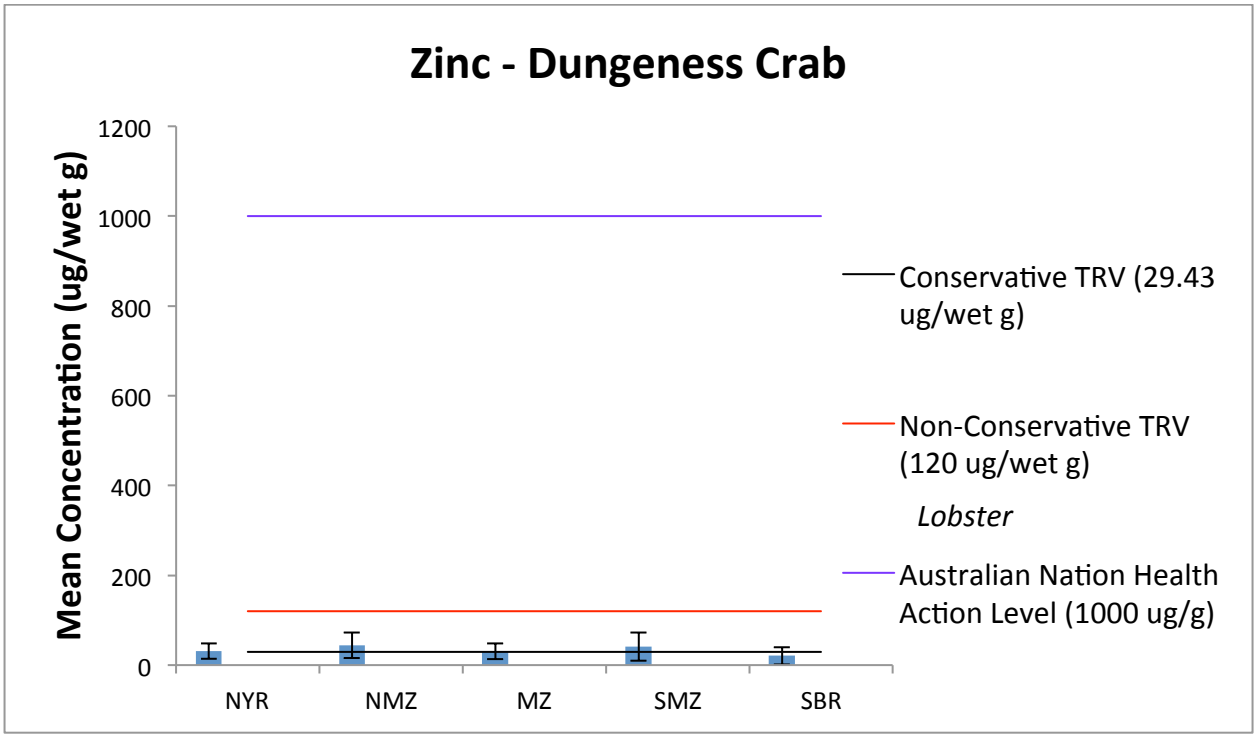
Vanadium

Vanadium concentrations in crabs collected for this study ranged from 0.045 to 0.603 µg/g; only two samples exceeded the conservative TRV of 0.6 µg/g. This was the only metal where differences among sites were detected, and the South Beach Reference site had the highest concentrations. There is no FDA Regulatory Level for vanadium, and studies on humans and animals suggest that most ingested vanadium is not absorbed into the bloodstream (less than 3 percent; ATSDR 1992). Thus, vanadium concentrations in crab tissue collected for this study do not indicate potential concerns for crabs or for human consumption.



Zinc

Zinc also is an essential nutrient and considered a metal with modest potential for toxicity (IOM 2001). There is no FDA Regulatory Level for zinc, but the Australian National Health and Medical Research Council set an Action Level at 1000 µg/g in seafood and the FAO/WHO acceptable daily intake is 15,000 µg/day (IOM 2001). Zinc concentrations in crabs collected for this study ranged from 21.3 to 44.6 µg/g, well below human heath action levels. Nearly all samples did exceed the conservative TRV of 29.43 µg/g but were well below the less conservative TRV of 120 µg/g.



Appendix 2

Additional Data Tables

Table A2-1: Summary of sampling events. Data are presented for each site showing species collected, the sampling date and corresponding number of organisms collected on that date (n), the type of sampling, and the sampling location coordinates. For trawl samples the starting latitude and longitude for tows are presented.

Site	Organism	Date (n)	Sampling Type	Latitude	Longitude
North Yaquina Reference	Dungeness crab	8/2/2012 (20) 8/18/2012 (30)	Crab Pot 1	44.7065	-124.0774
			Crab Pot 2	44.704	-124.0764
			Crab Pot 3	44.7018	-124.0762
	Speckled sanddab	9/19/2012 (5)	Trawl	44.7074	-124.081
	Crangon shrimp	9/19/2012 (250 ml)	Trawl	44.7074	-124.081
Moolack Beach	Olive snails	8/27/2012 (50)	Beach	44.7006	-124.0637
	Mytilus mussels	10/17/2012 (50)	Beach	44.7006	-124.0637
North Mixing Zone	Dungeness crab	8/2/2012 (25) 8/18/2012 (19) 8/19/2012 (6)	Crab Pot 1	44.6501	-124.0767
			Crab Pot 2	44.6488	-124.0766
			Crab Pot 3	44.6476	-124.0764
	Speckled sanddab	9/19/2012 (10)	Trawl	44.6504	-124.0761
	Crangon shrimp	9/19/2012 (250 ml)	Trawl	44.6504	-124.0761
Nye Beach N	Olive snails	8/27/2012 (50)	Beach	44.6392	-124.0647
Mixing Zone	Rock scallops	9/22/2013 (40)	Diving	44.6465	-124.0838
	Dungeness crab	8/2/2012 (32) 8/18/2012 (17) 8/19/2012 (1)	Crab Pot 1	44.643	-124.0774
			Crab Pot 2	44.6408	-124.0779
			Crab Pot 3	44.6392	-124.0775
	Speckled sanddab	9/19/2012 (10)	Trawl	44.643	-124.076
	Crangon shrimp	9/19/2012 (250 ml)	Trawl	44.643	-124.076
Nye Beach	Olive snails	8/27/2012 (50)	Beach	44.6366	-124.0655
	Mytilus mussels	10/17/2012 (50)	Beach	44.6366	-124.0655
South Mixing Zone	Dungeness crab	8/2/2012 (36) 8/18/2012 (14)	Crab Pot 1	44.6361	-124.0771
			Crab Pot 2	44.6349	-124.0779
			Crab Pot 3	44.6348	-124.0766
	Speckled sanddab	9/19/2012 (8)	Trawl	44.6348	-124.0814
	Crangon shrimp	9/19/2012 (250 ml)	Trawl	44.6348	-124.0814
Nye Beach S	Olive snails	8/27/2012 (50)	Beach	44.6343	-124.0662
South Beach Reference	Dungeness crab	8/2/2012 (10) 8/18/2012 (25) 8/19/2012 (15)	Crab Pot 1	44.5642	-124.0862
			Crab Pot 2	44.5628	-124.0864
			Crab Pot 3	44.5607	-124.0862
	Speckled sanddab	9/19/2012 (5)	Trawl	44.568	-124.0876
	Crangon shrimp	9/19/2012 (250 ml)	Trawl	44.568	-124.0876
Lost Creek	Olive snails	8/28/2012 (50)	Beach	44.4948	-124.0846
Seal Rock	Rock scallops	12/5/2013 (52)	Dive	44.54950	-124.1154
Seal Rock	*Mytilus* mussels	10/17/2012 (50)	Beach	44.5511	-124.0726

Table A2-2: Individual Organochlorine pesticides, PCBs, PBDEs, and phenolics analyzed. All compounds were tested for in all collected organisms.

Parameter	Method	Group	MDL	MDL_Units	RL
PCB Congeners					
PCB003	EPA 8270C	PCB Congeners	1	ng/g	5
PCB008	EPA 8270C	PCB Congeners	1	ng/g	5
PCB018	EPA 8270C	PCB Congeners	1	ng/g	5
PCB028	EPA 8270C	PCB Congeners	1	ng/g	5
PCB031	EPA 8270C	PCB Congeners	1	ng/g	5
PCB033	EPA 8270C	PCB Congeners	1	ng/g	5
PCB037	EPA 8270C	PCB Congeners	1	ng/g	5
PCB044	EPA 8270C	PCB Congeners	1	ng/g	5
PCB049	EPA 8270C	PCB Congeners	1	ng/g	5
PCB052	EPA 8270C	PCB Congeners	1	ng/g	5
PCB056/060	EPA 8270C	PCB Congeners	1	ng/g	5
PCB066	EPA 8270C	PCB Congeners	1	ng/g	5
PCB070	EPA 8270C	PCB Congeners	1	ng/g	5
PCB074	EPA 8270C	PCB Congeners	1	ng/g	5
PCB077	EPA 8270C	PCB Congeners	1	ng/g	5
PCB081	EPA 8270C	PCB Congeners	1	ng/g	5
PCB087	EPA 8270C	PCB Congeners	1	ng/g	5
PCB095	EPA 8270C	PCB Congeners	1	ng/g	5
PCB097	EPA 8270C	PCB Congeners	1	ng/g	5
PCB099	EPA 8270C	PCB Congeners	1	ng/g	5
PCB101	EPA 8270C	PCB Congeners	1	ng/g	5
PCB105	EPA 8270C	PCB Congeners	1	ng/g	5
PCB110	EPA 8270C	PCB Congeners	1	ng/g	5
PCB114	EPA 8270C	PCB Congeners	1	ng/g	5
PCB118	EPA 8270C	PCB Congeners	1	ng/g	5
PCB119	EPA 8270C	PCB Congeners	1	ng/g	5
PCB123	EPA 8270C	PCB Congeners	1	ng/g	5
PCB126	EPA 8270C	PCB Congeners	1	ng/g	5
PCB128	EPA 8270C	PCB Congeners	1	ng/g	5
PCB138	EPA 8270C	PCB Congeners	1	ng/g	5
PCB141	EPA 8270C	PCB Congeners	1	ng/g	5
PCB149	EPA 8270C	PCB Congeners	1	ng/g	5
PCB151	EPA 8270C	PCB Congeners	1	ng/g	5

PCB153	EPA 8270C	PCB Congeners	1	ng/g	5
PCB156	EPA 8270C	PCB Congeners	1	ng/g	5
PCB157	EPA 8270C	PCB Congeners	1	ng/g	5
PCB158	EPA 8270C	PCB Congeners	1	ng/g	5
PCB167	EPA 8270C	PCB Congeners	1	ng/g	5
PCB168+132	EPA 8270C	PCB Congeners	1	ng/g	5
PCB169	EPA 8270C	PCB Congeners	1	ng/g	5
PCB170	EPA 8270C	PCB Congeners	1	ng/g	5
PCB174	EPA 8270C	PCB Congeners	1	ng/g	5
PCB177	EPA 8270C	PCB Congeners	1	ng/g	5
PCB180	EPA 8270C	PCB Congeners	1	ng/g	5
PCB183	EPA 8270C	PCB Congeners	1	ng/g	5
PCB187	EPA 8270C	PCB Congeners	1	ng/g	5
PCB189	EPA 8270C	PCB Congeners	1	ng/g	5
PCB194	EPA 8270C	PCB Congeners	1	ng/g	5
PCB195	EPA 8270C	PCB Congeners	1	ng/g	5
PCB199/200	EPA 8270C	PCB Congeners	1	ng/g	5
PCB201	EPA 8270C	PCB Congeners	1	ng/g	5
PCB206	EPA 8270C	PCB Congeners	1	ng/g	5
PCB209	EPA 8270C	PCB Congeners	1	ng/g	5
Phenols					
2,3,4,6-Tetrachlorophenol	EPA 8270C	Phenols	50	ng/g	10
2,4,5-Trichlorophenol	EPA 8270C	Phenols	50	ng/g	10
2,4,6-Trichlorophenol	EPA 8270C	Phenols	50	ng/g	10
2,4-Dichlorophenol	EPA 8270C	Phenols	50	ng/g	10
2,4-Dimethylphenol	EPA 8270C	Phenols	100	ng/g	20
2,4-Dinitrophenol	EPA 8270C	Phenols	100	ng/g	20
2,6-Dichlorophenol	EPA 8270C	Phenols	50	ng/g	10
2-Chlorophenol	EPA 8270C	Phenols	50	ng/g	10
2-Methyl-4,6-dinitrophenol	EPA 8270C	Phenols	100	ng/g	20
2-Methylphenol	EPA 8270C	Phenols	100	ng/g	20
2-Nitrophenol	EPA 8270C	Phenols	100	ng/g	20
3+4-Methylphenol	EPA 8270C	Phenols	100	ng/g	20
4-Chloro-3-methylphenol	EPA 8270C	Phenols	100	ng/g	20
4-Methylphenol	EPA 8270C	Phenols	100	ng/g	20
4-Nitrophenol	EPA 8270C	Phenols	100	ng/g	20
Pentachlorophenol	EPA 8270C	Phenols	50	ng/g	10
Phenol	EPA 8270C	Phenols	100	ng/g	20
Organochlorine Pesticides					

2,4'-DDD	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
2,4'-DDE	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
2,4'-DDT	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
4,4'-DDD	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
4,4'-DDE	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
4,4'-DDT	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Aldrin	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
BHC-gamma	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Chlordane-alpha	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Dieldrin	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Endosulfan Sulfate	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Endosulfan-I	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Endosulfan-II	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Endrin	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Heptachlor	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Heptachlor Epoxide	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Hexachlorobenzene	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
Mirex	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
trans-Nonachlor	EPA 8270C	Organochlorine Pesticides	1	ng/g	5
PBDE Congeners					
PBDE017	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE028	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE047	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE066	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE071	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE085	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE099	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE100	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE138	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE153	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE154	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE183	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE190	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5
PBDE209	EPA 8270CNCI	PBDE Congeners by NCI	1	ng/g	5

Table A2-3: Individual trace metals analyzed. All metals were tested for in all collected organisms.

Parameter	Method	Group	MDL	MDL_Units	RL
Trace Elements					
Aluminum (Al)	EPA 6020	Trace Elements	1	µg/g	5
Antimony (Sb)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Arsenic (As)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Barium (Ba)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Beryllium (Be)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Cadmium (Cd)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Chromium (Cr)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Cobalt (Co)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Copper (Cu)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Iron (Fe)	EPA 6020	Trace Elements	1	µg/g	5
Lead (Pb)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Manganese (Mn)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Molybdenum (Mo)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Nickel (Ni)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Selenium (Se)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Silver (Ag)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Strontium (Sr)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Thallium (Tl)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Tin (Sn)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Titanium (Ti)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Vanadium (V)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Zinc (Zn)	EPA 6020	Trace Elements	0.025	µg/g	0.05
Parameter	**Method**	**Group**	**MDL**	**MDL_Units**	**RL**
Trace Mercury					
Mercury (Hg)	EPA 245.7	Trace Metals	0.00001	µg/g	2E-05

Table A2-4: Data quality objectives for organochlorine pesticides, PCBs, PBDEs, and phenolics.

Element or Sample Type	Minimum Frequency	Acceptance Criteria
5-Point Calibration	Initially and when CCAL fails	%RSD $\leq$ 25% for all analytes
Continuing Calibration	Start and End of Each Analytical Sequence	± 25% of the true value for each analyte using a second source standard
GCMS Tune	Initially and beginning of each batch	3-6 ions within EPA CFR40 Part 136 Acceptance Criteria
Reference Material	1 per Batch (max of 20 samples per batch)	± 30% of CI for True Value
Method Blank	1 per Batch	No analytes > 3 times the MDL unless analyte not detected in associated samples or analyte concentration > 10x blank value
Matrix Spike	Every Batch	% Recovery 50% – 125% if sample concentration is < 4x the matrix spike concentration
Sample Duplicate	1 per Batch	RPD < 30% if > 10x MDL
Surrogates	Every Sample added prior to extraction	% Recovery = 50 – 125%

Table A2-5: Data quality objectives for trace metals.

Element or Sample Type	Minimum Frequency	Acceptance Criteria
5-Point Calibration	Once Each Day	%RSD $\leq$ 15% for all analytes
Continuing Calibration	Between Each Batch of Samples	± 15% of the true value for each analyte using a second source standard
Reference Material	1 per Batch (max of 15 samples per batch)	± 25% of CI for True Value
Method Blank	1 per Batch	No analytes > 3 times the MDL unless analyte not detected in associated samples or analyte concentration > 10x blank value
Matrix Spike	Every Batch	% Recovery 75% – 125% if sample concentration is < 4x the matrix spike concentration
Sample Duplicate	1 per Batch	RPD < 25% if > 10x MDL

Appendix 3

Physis Quality Control Process

Physis Quality Control Process

Physis' quality control process is explained in The Quality Manual for Physis Environmental Laboratories, Revision #2. This living document outlines the utility and functionality of our quality system for the laboratory; setting forth and defining the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality. This is designed so as to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance. All laboratory operations are performed by these standards in this manual including the laboratory's organization, standard operating procedures, sample management, document control/storage and staff training.

Upon request, an entire electronic copy of the manual will be made available to the contractor by Physis. Below is information offered from the manual to explain our quality control process:

SECTION 5 – QUALITY SYSTEMS

The Quality Systems describe the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of the organization for ensuring quality in its work processes, products, and services.

5.1 Quality Policy

The quality policy statement demonstrates management's commitment to integrity, ethics, and the quality system and associated standards.

Quality Policy Statement

The objective of the quality system and the commitment of management is to consistently provide our customers with data of known and documented quality that meets their requirements. Our policy is to use good professional practices, to maintain quality, to uphold the highest quality of service, and to comply with ELAP. The laboratory ensures that personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through the unequivocal commitment of management, at all levels, to the Quality Assurance (QA) principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the laboratory organization. Every laboratory employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. All employees are trained annually on ethical principles and procedures surrounding the data that is generated. The laboratory will maintain a strict policy of client confidentiality.

SECTION 23 – QUALITY OF TEST RESULTS

23.1 Essential Quality Control Procedures

All essential quality control elements are collected and assessed on a continuing basis.
For test methods that do not provide acceptance criteria for an essential quality control element or where no regulatory criteria exist, acceptance criteria are developed. Control limits are developed using the mean, plus or minus 3 standard deviations; or static limits such as +/- 20 or 25 percent, depending on matrix/analyte of interest. The quality control procedures specified in test methods are followed by laboratory personnel. The most stringent of control procedures is used in cases where multiple controls are offered. If it is not clear which is the most stringent, that mandated by test method or regulation is followed. To monitor the validity of environmental tests performed, review includes any one combination of the techniques below:

a) use of certified reference materials and/or internal quality control using secondary reference materials;
b) participation in proficiency testing programs; and
c) replicate testing using the same or different methods.

Written procedures to monitor quality controls including acceptance criteria are located in the test method SOPs, except where noted, and include such procedures as:

a) use of blank spikes and blanks to serve as positive and negative controls for chemistry methods;
b) use of blank to monitor test variability of laboratory results;
c) use of calibrations, continuing calibrations, certified reference materials and/or PT samples to monitor accuracy of the test method;
d) measures to monitor test method capability, such as method detection limits, reporting limits, and/or range of test applicability, such as linearity;
e) use of regression analysis, internal/external standards, or statistical analysis to reduce raw data to final results;
f) use of reagents and standards of appropriate quality;
g) procedures to ensure the selectivity of the test method; and
h) measures to assure constant and consistent test conditions, such as temperature, humidity, rotation speed, etc., when required by test method.

23.2 Internal Quality Control Practices

Measurement Quality Objectives from the SWAMP 2008 QAP, Appendix A, will be used to summarize the key elements of our quality control system. Analytical data generated with QC samples that fall within prescribed acceptance limits indicate the test method is in control. QC samples that fall outside QC limits indicate the test method is out of control (non-conforming) and that corrective action is required or that the data are qualified. All QC measures are assessed and evaluated on an on-going basis, so that trends are detected. The following general controls are used:

Positive and Negative Controls such as:

a) Blanks (negative)
b) Blank spike (positive)

Selectivity is assured through:

a) absolute and relative retention times in chromatographic analyses;
b) two-column confirmation when using non-specific detectors;
c) use of acceptance criteria for mass-spectral tuning (found in test method SOPs);
d) use of the correct method according to its scope assessed during method validation

Consistency, Variability, Repeatability, and Accuracy are assured through:

a) proper installation and operation of instruments according to manufacturer's recommendations or according to the processes used during method validation;
b) monitoring and controlling environmental conditions (temperature, access, proximity to potential contaminants);
c) selection and use of reagents and standards of appropriate quality; and
d) cleaning glassware appropriate to the level required by the analysis. Cleaning procedures not provided in test method SOPs are provided in a separate SOP.
e) following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;
f) testing to define the variability and/or repeatability of the laboratory results, such as replicates;
g) use of measures to assure the accuracy of the test method, including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures.

Acceptance or rejection criteria are created according to laboratory policy where no method or regulatory criteria exist. Acceptance criteria define the boundary for the appropriate response from laboratory personnel, such as corrective action, reporting with qualifiers, reanalysis, review, and others.

Test Method Capability is assured through:

a) establishment of the method detection limit where appropriate;
b) establishment of the reporting level; and/or
c) establishment of the range of applicability such as linearity;

Data reduction is assured to be accurate by:

a) selection of appropriate formulae to reduce raw data to final results such as regression;
b) periodic review of data reduction processes to assure applicability;
c) data reduction and statistical interpretations specified by each test method.

23.3 Batches

The minimum requirements of a preparation batch are:
1) The number of samples in a batch is not to exceed 20.
2) All samples in a batch are of the same matrix.
3) The QC samples to be processed with the samples include:
a) Method Blank

Function: Determination of laboratory contamination.

b) Blank Spike

Function: Assessment of method performance

c) Matrix Spiked Sample

Function: Assessment of matrix problems

NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked.

d) Duplicate Blank Spike, Matrix Spiked Sample and Duplicate Sample

Function: Assessment of batch precision

NOTE: A sample identified as a field blank, an equipment blank, or a trip blank is not to be duplicated.

4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

7) The QC samples are to be analyzed in conjunction with the associated samples prepared with them. However, the QC samples in the batch do not require analysis each time a sample within the preparation batch is analyzed (multiple instrument sequences to analyze all samples in the batch need not include re-analyses of the QC samples).

8) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the samples.

9) Batch QC refers to the QC samples that are analyzed in a batch of samples.

10) Specific project, program, or method SOP requirements may be exceptions. If project, program, or method SOP requirements are more stringent than these laboratory minimum requirements, then the project, program, or method SOP requirements will take precedence. However, if the project, program, or method SOP requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

23.4 Method Blanks

The method blank is analyte-free water subjected to the entire analytical process. Contaminated blanks are identified according to the acceptance limits in the test method SOP. Samples associated with a contaminated blank are evaluated as to the appropriate corrective action for the samples (e.g. reprocessing or data qualifying codes). When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Data that are unaffected by the blank contamination (non-detects or other analytes) are reported unqualified. Sample data that are suspect due to the presence of a contaminated blank are reanalyzed, qualified, or deleted.

23.5 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve.

23.6 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater.

23.7 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard solutions that are, in turn, prepared from stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

23.8 Initial Calibration Verification Standards

Initial calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity of the standards used in the calibration. The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards. ICVs are also analyzed in accordance with method-specific requirements.

23.9 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

23.10 Internal Standards

Internal standards consist of known amounts of specific compounds that are added to each sample following sample preparation or extraction. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by certain matrix effects. The integrated area of the internal standard compared to the continuing calibration check standard should vary by no more than the limits specified in each method.

23.11 Blank Spikes

The results of blank spikes (BS) are calculated in percent recovery. See the calculation below for MS. The individual BS is compared to the acceptance criteria as published in the mandated test method, or where there are no established criteria, the laboratory established limits.

23.12 Matrix Spikes

The matrix spike (MS) results are used to help assess the effect of the sample matrix on method performance. The laboratory procedure for MS includes spiking appropriate analytes at

appropriate concentrations, calculating percent recoveries and evaluating and reporting the results. Spike recoveries are calculated as follows:

Recovery (%) = (S - A) x 100 ÷ T
Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

Where there are no established criteria, the laboratory uses the mean plus or minus three times standard deviations as the control limits for MS. For MS results outside established criteria corrective action is documented or the data are reported with appropriate data qualifying codes.

23.13 Duplicate Samples, Matrix Spike Duplicates and Blank Spike Duplicates
Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis and/or a matrix spiked sample and duplicate matrix spiked sample (MS/MSD), and/or a blank spike and blank spike duplicate (BS/BSD) are analyzed. The relative percent difference between duplicate analyses or between an MS/BS and MSD/BSD is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

Relative Percent Difference (RPD) = (S1 - S2) x 100 ÷ Save
Where: S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and
Save = The average of observed analyte concentrations in the sample and its duplicate, or in the spike and its spike duplicate.

23.14 Surrogate Spikes
Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, blank spikes and blank spike duplicates, matrix spike samples, and matrix spike duplicate samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance. Surrogate recovery results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory uses the mean plus or minus three standard deviations as surrogate control limits. For surrogate results outside established criteria, data are evaluated to determine the impact. Corrective actions include rerunning the samples, qualifying the data, and/or client discussion, as appropriate.

23.15 Proficiency Test Samples or Inter-laboratory Comparisons

The laboratory participates in proficiency test (PT) samples once per year. Corrective action procedures are instituted for all failed PT samples. The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider. Proficiency Testing (PT) or Proficiency Evaluation (PE) samples are treated as typical samples in the normal production process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. PT samples are not analyzed multiple times unless routine environmental samples are analyzed multiple times.

23.16 Data Review

The laboratory reviews all data generated in the laboratory for compliance with method, laboratory and, where appropriate, client requirements. All data review is documented. Initially, the analyst reviews data for acceptability of quality control measures and accuracy of the final result(s). After the initial review, the appropriate Supervisor acts as a second reviewer and considers all manual transfers and calculations of data in detail and spot checks all electronic transfers of data. Final reports are compared to raw data either directly or through several reviews.

Data Review Procedure

Bench sheets are used to record the information required for traceability of the analysis. The bench sheets include quality control measurements and acceptance criteria. Data are recorded on the bench sheets promptly at the time of the analysis, in ink. Analysts review sample data and the QC information at the time of analysis and indicate if the QC parameters meet the acceptance criteria by marking the bench sheet. The analyst signs and dates the bench sheet to indicate that they have performed the steps indicated and that the analysis meets acceptance criteria or has exceptions that are noted in the comments section of the bench sheet. When the analyst has finished the primary analysis review, the Supervisor in the laboratory checks the bench sheet for the following items:

a) All required information has been recorded on the bench sheet.

b) QC criteria have been met or exceptions are documented in the comments section of the bench sheet.

c) Manual calculations are checked for accuracy.

When these checks have been completed, the reviewer signs and dates the bench sheet to document that the review has been performed. The bench sheet is used by office personnel to enter the data in LIMS. The report is generated, reviewed and signed by the Project Manager. This final review includes verifying that the data entered on the worksheet has been appropriately transferred to the LIMS and that the data is coherent, that QC results are acceptable, QC exceptions are appropriately reflected on the final report, and results are in line with historical values, if known.