

Open-File Report 2009–1123

U.S. Department of the Interior U.S. Geological Survey

By Thomas W. May, Michael J. Walther, Michael K. Saiki, and William G. Brumbaugh

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### **U.S. Department of the Interior**

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## **Conversion Factors**

Multiply	Ву	To obtain
	Length	
millimeter (mm)	0.03937	inch (in.)
micrometer	0.0000393	inch (in.)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
milliliter (mL)	.034	ounce, fluid (fl. oz)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
nilligram (mg)	.000035	ounce (oz)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

#### °F=(1.8×°C)+32

Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter ( $\mu$ g/L).

Concentrations of chemical constituents in solid materials are given in micrograms per gram ( $\mu$ g/g) dry weights.

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## Abstract

This report presents the results for two sampling periods (October 2008 and January 2009) during a 4-year monitoring program to characterize selenium concentrations in selected irrigation drains flowing into the Salton Sea, California. Total selenium, selenium species (dissolved selenite, selenate, organoselenium), and total suspended solids were determined in water samples. Total selenium also was determined in water column particulates and in sediment, detritus, and biota that included algae, plankton, midge larvae (family, Chironomidae), and two fish species (western mosquitofish, Gambusia affinis, and sailfin molly, *Poecilia latipinna*). In addition, sediments were analyzed for percent total organic carbon and particle size. Mean total selenium concentrations in water for both sampling periods ranged from 1.00 to 33.6 micrograms per liter, predominately as selenate, which is typical of waters where selenium is leached out of selenium-containing marine shales and associated soils under alkaline and oxidizing conditions. Total selenium concentrations (micrograms per gram dry weight) ranged as follows: algae, 1.52 to 8.26; plankton, 0.79 to 3.66; midges, 2.68 to 50.6; fish, 3.09 to 30.4; detritus, 1.78 to 58.0; and sediment, 0.42 to 10.0.

## Introduction

Monitoring surveys are being conducted by the U.S. Geological Survey (USGS) for 4 years to provide a profile of selenium concentrations in selected irrigation drain inflows to the Salton Sea, California. To accomplish this goal, total selenium, selenium species (dissolved selenite, selenate, organoselenium), and total suspended solids were determined in water samples. Total selenium was measured in water column particulates and in sediment, detritus, and biota that included algae, plankton, midge larvae (family, Chironomidae), and two fish species (western mosquitofish, *Gambusia affinis*, and sailfin molly, *Poecilia latipinna*). In addition, sediments were analyzed for percent total organic carbon and particle size. The results in this report were derived from samples collected during October 2008 and January 2009.

## Methods

### **Field Collection and Preservation**

The USGS sampling team used the laboratory at the U.S. Fish and Wildlife Service Sonny Bono Salton Sea National Wildlife Refuge (henceforth referred to as "the Refuge") for certain aspects of sample processing and for preparing samples for shipment during field trips to irrigation drains.

Unfiltered Water: Each water sample to be analyzed for total selenium was poured through a 1-millimeter (mm) polypropylene sieve attached to a 1-liter (L) precleaned borosilicate glass bottle. Upon collection, the water sample was acidified to less than pH 2 with 6 normal (N) hydrochloric acid (HCl), chilled to approximately four degrees Celsius (~4 °C), and kept in the dark during transport to the USGS. Each water sample intended for analysis of total suspended solids (TSS) was poured through a 1-mm polypropylene sieve attached to a precleaned widemouth 1-L polypropylene bottle. The TSS samples were chilled (~4 °C) during transport to the USGS.

*Filtered Water*: Water for selenium speciation was filtered using a Geotech<sup>®</sup> peristaltic pump equipped with a standard pumphead and high-capacity 0.45 micrometer ( $\mu$ m) filter capsule certified for trace-element background. All tubing was acid-cleaned silicone and a new length was used at each site and for the blank. At each site, 1 L of deionized (DI) water was filtered through the filter capsule followed by site water. The first 200 milliliters (mL) of site water eluant were discarded, then 1 L of eluant was collected in an acid-cleaned 1-L borosilicate glass bottle, acidified, and stored as described earlier for unfiltered water.

*Particulates*: A polycarbonate Geotech<sup>®</sup> 142-mm plate filter apparatus was used with a 142-mm 0.4-µm polycarbonate filter. At each site, 0.5 L of DI water was filtered through the plate filter, followed by up to 1 L of site water; after volume notation, the filtrate was discarded. Each filter was placed in a precleaned plastic petri dish (150 mm x 15 mm) with the particulate side up and sealed with its corresponding cover for freezer storage and transport to the USGS. The plate filtration unit was rinsed with 0.1 percent nitric acid (HNO<sub>3</sub>), followed by a DI water rinse after sampling was completed at each site.

Sediment: Five sampling points for sediment collection were identified along the length of each drain. At each sampling point, the uppermost 2 to 6 cm of sediment were collected with a stainless steel dredge. The dredge was cleared of mud and rinsed with site water while used within a drain. At a new site, the dredge was rinsed with DI water followed by site water before the first sample was collected. From each of the five sampling points, enough sediment to fill a 250-mL container was collected and mixed to form a composite sample (1,250-mL total). An aliquot of this composite was then placed into a 120- and a 500-mL polypropylene container for each drain sampled. All containers were placed on ice in the field. Samples were chilled (~4 °C) during transport to the USGS.

*Midge Larvae and Detritus*: An insect sweep net was used to collect samples of midge larvae and detritus, which were then sorted and hand picked with plastic tweezers in a polypropylene sieve, and stored temporarily in a plastic foodstorage container on ice. After rinsing with DI water, samples were wrapped in plastic wrap, stored in separate plastic bags, and frozen.

*Fish*: Composite samples of western mosquitofish and sailfin molly were collected with seine nets and minnow traps; sampled fish from each site were stored temporarily in a plastic food-storage container on ice. Upon return to the Refuge laboratory, the whole-body fish were measured for standard length, weighed, and rinsed with DI water. Each fish composite sample (n=36) was wrapped in plastic wrap and placed into a plastic bag and frozen.

Algae and Plankton: Algae were collected from floating masses or scraped from sticks and rocks at each drain site and stored temporarily in a sealable plastic food-storage container on ice. Following collection, the material was rinsed with DI water, wrapped with plastic wrap, stored in a plastic bag, and frozen. Plankton was collected with a tow net and after draining site water, each sample was rinsed three times with DI water. The plankton and the DI rinsing water were placed in a 120-mL polypropylene container. Collected samples were stored on ice in the field and frozen immediately upon return to the Refuge.

### **Sampling History**

The irrigation drain monitoring samples collected by USGS personnel were received in six shipments by the Environmental Chemistry Branch Inorganic Section (henceforth referred to as "the laboratory") of the USGS shortly after collection to meet the 7 day holding time specified for TSS in water and the 14-day holding time for total organic carbon (TOC) in sediments. The first set of samples was collected from October 15 to 17, 2008, received by the laboratory on October 21, 2008, and contained 17 TSS water samples, 26 water samples for total selenium, and 6 water samples for total dissolved selenium. The samples were assigned USGS batch number 1525 and USGS sample identification numbers 44164 to 44212.

The second set of samples was collected from October 15 to 21, 2008, received by the laboratory on October 23, 2008, and contained 14 TSS water samples, 22 total selenium water samples, 11 total dissolved selenium water samples, 8 sediment samples for total selenium, 8 sediment samples for TOC and particle size analysis (PSA), and 15 particulate selenium filter samples. The samples were assigned USGS batch number 1530 and USGS sample identification numbers 44395 to 44472.

The third set of samples was received by the laboratory on October 24, 2008, and contained two filters to serve as blanks. The samples were assigned USGS batch number 1533 and USGS sample identification numbers 44485 to 44486.

The fourth set of samples was collected from October 15 to 21, 2008, received by the laboratory on November 5, 2008, and contained 42 fish samples, 21 algae samples, 21 midge samples, 21 detritus samples, and 21 plankton samples. The samples were assigned USGS batch number 1538 and USGS sample identification numbers 44570 to 44695.

The fifth set of samples was collected from January 8 to 11, 2009, received by the laboratory on January 13, 2009, and contained 31 TSS water samples and 62 total selenium water samples. The samples were assigned USGS batch number 1553 and USGS sample identification numbers 45067 to 45159.

#### Homogenization and Lyophilization

Frozen fish samples were minced with a small ceramic knife before freeze drying. Particulates, biota, detritus, and sediment samples were lyophilized in a Virtis Genesis® 35EL freeze dryer and percent moisture was determined as part of the lyophilization process; however, percent moisture was not determined for plankton samples because the sample matrix included DI water. After lyophilization, all midge larvae, detritus, plankton, algae, and fish samples were homogenized by grinding with a glass rod against the container surface. Dried sediment was placed into a plastic bag, sealed, and then pulverized by using a rolling pin on the plastic bag to produce a coarse powder product. Dried filters containing particulates did not require any additional homogenization after freeze drying.

#### **Chemical Procedures**

*Total Selenium in Water*: Before analysis, all water samples were stored in the dark at ~4 °C. For the subsequent determination of total selenium in filtered and unfiltered samples, a 20-mL aliquot of each acidified water sample was subjected

to an HNO<sub>3</sub>-magnesium nitrate  $[Mg(NO_3)_2]$  ashing procedure, followed by treatment with HCl. The ashing procedure consisted of three steps: boiling with HNO<sub>3</sub> for solubilization and partial oxidation; ashing at 500 °C with Mg(NO<sub>3</sub>)<sub>2</sub> to complete the oxidation and decompose remaining organic matter; and heating with 20 mL of 50 percent (v/v) HCl to dissolve the ash and chemically reduce selenium to the selenite (Se<sup>+4</sup>) oxidation state required for detection by hydride generation atomic absorption spectrophotometry. Following reduction, digestates were diluted to ~100 mL with DI water, yielding a final acid matrix of 10 percent HCl.

Selenite + Selenate in Water: Ten mL of filtered water and 5 mL of concentrated HCl were placed in a 25-mL borosilicate test tube and heated to about 130 °C in a well incubator block for 3 to 4 hours. After cooling, the liquid was transferred into a 125-mL polyethylene bottle, and the final volume was adjusted to 50 mL with DI water. The final matrix was 10 percent HCl.

*Filtered Particulates*: A dried filter containing particulates was rolled up, cut into pieces, and the entire filter was put into a 100-mL glass beaker. The filter was then subjected to the ashing procedure as described earlier for total selenium in water. The same procedure was conducted on clean filters, which served as blanks.

*Biota, Detritus, and Sediment*: An approximately 0.25gram (g) aliquant of each dried sample was subjected to a  $HNO_3-Mg(NO_3)_2$  ashing procedure, followed by HCl chemical reduction of selenate to selenite for the determination of selenium. The steps in the procedure were the same as those described above for total selenium in water. Digestates were diluted to about 100 mL with DI water, yielding a final acid matrix of 10 percent HCl.

#### **Instrumental Analysis**

*Total Selenium*: Total selenium was determined in all ashed samples by flow injection hydride generation atomic absorption spectrophotometry (FIHGAAS). In this procedure, the digestate is mixed with an HCl-carrier solution and then reduced by sodium tetrahydridoborate that has been stabilized with sodium hydroxide. Selenium in the sample is converted to volatile hydrogen selenide and transferred with argon carrier gas into a heated quartz cell mounted on an atomic absorption spectrophotometer for decomposition into atomic vapor and measurement.

*Selenite in Water*: An aliquot of each filtered water sample was analyzed directly by FIHGAAS after acidification to 10 percent HCl.

Selenate and Selenite in Water: Samples prepared in this manner were analyzed directly by FIHGAAS to provide selenate + selenite concentrations. The selenate concentration was calculated by difference using the formula:

$$selenate = (selenate + selenite) - selenite.$$
 (1)

*Particulate Selenium in Water*: Selenium associated with filtered particulates was determined by analyzing ashed filters by FIHGAAS. The mass of selenium in micrograms for the particulates was divided by the volume of water filtered for each drain site (0.5 or 1 L) to produce a microgram per liter concentration.

*Dissolved Organic Selenium in Water*: Dissolved organic selenium was estimated using the following formula:

Total dissolved selenium is defined as the analysis of filtered water for total selenium.

Total Suspended Solids: Upon arrival at the laboratory, all TSS samples were transferred to the Ecology Branch for TSS analysis. TSS were analyzed with methods recommended by the American Public Health Association (1998). Samples were brought to room temperature and mixed with a magnetic stirrer and subsequent manual inversions of the sample container. The sample was measured into a graduated cylinder, poured into a filtration apparatus, and filtered through a ProWeigh® glass fiber filter. The samples were prewashed three times in DI water, dried at 105 °C, and weighed to the nearest 0.1 milligram (mg). Sample volume varied to yield a dried residue between 2.5 and 200 mg. For each volume of sample used, an equal volume of DI water also was filtered for a blank determination. After filtering, large or nonhomogeneous materials were removed from the filter and the filter was rinsed with three 10-mL aliquots of DI water. Filters were then dried for at least 1 hour in a 103 to 105 °C oven and cooled to room temperature in a desiccator, then filter and residue were weighed to the nearest 0.1 mg. Drying, cooling, and weighing of the filter were repeated until the weight difference was less than (<) 4 percent or 0.5 mg, whichever was less. The average of these weights was used to determine the constant weight of the filter and residue, which was then corrected for any weight gain or loss of the blank. After subtracting the filter weight, this blank-corrected dried residue in milligrams was divided by the sample volume in liters to yield TSS in milligrams per liter (mg/L).

Particle-Size Analysis: Sediment samples designated for PSA were transferred to the USGS Ecology Branch upon arrival. The method requires use of a Bouyoucos hydrometer, adapted from American Society for Testing and Materials (2003). Wet sediment was sieved through a 2-mm sieve to remove any particles larger than coarse sand and then dried at 60 °C using a convectional drying oven. Approximately 100 g of dried sediment was mixed with 250 mL of DI water and 100 mL of a 50 mg/L sodium hexametaphosphate solution. A stir bar was then added and the mixture was stirred with a magnetic stirring plate. After calibrating the hydrometer, the suspended sediment mixture was transferred to a sedimentation cylinder and the volume adjusted to 1 L with DI water. After allowing for thermal equilibration, the temperature was recorded. Cylinder contents were then thoroughly mixed, and the hydrometer was inserted into the suspension. The meniscus reading was taken after 30 seconds and the hydrometer was removed and dried. After 120 minutes, the hydrometer was reinserted and the meniscus read again. All hydrometer meniscus readings were corrected by adjusting +0.25 for each degree above 18 °C and -0.25 for each degree below 18 °C. Percent fractions were determined as follows:

grams sand = sediment dry weight – (corrected 30 second reading – corrected calibration); percent sand = grams sand/ sediment dry weight x 100;

grams clay = sediment dry weight – (corrected 120 minute reading – corrected calibration); percent clay = grams clay/ sediment dry weight x 100; and percent silt = 100 - (percent sand + percent clay).

Total Organic Carbon: TOC was determined with a Universal Instruments Corporation (UIC) Model 5014 Coulometer that determines carbon in any carbon dioxide  $(CO_2)$ containing gas stream (Universal Instruments Corporation, 1999). The coulometer is used as a detector with different carbon front-end units and can detect carbon in the range of 0.01 micrograms (µg) to 100 mg. The coulometer cell is filled with a proprietary solution containing monoethanolamine and a colorimetric pH indicator. Platinum (cathode) and silver (anode) electrodes are positioned in the cell. The cell assembly is then placed in the coulometer cell compartment between a light source and a photodetector in the coulometer. As a CO<sub>2</sub> gas stream passes into the cell, the CO<sub>2</sub> is quantitatively absorbed and reacts with the monoethanolamine to form a titratable acid. This acid causes the color indicator to fade. A photodetector monitors the change in the color of the solution as a percent transmittance (percent T). As the percent T increases, the titration current automatically is activated to electrochemically generate base at a rate proportional to the percent T (approximately 1,500 µg carbon/minute). When the solution returns to its original color (original percent T), the current stops.

For TOC analysis, total carbon (TC,  $\mu$ g/mg) and total inorganic carbon (TIC,  $\mu$ g/mg) are determined. Total carbon is determined by combustion of weighed sediments at 925 °C. In TIC analysis, weighed sediments are exposed to heated 2 N sulfuric acid. Any inorganic carbonates are chemically reduced to mineral components and CO<sub>2</sub> gas. The gas is carried in high purity oxygen to the coulometer cell, where it is measured by the procedure described above. Percent TOC is calculated as follows:

Percent TOC = 
$$[(TC - TIC)/TC] \times 100$$
 (3)

### **Quality Assurance**

Samples were processed through the preparative and analytical flow scheme in 11 analytical blocks for selenium, 2 blocks for TSS, and 1 block each for PSA and TOC. Each block was assigned a block initiation date (BID) used to identify samples and quality-control samples/materials prepared and analyzed collectively as a unit. For samples analyzed by atomic absorption for total selenium, predigestion quality control included digestion blanks, replicates, spikes, and reference solutions. Analytical quality-control for selenium included calibration verification solutions, replicate analyses, and analysis spikes. Quality control for the TSS, PSA, and TOC determinations included reference materials, duplicates, and replicates.

### Results

*Total Selenium*: Total selenium concentrations [micrograms per liter,  $(\mu g/L)$ ] in unfiltered water samples for the October 2008 samples are listed in table 1. Mean selenium concentrations were most elevated in water from Trifolium 22 drain (8.04), followed by P drain (7.07). The lowest mean selenium concentration was from San Felipe Wash drain (1.00). Data for the January 2009 samples are listed in table 2. Mean selenium concentrations were highest in Trifolium Storm drain (33.6), followed by Trifolium 18 drain (19.7), and Q drain (11.4). The lowest mean selenium concentration was from U drain (1.26).

Total Dissolved Selenium and Selenium Species: Dissolved selenite, dissolved selenate, dissolved organic selenium, total dissolved selenium, and particulate selenium concentrations ( $\mu$ g/L) from filtered water samples collected during the October 2008 sampling are presented in table 3. The dissolved organic selenium fraction is assumed to include seleno-amino acids and dissolved seleno-peptides, Se(0) as a pseudo-dissolved microcolloid, and inorganic Se(-II) species (Cutter and Bruland, 1984). Speciation measurements revealed that selenium in the sampled drains exists predominately as selenate (61 to 93 percent) followed by selenite (4 to 27 percent), typical of waters where selenium is leached out of selenium containing marine shales and associated soils under alkaline and oxidizing conditions.

*Total Suspended Solids*: TSS concentrations (mg/L) in unfiltered water collected during the October 2008 and January 2009 samplings are presented in table 4. TSS concentrations ranged from a high of 192 (T drain) to a low of 6.1 (Former Trifolium 20 drain) for the October 2008 collection, and a high of 216 (Vail 5 drain) to a low of 9.00 (Niland 4 drain) for the January 2009 collection.

*Biota*: Percent moisture and concentrations of selenium [micrograms per gram ( $\mu$ g/g) dry weight] in biota (algae, plankton, midge larvae, western mosquitofish, and sailfin molly) are presented in table 5. Selenium concentration ranges for each matrix were as follows: algae, 1.68 to 8.26; plankton, 0.79 to 3.66; midges, 2.68 to 50.6; and fish, 3.09 to 30.4.

Detritus and Sediment: Percent moisture and selenium concentrations ( $\mu$ g/g dry weight) in detritus and sediment are presented in table 6. Selenium in detritus ranged from 1.78 to

Table 1.Total selenium concentrations in duplicates of unfiltered irrigation drain water samples, Salton Sea, California,<br/>October 2008.

[USGS, United States Geological Survey; ID, identification; Rep, field replicate;  $\mu g/L$ , microgram per liter; SD, standard deviation; ---, no data; <, less than; nc, not collected]

			_	Total se	elenium concer	tration	
USGS ID	Field ID	Drain name/ID	Collection date	Rep 1 (µg/L)	Rep 2 (µg/L)	Mean (μg/L)	SD
44440	BLANK-1		10/23/08	< 0.15			
44441	BLANK-2		10/23/08	< 0.15			
44442	BLNDWATSE14		10/16/08	4.31			
44443	BLNDWATSE14B		10/21/08	2.98			
44205,44206	LKLNWATSE14	Lack & Linsey Pond	10/17/08	3.64	3.53	3.58	0.08
44181,44182	NLD1WATSE14	Niland 1	10/16/08	1.53	1.53	1.53	0.00
44183,44184	NLD2WATSE14	Niland 2	10/16/08	2.17	2.19	2.18	0.02
44185,44186	NLD3WATSE14	Niland 3	10/16/08	1.87	1.92	1.89	0.03
44187,44188	NLD4WATSE14	Niland 4	10/16/08	2.26	2.39	2.33	0.09
nc <sup>1</sup>	OOOOWATSE14	0					
44199,44200	PPPPWATSE14	Р	10/17/08	7.14	7.00	7.07	0.10
nc <sup>1</sup>	POEDWATSE14	Poe Rd					
44203,44204	PUMCWATSE14	Pumice	10/17/08	4.90	4.86	4.88	0.03
44197,44198	QQQQWATSE14	Q	10/17/08	3.67	3.79	3.73	0.09
44195,44196	RRRRWATSE14	R	10/17/08	2.13	2.01	2.07	0.08
44193,44194	SSSSWATSE14	S	10/17/08	2.72	2.65	2.68	0.05
44448,44449	SFWHWATSE14	San Felipe Wash	10/18/08	0.98	1.03	1.00	0.03
$nc^1$	TTTTWATSE14	Т					
44452,44453	TR01WATSE14	Trifolium 1	10/21/08	4.96	4.97	4.97	0.01
44456,44457	TR12WATSE14	Trifolium 12	10/21/08	4.75	4.75	4.75	0.00
44454,44455	TR13WATSE14	Trifolium 13	10/19/08	3.50	3.59	3.54	0.06
$nc^1$	TR14WATSE14	Trifolium 14					
nc <sup>1</sup>	TR18WATSE14	Trifolium 18					
44460,44461	TR19WATSE14	Trifolium 19	10/18/08	1.90	1.82	1.86	0.06
44444,44445	FT20WATSE14	Former Trifolium 20	10/18/08	4.30	4.34	4.32	0.03
nc <sup>1</sup>	TR20WATSE14	Trifolium 20					
44446,44447	TR22WATSE14	Trifolium 22	10/18/08	8.03	8.05	8.04	0.01
44450,44451	TR23WATSE14	Trifolium 23	10/18/08	3.77	3.83	3.80	0.04
44458,44459	TRSTWATSE14	Trifolium Storm	10/21/08	3.15	3.06	3.10	0.06
44191,44192	UUUUWATSE14	U	10/16/08	1.70	1.66	1.68	0.03
44201,44202	VL05WATSE14	Vail 5	10/17/08	4.73	5.02	4.88	0.20
44189,44190	WWWWWATSE14	W	10/16/08	4.39	4.40	4.40	0.002
nc <sup>1</sup>	ZSPLWATSE14	Z Spill					

<sup>1</sup>Drain was one of seven selected for intensive sampling (see table 3).

**Table 2.**Total selenium concentrations in duplicates of unfiltered irrigation drain water samples, Salton Sea, California,<br/>January 2009.

[USGS, United States Geological Survey; ID, identification; Rep, field replicate; µg/L, micrograms per liter; SD, standard deviation; <, less than]

		Drain	Collection –	Total s	Total selenium concentration		
USGS ID	Field ID	name/ID	date	Rep 1 (µg/L)	Rep 2 (µg/L)	Mean (µg/L)	SD
45098	Blank 1		01/13/09	< 0.080			
45099	Blank 2		01/13/09	< 0.080			
45100	BLNDWATSE15		01/08/09	3.02			
45101	BLNDWATSE15B		01/10/09	4.72			
45104,45105	LKLNWATSE15	Lack & Linsey Pond	01/10/09	5.05	5.03	5.04	0.01
45106,45107	NLD1WATSE15	Niland 1	01/08/09	1.41	1.48	1.44	0.05
45108,45109	NLD2WATSE15	Niland 2	01/08/09	1.76	1.74	1.75	0.02
45110,45111	NLD3WATSE15	Niland 3	01/08/09	1.58	1.64	1.61	0.05
45112,45113	NLD4WATSE15	Niland 4	01/08/09	2.91	2.72	2.82	0.13
45114,45115	OOOOWATSE15	0	01/10/09	3.59	3.74	3.66	0.11
45118,45119	PPPPWATSE15	Р	01/08/09	2.14	2.17	2.16	0.02
45116,45117	POEDWATSE15	Poe Rd	01/09/09	4.57	4.39	4.48	0.13
45120,45121	PUMCWATSE15	Pumice	01/11/09	2.11	2.68	2.39	0.40
45122,45123	QQQQWATSE15	Q	01/08/09	11.5	11.3	11.4	0.14
45124,45125	RRRRWATSE15	R	01/08/09	1.60	1.45	1.53	0.11
45128,45129	SSSSWATSE15	S	01/08/09	2.56	2.48	2.52	0.05
45126,45127	SFWHWATSE15	San Felipe Wash	01/10/09	2.45	2.07	2.26	0.27
45150,45151	TTTTWATSE15	Т	01/08/09	1.40	1.25	1.33	0.11
45130,45131	TR01WATSE15	Trifolium 1	01/09/09	5.80	5.72	5.76	0.05
45132,45133	TR12WATSE15	Trifolium 12	01/09/09	4.18	4.33	4.26	0.10
45134,45135	TR13WATSE15	Trifolium 13	01/09/09	5.04	4.78	4.91	0.19
45136,45137	TR14WATSE15	Trifolium 14	01/09/09	3.34	3.10	3.22	0.17
45138,45139	TR18WATSE15	Trifolium 18	01/09/09	19.6	19.8	19.7	0.08
45140,45141	TR19WATSE15	Trifolium 19	01/10/09	3.51	3.47	3.49	0.02
45102,45103	FT20WATSE15	Former Trifolium 20	01/10/09	4.54	4.28	4.41	0.18
45142,45143	TR20WATSE15	Trifolium 20	01/10/09	5.07	4.95	5.01	0.09
45144,45145	TR22WATSE15	Trifolium 22	01/10/09	4.85	4.68	4.76	0.12
45146,45147	TR23WATSE15	Trifolium 23	01/10/09	3.32	3.31	3.31	0.005
45148,45149	TRSTWATSE15	Trifolium Storm	01/09/09	33.6	33.6	33.6	0.04
45152,45153	UUUUWATSE15	U	01/08/09	1.33	1.19	1.26	0.11
45154,45155	VL05WATSE15	Vail 5	01/11/09	5.21	4.81	5.01	0.28
45156,45157	WWWWWATSE15	W	01/08/09	3.05	2.93	2.99	0.09
45158,45159	ZSPLWATSE15	Z Spill	01/08/09	4.21	4.12	4.16	0.06

# Table 3. Total dissolved selenium, dissolved selenium species, and particulate selenium concentrations in filtered irrigation drain water samples, Salton Sea, California, October 2008.

USGS ID	Field ID	Drain name/ID	Rep	Collection fate	Dissolved [Se03]-2 (µg/L)	<sup>1</sup> Calculated dissolved [Se04]-2 (µg/L)	²Calculated dissolved organic Se (µg/L)	Measured total dis- solved Se (µg/L)	<sup>3</sup> Measured particulate Se (µg/L)	⁴Calculated total Se (µg/L)
44463	BLNKWADSE14		1	10/19/08	< 0.072	< 0.11		< 0.38	< 0.004	
44464	BLNKWADSE14B		2	10/19/08	< 0.072	< 0.11		< 0.38		
44462	BLNDWADSE14			10/16/08	0.63	3.65	0.11	4.39	0.103	4.49
44207	OOOOWADSE14	0	1	10/15/08	0.71	3.39	0.10	4.20	0.043	4.24
44208	OOOOWADSE14B	0	2	10/15/08	0.72	3.53	0.00	4.22	0.063	4.28
44465	POEDWADSE14	Poe Rd	1	10/20/08	1.81	7.40	0.94	10.1	0.100	10.25
44466	POEDWADSE14B	Poe Rd	2	10/20/08	1.96	7.83	0.36	10.2	0.065	10.22
44209	TTTTWADSE14	Т	1	10/17/08	0.35	1.06	0.22	1.63	0.123	1.75
44210	TTTTWADSE14B	Т	2	10/17/08	0.34	1.06	0.11	1.50	0.091	1.59
44467	TR14WADSE14	Trifolium 14	1	10/19/08	0.30	7.7	0.21	8.21	0.044	8.26
44468	TR14WADSE14B	Trifolium 14	2	10/19/08	0.35	7.1	0.22	7.67	0.054	7.72
44469	TR18WADSE14	Trifolium 18	1	10/21/08	1.52	12.8	0.49	14.8	0.051	14.8
44470	TR18WADSE14B	Trifolium 18	2	10/21/08	1.55	12.9	0.31	14.8	0.027	14.8
44471	TR20WADSE14	Trifolium 20	1	10/18/08	0.56	1.45	0.01	2.01	0.070	2.08
44472	TR20WADSE14B	Trifolium 20	2	10/18/08	0.55	1.44	0.22	2.22	0.085	2.30
44211	ZSPLWADSE14	Z Spill	1	10/16/08	0.57	3.66	0.18	4.41	0.108	4.52
44212	ZSPLWADSE14B	Z Spill	2	10/16/08	0.58	3.74	0.04	4.37	0.095	4.46

[USGS, United States Geological Survey; ID, identification; Rep, replicate; [Se03]-2, selenite; [Se04]-2, selenite; Se, selenium; µg/L, micrograms per liter; ---, no data; <, less than]

<sup>1</sup>Calculated dissolved  $[Se0_4]^{-2}$  = measured  $([Se0_4]^{-2} + [Se0_3]^{-2})$  - measured  $[Se0_3]^{-2}$ .

<sup>2</sup>Calculated dissolved Organic Se = measured total dissolved Se - measured ( $[Se0_4]^{-2} + [Se0_3]^{-2}$ ).

<sup>3</sup>Measured particulate Se =  $\mu$ g of Se in filtered particulates divided by volume of site water filtered.

<sup>4</sup>Calculated total Se = measured particulate Se + measured total dissolved Se.

 Table 4.
 Total suspended solids concentrations in unfiltered Salton Sea, California, irrigation drain water samples, October 2008

 and January 2009.
 2009.

[USGS, United States Geological Survey; ID, identification; TSS, total suspended solids; mg/L, milligram per liter; bold and italicized values are less than the method quantitation limit and have high uncertainty]

	Ducin	October, 2008		January, 2009		
Field ID	Drain — name/ID	USGS ID	TSS (mg/L)	USGS ID	TSS (mg/L)	
BLNDWATSSA	Blind A	44178	99.	45067	32.	
BLNDWATSSB	Blind B	44407	29.	45068	40.	
LKLNWATSS	Lack & Linsey Pond	44180	93.	45070	46.	
NLD1WATSS	Niland 1	44164	83.	45071	36.	
NLD2WATSS	Niland 2	44165	20.	45072	41.	
NLD3WATSS	Niland 3	44166	64.	45073	43.	
NLD4WATSS	Niland 4	44167	47.	45074	9.0	
DOOOWATSS	0	44176	118.	45075	35.	
PPPPWATSS	Р	44175	86.	45077	115.	
POEDWATSS	Poe Rd	44400	77.	45076	179.	
PUMCWATSS	Pumice	44177	151.	45078	99.	
QQQQWATSS	Q	44174	28.	45079	33.	
RRRWATSS	R	44173	87.	45080	59.	
SSSSWATSS	S	44172	92.	45082	48.	
SFWHWATSS	San Felipe Wash	44397	44.	45081	13.	
ITTTWATSS	Т	44171	192.	45093	54.	
TR01WATSS	Trifolium 1	44408	19.	45083	66.	
TR12WATSS	Trifolium 12	44404	33.	45084	213.	
FR13WATSS	Trifolium 13	44403	40.	45085	84.	
R14WATSS	Trifolium 14	44406	94.	45086	55.	
TR18WATSS	Trifolium 18	44399	7.2	45087	23.	
TR19WATSS	Trifolium 19	44401	16.	45088	40.	
FT20WATSS	Former Trifolium 20	44395	6.1	45069	43.	
TR20WATSS	Trifolium 20	44402	78.	45089	66.	
FR22WATSS	Trifolium 22	44396	21.	45090	62.	
TR23WATSS	Trifolium 23	44398	28.	45091	114.	
RSTWATSS	Trifolium Storm	44405	17.	45092	186.	
JUUUWATSS	U	44170	40.	45094	28.	
/LO5WATSS	Vail 5	44179	31.	45095	216.	
WWWWWATSS	W	44169	112.	45096	153.	
ZSPLWATSS	Z Spill	44168	89.	45097	45.	

<sup>1</sup>Trifolium 1 drain was dry at time of collection.

 Table 5.
 Selenium concentrations in biota samples collected from Salton Sea, California, irrigation drains, October 2008.

	C 1 1 10 ID	1		1 ( )
UNGN United States	( reological Survey: 11)	identification: IIg/g	microgram per o	pram: no data l
[0000, Onica States	Geological Survey; ID	, identification, $\mu_{\rm B}$ s,	interogram per g	Sium, , no uuui

USGS ID	Field ID	Matrix	Drain name/ID	Moisture (percent)	Selenium (µg/g dry weight)
44621	OOOOALGTSE14A	algae	0	70.5	3.28
44622	OOOOALGTSE14B	algae	0	70.2	3.38
44623	OOOOALGTSE14C	algae	0	70.5	3.46
44618	POEDALGTSE14A	algae	Poe	56.8	2.26
44619	POEDALGTSE14B	algae	Poe	56.0	2.31
44620	POEDALGTSE14C	algae	Poe	55.2	2.29
44612	TTTTALGTSE14A	algae	Т	75.4	4.99
44613	TTTTALGTSE14B	algae	Т	76.6	5.76
44614	TTTTALGTSE14C	algae	Т	75.4	5.58
44624	TR14ALGTSE14A	algae	Trifolium 14	63.3	1.88
44625	TR14ALGTSE14B	algae	Trifolium 14	63.1	1.89
44626	TR14ALGTSE14C	algae	Trifolium 14	62.8	1.82
44630	TR18ALGTSE14A	algae	Trifolium 18	66.4	7.84
44631	TR18ALGTSE14B	algae	Trifolium 18	66.5	8.05
44632	TR18ALGTSE14C	algae	Trifolium 18	65.7	8.26
44615	TR20ALGTSE14A	algae	Trifolium 20	67.3	2.65
44616	TR20ALGTSE14B	algae	Trifolium 20	64.9	2.68
44617	TR20ALGTSE14C	algae	Trifolium 20	66.0	2.91
44627	ZSPLALGTSE14A	algae	Z Spill	67.8	1.68
44628	ZSPLALGTSE14B	algae	Z Spill	65.5	1.52
44629	ZSPLALGTSE14C	algae	Z Spill	68.9	1.76
44684	OOOONPTSE14A	plankton	0		2.72
44685	OOOONPTSE14B	plankton	0		3.12
44686	OOOONPTSE14C	plankton	0		2.44
44681	POEDNPTSE14A	plankton	Poe		1.45
44682	POEDNPTSE14B	plankton	Poe		1.10
44683	POEDNPTSE14C	plankton	Poe		0.79
44675	TTTTNPTSE14A	plankton	Т		2.99
44676	TTTTNPTSE14B	plankton	Т		2.82
44677	TTTTNPTSE14C	plankton	Т		3.45
44687	TR14NPTSE14A	plankton	Trifolium 14		2.56
44688	TR14NPTSE14B	plankton	Trifolium 14		2.15
44689	TR14NPTSE14C	plankton	Trifolium 14		1.81
44693	TR18NPTSE14A	plankton	Trifolium 18		1.88
44694	TR18NPTSE14B	plankton	Trifolium 18		2.53
44695	TR18NPTSE14C	plankton	Trifolium 18		2.59
44678	TR20NPTSE14A	plankton	Trifolium 20		1.16
44679	TR20NPTSE14B	plankton	Trifolium 20		3.66
44689	TR20NPTSE14C	plankton	Trifolium 20		1.95
44690	ZSPLNPTSE14A	plankton	Z Spill		3.01
44691	ZSPLNPTSE14B	plankton	Z Spill		2.62

**Table 5.**Selenium concentrations in biota samples collected from Salton Sea, California, irrigation drains, October 2008.—Continued

[USGS, United States Ge	ological Survey; ID, identification;	; µg/g, microgram per grar	n;, no data]

USGS ID	Field ID	Matrix	Drain name/ID	Moisture (percent)	Selenium (µg/g dry weight
44692	ZSPLNPTSE14C	plankton	Z Spill		2.45
44642	OOOOCHITSE14A	midge	0	81.8	3.06
44643	OOOOCHITSE14B	midge	0	82.0	2.68
44644	OOOOCHITSE14C	midge	0	81.5	2.97
44639	POEDCHITSE14A	midge	Poe	83.0	17.2
44640	POEDCHITSE14B	midge	Poe	81.1	15.2
44641	POEDCHITSE14C	midge	Poe	84.0	18.2
44633	TTTTCHITSE14A	midge	Т	81.3	6.37
44634	TTTTCHITSE14B	midge	Т	80.8	6.66
44635	TTTTCHITSE14C	midge	Т	81.6	6.43
44645	TR14CHITSE14A	midge	Trifolium 14	80.2	7.09
44646	TR14CHITSE14B	midge	Trifolium 14	79.8	7.72
44647	TR14CHITSE14C	midge	Trifolium 14	80.3	6.55
44651	TR18CHITSE14A	midge	Trifolium 18	84.6	44.2
44652	TR18CHITSE14B	midge	Trifolium 18	84.3	44.0
44653	TR18CHITSE14C	midge	Trifolium 18	85.0	50.6
44636	TR20CHITSE14A	midge	Trifolium 20	82.0	6.59
44637	TR20CHITSE14B	midge	Trifolium 20	80.4	6.09
44638	TR20CHITSE14C	midge	Trifolium 20	82.7	6.47
44648	ZSPLCHITSE14A	midge	Z Spill	79.4	4.15
44649	ZSPLCHITSE14B	midge	Z Spill	79.2	3.63
44650	ZSPLCHITSE14C	midge	Z Spill	77.0	3.78
44588	OOOOGMBTSE14A	gambusia	О	78.5	4.41
44589	OOOOGMBTSE14B	gambusia	0	77.8	4.24
44590	OOOOGMBTSE14C	gambusia	0	78.8	4.56
44591	OOOOSLMTSE14A	sailfin molly	0	78.5	3.09
44592	OOOOSLMTSE14B	sailfin molly	0	78.7	4.11
44593	OOOOSLMTSE14C	sailfin molly	0	78.8	3.71
44582	POEDGMBTSE14A	gambusia	Poe	77.2	16.6
44583	POEDGMBTSE14B	gambusia	Poe	76.6	15.2
44584	POEDGMBTSE14C	gambusia	Poe	77.3	13.2
44585	POEDSLMTSE14A	sailfin molly	Poe	78.8	16.0
44586	POEDSLMTSE14B	sailfin molly	Poe	78.9	17.2
44587	POEDSLMTSE14C	sailfin molly	Poe	77.8	15.2
44570	TTTTGMBTSE14A	gambusia	Т	74.8	6.23
44571	TTTTGMBTSE14B	gambusia	Т	75.1	6.07
44572	TTTTGMBTSE14C	gambusia	Т	75.3	6.08
44573	TTTTSLMTSE14A	sailfin molly	Т	75.8	5.14
44574	TTTTSLMTSE14B	sailfin molly	Т	76.4	6.44
44575	TTTTSLMTSE14C	sailfin molly	Т	75.7	5.31
44594	TR14GMBTSE14A	gambusia	Trifolium 14	77.7	4.07

Table 5.Selenium concentrations in biota samples collected from Salton Sea, California, irrigation drains, October 2008.—Continued

USGS ID	Field ID	Matrix	Drain name/ID	Moisture (percent)	Selenium (µg/g dry weight)
44595	TR14GMBTSE14B	gambusia	Trifolium 14	78.2	4.54
44596	TR14GMBTSE14C	gambusia	Trifolium 14	77.3	4.29
44597	TR14SLMTSE14A	sailfin molly	Trifolium 14	75.9	4.59
44598	TR14SLMTSE14B	sailfin molly	Trifolium 14	76.1	4.65
44599	TR14SLMTSE14C	sailfin molly	Trifolium 14	76.3	4.79
44606	TR18GMBTSE14A	gambusia	Trifolium 18	76.7	17.4
44607	TR18GMBTSE14B	gambusia	Trifolium 18	75.4	17.8
44608	TR18GMBTSE14C	gambusia	Trifolium 18	75.7	19.2
44609	TR18SLMTSE14A	sailfin molly	Trifolium 18	78.8	30.4
44610	TR18SLMTSE14B	sailfin molly	Trifolium 18	77.9	25.3
44611	TR18SLMTSE14C	sailfin molly	Trifolium 18	77.0	25.0
44576	TR20GMBTSE14A	gambusia	Trifolium 20	75.4	5.33
44577	TR20GMBTSE14B	gambusia	Trifolium 20	78.0	5.96
44578	TR20GMBTSE14C	gambusia	Trifolium 20	76.5	6.13
44579	TR20SLMTSE14A	sailfin molly	Trifolium 20	76.3	7.57
44580	TR20SLMTSE14B	sailfin molly	Trifolium 20	76.7	7.70
44581	TR20SLMTSE14C	sailfin molly	Trifolium 20	75.4	7.14
44600	ZSPLGMBTSE14A	gambusia	Z Spill	77.6	6.77
44601	ZSPLGMBTSE14B	gambusia	Z Spill	79.1	6.61
44602	ZSPLGMBTSE14C	gambusia	Z Spill	79.4	6.18
44603	ZSPLSLMTSE14A	sailfin molly	Z Spill	75.9	3.93
44604	ZSPLSLMTSE14B	sailfin molly	Z Spill	76.4	4.84
44605	ZSPLSLMTSE14C	sailfin molly	Z Spill	77.1	5.31

[USGS, United States Geological Survey; ID, identification; µg/g, microgram per gram; ---, no data]

**Table 6.**Selenium concentrations in detritus and sediment samples collected from Salton Sea, California, irrigation drains,<br/>October 2008.

<b>[USGS, United States</b> ]	Geological Survey: ID	. identification: ug/g	, micrograms per gram]
[0000, 011104 014100	Geologieal Survey, 12	, 100110110000, 48 8	, merograms per gramj

USGS ID	Field ID	Matrix	Drain name/ID	Moisture (percent)	Selenium (µg/g dry weight)
44663	OOOODETTSE14A	detritus	0	86.3	3.82
44664	OOOODETTSE14B	detritus	0	85.7	4.85
44665	OOOODETTSE14C	detritus	0	85.9	6.42
44660	POEDDETTSE14A	detritus	Poe	74.4	7.10
44661	POEDDETTSE14B	detritus	Poe	75.3	7.09
44662	POEDDETTSE14C	detritus	Poe	71.8	6.59
44654	TTTTDETTSE14A	detritus	Т	77.6	2.52
44655	TTTTDETTSE14B	detritus	Т	77.2	1.78
44656	TTTTDETTSE14C	detritus	Т	79.6	2.22
14666	TR14DETTSE14A	detritus	Trifolium 14	80.7	9.86
14667	TR14DETTSE14B	detritus	Trifolium 14	82.1	5.14
14668	TR14DETTSE14C	detritus	Trifolium 14	80.6	6.42
4672	TR18DETTSE14A	detritus	Trifolium 18	82.4	39.7
4673	TR18DETTSE14B	detritus	Trifolium 18	83.8	27.4
14674	TR18DETTSE14C	detritus	Trifolium 18	80.5	58.0
14657	TR20DETTSE14A	detritus	Trifolium 20	79.0	3.94
14658	TR20DETTSE14B	detritus	Trifolium 20	77.6	12.4
14659	TR20DETTSE14C	detritus	Trifolium 20	79.2	4.07
14669	ZSPLDETTSE14A	detritus	Z Spill	83.2	4.00
14670	ZSPLDETTSE14B	detritus	Z Spill	82.2	3.14
44671	ZSPLDETTSE14C	detritus	Z Spill	79.6	3.57
14424	BLNDSDTSE14	sediment	Blind	65.4	9.95
44421	OOOOSDTSE14	sediment	0	48.7	0.84
44418	POEDSDTSE14	sediment	Poe	23.1	0.42
14422	TTTTSDTSE14	sediment	Т	50.6	0.72
14420	TR14SDTSE14	sediment	Trifolium 14	50.7	1.90
44419	TR18SDTSE14	sediment	Trifolium 18	65.8	10.0
44417	TR20SDTSE14	sediment	Trifolium 20	30.9	1.10
44423	ZSPLSDTSE14	sediment	Z Spill	42.3	0.65

58.0 and from 0.42 to 10.0 in sediment. The particle size analyses of sediments, expressed as percent sand, silt, and clay, are presented in table 7. Percent sand ranged from 14 to 76; percent silt from 15 to 57; and percent clay from 7.5 to 50. Percent TOC in sediments is given in table 8 and ranged from 0.2 to 1.5.

## **Quality Control Results**

Calibration Verification: During the selenium determinations, a calibration verification solution (Spex Claritas PPT<sup>®</sup>; Cat No. CLSe2-2Y) was analyzed at the beginning and end of each analytical run. Calibration was considered acceptable if the check solution was within plus or minus 10 percent of the actual concentration (3  $\mu$ g/L), which was achieved during all analyses.

*Reference Materials*: Recoveries of selenium from QC Plus <sup>+</sup> Trace Metals Quality Control Standard [n=7

(7 samples)] and National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1643e Trace Elements in Water (n=3) averaged 103 percent. The recoveries of selenium from National Research Council of Canada (NRCC) SRM PACS-1 marine sediment (n=1) was 100 percent. The International Atomic Energy Agency (IAEA) copepod reference material MA-A-1 (n=2) and the Institute for Reference Materials and Measurements Certified Reference Material (CRM) 414 Trace Elements in Plankton (n=2) all exhibited selenium recoveries of 100 percent. Recoveries of selenium in NRCC CRM DORM-2 dogfish muscle (n=1) and IAEA CRM 407 whole-body fish (n=1) were 100 and 91 percent, respectively. Recoveries of TSS from a TSS reference solution (Environmental Resource Associates Hardness Wastewater Standard 507; n=5) averaged 101 percent. The recovery of total carbon from a carbon reference material (Environmental Resource Associates Nutrients in Soil 542; n=1) was 100 percent. Recoveries of percent sand, silt, and clay from a CERC research sediment material ranged from 79 to 99 percent.

Table 7. Particle size distributions in sediment samples collected from Salton Sea, California, irrigation drains, October 2008.

		Dusin	Particle size category			
USGS ID	Field ID	Drain – name/ID	> 2 mm (percent)	Sand (percent)	Silt (percent)	Clay (percent)
44416	BLNDSDTOC14	Blind	0.	55.	37.	7.
44413	OOOOSDTOC14	0	0.	24.	37.	39.
44410	POEDSDTOC14	Poe Rd	0.	76.	15.	10.
44414	TTTTSDTOC14	Т	0.	15.	36.	50.
44412	TR14SDTOC14	Trifolium 14	0.	14.	57.	29.
44411	TR18SDTOC14	Trifolium 18	0.	55.	37.	8.
44409	TR20SDTOC14	Trifolium 20	1.	47.	27.	25.
44415	ZSPLSDTOC14	Z Spill	7.	15.	33.	45.

[USGS, United States Geological Survey; ID, identification; >, greater than; mm, millimeter]

**Table 8.** Percent total organic carbon in sediment samples collected from Salton Sea, California, irrigation drains, October 2008.[USGS, United States Geological Survey; ID, identification]

USGS ID	Field ID	Drain name/ID	Total organic carbon (percent)
42216	BLNDSDTOC14	Blind	1.5
42209	TR20SDTOC14	Trifolium 20	0.7
42210	POEDSDTOC14	Poe Rd.	0.2
42211	TR18SDTOC14	Trifolium 18	1.3
42212	TR14SDTOC14	Trifolium 14	1.1
42213	OOOOSDTOC14	0	0.6
42214	TTTTSDTOC14	Т	0.5
42215	ZSPLSDTOC14	Z Spill	1.2

Analytical and Method Precision: Instrumental precision for selenium as determined by repeated analysis of a standard throughout the run for each block (n=11) was less than 5 percent relative standard deviation (RSD). Relative percent differences (RPDs) between field duplicates (n=73) of either unfiltered or filtered water samples analyzed for selenium or selenium species mostly were  $\leq$  (less than or equal) 12 (n=70), but two of these duplicates exhibited greater RPDs of 15 and 24. Both of these RPDs were associated with relatively low selenium concentrations ( $< 5 \mu g/L$ ). RSDs for triplicate field samples of detritus (n=7), algae (n=7), plankton (n=7), midge larvae (n=7), and whole-body fish (n=14) analyzed for selenium were as follows: algae, 1.0 to 7.5 percent; plankton, 11 to 56 percent; detritus, 4.2 to 71 percent; midge larvae, 2.4 to 9.1 percent; and whole-body fish, 1.5 to 15 percent. Once again, many of the greatest RSDs were associated with low selenium concentrations, but in some instances greater variation apparently reflected greater natural variation of selenium in certain sample matrices, for example detritus and plankton. Laboratory method precision for triplicate (n=18) preparation and analysis of samples for selenium was <10 percent RSD. Instrumental precision was based on duplicate analysis of sample digestates from each sample matrix, which resulted in RPDs <2 percent. Duplicate analysis of water samples for TSS (n=4) resulted in RPDs ranging from 1.0 to 21 percent, whereas triplicate analysis for TSS (n=3) resulted in RSDs <5 percent. The duplicate analysis of a drain sediment for PSA resulted in RPDs <7 percent for the fractions.. Triplicate analyses of a drain sediment for PSA resulted in RSDs  $\leq 14$ percent for the fractions. The duplicate analysis of six drain sediments for TOC resulted in RPDs 6.9 to 22 percent for five drains, but one higher RPD of 82 percent, presumably because of a low TOC level ( $\leq 1$  percent) and the natural heterogeneity of the drain sediment. A replicate analysis of one drain sediment resulted in a RSD of 27 percent.

*Spikes*: Recoveries of selenium [selenite (Se<sup>+4</sup>), selenate (Se<sup>+6</sup>), or selenomethionine] spiked into a filter blanks (n=1) and water samples (n=21) ranged from 89 to 106 percent, averaging 98 percent. Recoveries of selenium spiked into sediment (n=2), detritus (n=4), and biota (n=10) ranged from 92 to 103 percent, averaging 99 percent. Recoveries of selenium spikes added to water during analysis (n=17) ranged from 97 to 110 percent, averaging 102 percent. Analysis spikes of sediment (n=1), filtered particulates (n=2), detritus (n=2), and biota (n=10) ranged from 98 to 106 percent, averaging 101 percent.

Blank Equivalent Concentrations: Blank equivalent concentrations (BECs) were computed for selenium for each matrix and for TSS blanks analyzed with each set of drain water samples. BECs for water and filter particulates were less than their respective method detection limits (MDLs). BECs ( $\mu$ g/g) for detritus, sediment, and all biota were greater than their respective MDLs ( $\mu$ g/g); however, BECs were inconsequential when compared to the lowest selenium concentrations in the samples of each matrix. The BECs and corresponding MDLs were as follows: detritus, 0.17 compared to 0.019; sediment, 0.17 compared to 0.009; fish, 0.16 compared to 0.022; algae, 0.17 compared to 0.012; midge, 0.17 compared to 0.012; plankton, 0.47 compared to 0.046. Except for plankton and sediment, these BECs are considered inconsequential relative to the lowest selenium concentrations in the samples of each matrix. BECs for TSS were less than their respective MDLs. TSS sample data were corrected for procedural blanks, whereas total selenium sample data were not blank corrected.

Instrument Detection, Method Detection, and Method Quantitation Limits: The FIHGAAS instrument detection limit for selenium was 0.02 and 0.033  $\mu$ g/L, and 0.04 mg for TSS. MDLs for each matrix for selenium were computed for each analytical block (n=15) using the formula:

$$3(SD_{b}^{2} + SD_{s}^{2})^{1/2}$$
 (4)

where

 $SD_{b}$  = standard deviation of a blank (n=3); and  $SD_{s}$  = standard deviation of a low level sample or

Calculated MDLs were: water, 0.071 to 0.38  $\mu$ g/L; filtered particulates, 0.004  $\mu$ g/L; sediment, 0.009  $\mu$ g/g dry weight; algae, 0.012  $\mu$ g/g dry weight; midge larvae, 0.012  $\mu$ g/g dry weight; plankton, 0.046  $\mu$ g/g dry weight; detritus, 0.019  $\mu$ g/g dry weight; and whole-body fish, 0.022  $\mu$ g/g dry weight. Method quantitation limits (MQLs) for each matrix were calculated as 3.3 x MDLs. MDLs for TSS were 3.09 and 5.24 mg/L and MQLs were 10.2 and 17.3 mg/L. Overall, quality-control results for the study were within acceptable limits as specified by USGS.

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