Arch Environ Contam Toxicol (2013) 64:52–64
DOI 10.1007/s00244-012-9815-y

Contaminants in Stream Sediments From Seven United States Metropolitan Areas: Part II—Sediment Toxicity to the Amphipod *Hyalella azteca* and the Midge *Chironomus dilutus*

Nile E. Kemble · Douglas K. Hardesty · Christopher G. Ingersoll · James L. Kunz · Paul K. Sibley · Daniel L. Calhoun · Robert J. Gilliom · Kathryn M. Kuivila · Lisa H. Nowell · Patrick W. Moran

Received: 21 January 2012 / Accepted: 24 August 2012 / Published online: 6 November 2012

Abstract Relationships between sediment toxicity and sediment chemistry were evaluated for 98 samples collected from seven metropolitan study areas across the United States. Sediment-toxicity tests were conducted with the amphipod *Hyalella azteca* (28 day exposures) and with the midge *Chironomus dilutus* (10 day exposures). Overall, 33 % of the samples were toxic to amphipods and 12 % of the samples were toxic to midge based on comparisons with reference conditions within each study area. Significant correlations were observed between toxicity end points and sediment concentrations of trace elements, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), or organochlorine (OC) pesticides; however, these correlations were typically weak, and contaminant concentrations were usually below sediment-toxicity thresholds. Concentrations of the pyrethroid bifenthrin exceeded an estimated threshold of 0.49 ng/g (at 1 % total organic carbon) in 14 % of the samples. Of the samples that exceeded this bifenthrin toxicity threshold, 79 % were toxic to amphipods compared with 25 % toxicity for the samples below this threshold. Application of mean probable effect concentration quotients (PECQs) based on measures of groups of contaminants (trace elements, total PAHs, total PCBs, OC pesticides, and pyrethroid pesticides [bifenthrin in particular]) improved the correct classification of samples as toxic or not toxic to amphipods compared with measures of individual groups of contaminants.

Electronic supplementary material The online version of this article (doi:10.1007/s00244-012-9815-y) contains supplementary material, which is available to authorized users.

N. E. Kemble (✉) · D. K. Hardesty · C. G. Ingersoll · J. L. Kunz
Columbia Environmental Research Center, United States Geological Survey, Columbia, MO, USA
e-mail: nkemble@usgs.gov

P. K. Sibley
University of Guelph, Guelph, ON, Canada

D. L. Calhoun
Georgia Water Science Center, United States Geological Survey, Atlanta, GA, USA

R. J. Gilliom · K. M. Kuivila · L. H. Nowell
California Water Science Center, United States Geological Survey, Sacramento, CA, USA

P. W. Moran
Washington Water Science Center, United States Geological Survey, Tacoma, WA, USA

Sediments are a repository for many contaminants released into surface waters. Because of this, organisms inhabiting sediments may be exposed to a wide range of contaminants (United States Environmental Protection Agency (USEPA) United States Environmental Protection Agency 2000; American Society for Testing and Materials [ASTM] American Society for Testing and Materials International 2012). Contaminants of potential concern in sediments typically include trace elements (metals), organochlorine (OC) pesticides, polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs; Ingersoll et al. 2001). In 2000, the USEPA began to restrict the use of organophosphate pesticides, such as diazinon and chlorpyrifos (Spurlock and Lee 2008). These restrictions have led to increased use of pyrethroid pesticides, which have widespread applications in both agricultural and urban environments (Kuivila et al. 2012).

Pyrethroids are hydrophobic compounds that have been observed to accumulate in sediments (Laskowski 2002). Toxicity of pyrethroids in field-collected sediment from

Table 1 Whole-sediment toxicity to the amphipod *H. azteca* (28 day exposures) or to the midge *C. dilutus* (10 day exposures) by different end points compared with reference sediments within and across the study areas

Study area	Nos. of samples			Amphipod (% of all samples)				Midge (% of all samples)				All tests and end points (% of all samples)
	Total	Amphipod reference	Midge reference	Survival	Weight	Total biomass	All end points	Survival	Weight	Total biomass	All end points	
Atlanta	13	6	6	38	0	0	38	0	0	8	8	38
Boston	14	7	7	14	14	14	21	0	29	36	36	43
Dallas-Fort Worth	13	3	3	69	31	62	69	0	8	0	8	69
Denver	13	7	7	23	8	8	23	0	0	0	0	23
Milwaukee-Green Bay	12	7	8	33	8	17	33	0	17	17	17	33
Salt Lake City	12	5	5	33	17	25	33	0	17	17	17	33
Seattle-Tacoma	21	15	14	19	0	0	19	0	0	5	5	24
Total	98	50	50	32	10	16	33	0	9	11	12	37

small urban streams (Weston et al. 2005; Holmes et al. 2008; Ding et al. 2010; Domagalski et al. 2010) or with pyrethroids spiked into sediment (Amweg et al. 2006; Hintzen et al. 2009) have been evaluated primarily in 10 day lethality tests conducted with the amphipod *Hyalella azteca*. However, the sublethal effects in long-term exposures to pyrethroids in sediment have not been evaluated, and the distribution of pyrethroids sediments has not typically been evaluated in wadeable streams (Gilliom et al. 2006).

This article is the second in a series that describe the results of a study of the distribution and toxicity of pyrethroids and other co-occurring trace elements and organic contaminants (PCBs, PAHs, OC pesticides) in stream sediments from 7 metropolitan areas across the United States (Moran et al. 2012). The study evaluated 98 sediment samples collected from streams ranging from undeveloped to highly urban and differs from previous studies by sampling larger wadeable streams and avoiding point sources (such as storm drains) and other inflows (Gilliom et al. 2006). Part 1 of the series characterizes sediment contaminants in relation to urbanization and other factors in the 7 metropolitan study areas (Nowell et al. 2012). Part 2 (this article) evaluates relationships between sediment chemistry and sediment toxicity in 28 day whole-sediment exposures conducted with the amphipod *H. azteca* and in 10 day whole-sediment exposure conducted with the midge *Chironomus dilutus* (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012). Toxicity end points evaluated in the amphipod and midge exposures included the effects of these field-collected sediments on survival, weight, or biomass of the test organisms.

Materials and Methods

Sediment Collection and Characterization

Surficial bed sediment samples were collected in 2007 from 98 sites in 7 metropolitan study areas: Atlanta (GA); Boston (MA, NH); Milwaukee-Green Bay (WI [Milwaukee]); Dallas-Fort Worth (TX [Dallas]); Denver (CO, WY); Salt Lake City (UT); and Seattle-Tacoma (WA [Seattle]; [see Table 1 and see Fig. 1 in Nowell et al. 2012]). Sites within each study area ranged in urban intensity from undeveloped (<5 % urban land) to highly urbanized (Nowell et al. 2012). Most sampling sites were in wadeable streams in small- to moderate-sized basins (see Figs. 2–8 in Moran et al. 2012). One sediment sample was collected from each of the 98 sites, which ranged from approximately 150–300 m stream length depending on stream width (length sampled was approximately 20 times the average wetted width). Each sample was a composite of multiple grab samples of recently deposited material collected by skimming approximately the top 2 cm of sediment with Teflon sheeting or a clean small scoop from multiple depositional zones within the stream reach during low-flow conditions.

Sediment samples were analyzed for trace elements, OC pesticides, PAHs, PCBs, pyrethroids, total organic carbon (TOC), and grain size (Moran et al. 2012). Sediments were sieved in the field to <2 mm for organic contaminant analyses and toxicity testing and to <63 μm for trace-element analyses. Porewater was isolated from the sediments at the start of the toxicity tests by centrifugation at 4 °C for 15 min at 5,200 revolutions/min; Kemble et al. 1994) and

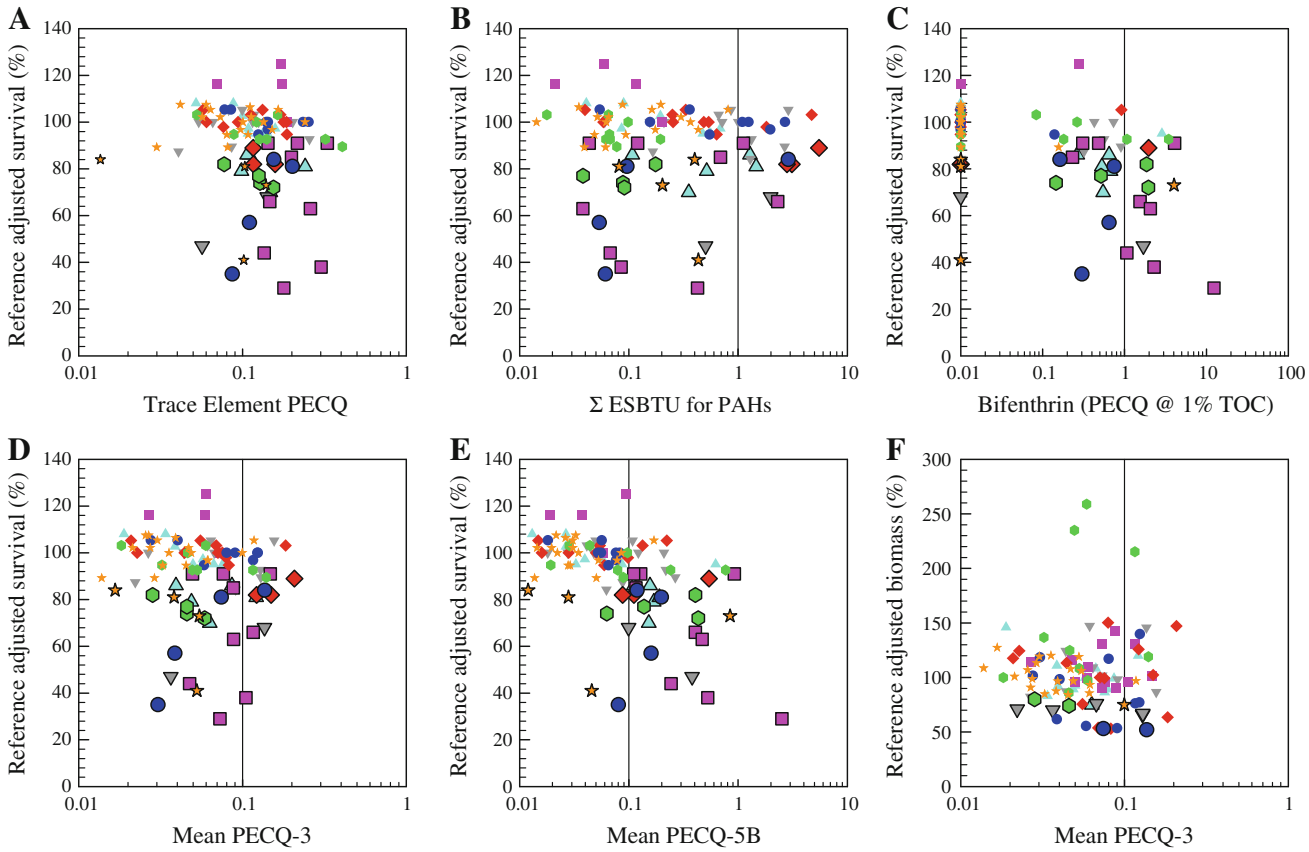

Fig. 1 Relationship between sediment chemistry and survival of *H. azteca* (HA) (Figs. 1a–e) or total biomass of *C. dilutus* (CD) (Fig. 1f). Study areas are represented by the following symbols: *pink square* = Dallas; *red diamond* = Denver; *green hexagon* = Salt Lake City; *blue circle* = Milwaukee; *grey triangle* = Boston; *turquoise triangle* = Atlanta; *orange star* = Seattle. Symbols outlined in *black* represent toxic samples with an end point response below the lower limit of a reference envelope value within a study area. The *vertical line* represents the appropriate sediment toxicity threshold (from Table 2 or 3) for each measure of sediment chemistry

was analyzed for ammonia, pH, hardness, alkalinity, and conductivity when sample volume was sufficient ($n = 32$ of 98 samples).

Sediment-Toxicity Tests

Sediment-toxicity tests were conducted with amphipods (28 day exposures) and midge (10 day exposures) according to methods outlined in USEPA (United States Environmental Protection Agency 2000) and ASTM (American Society for Testing and Materials International 2012) and listed in Table S1. Sediments were homogenized in a stainless steel bowl using a plastic spoon and added to exposure beakers 1 day before test organisms were added (day-1). Ten amphipods or midge were exposed to 100 ml sediment with 175 ml overlying water in 300 ml beakers with a total of four replicates per treatments per species with two volume additions/d of overlying water (Ingersoll et al. 2002). The source of overlying water was well water diluted with deionized water to a hardness of approximately 100 mg/L (as $CaCO_3$), alkalinity of approximately 95 mg/L (as $CaCO_3$), and pH of approximately 8.2. Amphipods in each beaker were fed 1.0 ml yeast–cerophyll–trout chow (1.7–1.9 g/L) in a water suspension daily. Midge in each beaker were fed 1.5 ml Tetrafin goldfish food (6.0 mg of dry solids [Tetra, Blacksburg, VA]) in a water suspension daily (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012). Toxicity end points for midge included 10 day survival, ash-free dry weight (AFDW), and total biomass (total biomass was calculated as the sum AFDW for all surviving organisms in each replicate chamber). Toxicity end points for amphipods included 28 day survival, length, weight, and total biomass. Surviving amphipods on a sampling date were preserved in 8 % sugar formalin for subsequent 28 day length measurement. The biomass of surviving amphipods from each replicate was estimated as the sum of individual amphipod weights calculated from the following empirical relationship: weight (mg) = $((0.177^{*}$ length (mm)) $- 0.0292)^3$ (Ingersoll et al. 2008; Moran et al. 2012).

Toxicity tests were conducted in four batches and were started within 1 month of sediment collection. Batch 1

testing started in May 2007 and included 21 samples from Seattle. Batch 2 and batch 3 testing started in June 2007 and included 13 samples from Atlanta and 13 samples from Dallas. Batch 4 testing started in October 2007 and included 51 samples from four study areas: Salt Lake City, Boston, Denver, and Milwaukee (Table 1). A control sediment collected from West Bearskin Lake, MN (approximately 2 %TOC; Ingersoll et al. 1998) was tested with each batch of sediments. Two changes were made to the USEPA (United States Environmental Protection Agency 2000) and ASTM (American Society for Testing and Materials International 2012) methods in exposures conducted with batch 3 and 4 sediments to improve control response of midge (Ingersoll et al. 2008) as follows: (1) exposures were started with approximately 7 day old midge larvae (rather than 10 day old larvae) to decrease the possibility of adult midge emerging by the end of a 10 day sediment exposure; and (2) exposures were started with midge larvae isolated from cultures still in their surrounding culture tubes rather than with larvae that have left (or have been removed from) their culture tubes. Larvae outside of their culture tubes may not be as healthy as larvae still inside their culture tubes (Dave Mount, USEPA, Duluth MN, personal communication, April 2007). Using these modifications to the midge exposure methods, our laboratory has seen improvement in sediment-toxicity tests conducted with midge during the past 5 years (e.g., control survival typically >90 % in 10 day exposures).

Data Analysis

Toxicity was established using a reference envelope approach by comparing responses of test organisms in test sediments to those of test organisms in reference sediments (Hunt et al. 2001; Ingersoll et al. 2009; Besser et al. 2009). The range of mean responses of test organisms in reference sediment samples with minimal levels of contamination was assumed to represent the normal range of responses of test organisms to uncontaminated sediments within a study area. The requirements for establishing a sample as a reference sediment for each toxicity test within a study area included the following: (1) mean PECQ for five classes of compounds (mean PECQ-5B) <0.1 (see later text for a description of the PECQ calculation), (2) a significant difference between sediments within a study area for a given toxicity end point, and (3) mean survival or weight of test organisms in a sample that met control test acceptability requirements (>80 % survival of amphipods, >70 % survival of midge, and >0.48 mg AFDW/individual midge; USEPA United States Environmental Protection Agency 2000, ASTM American Society for Testing and Materials International 2012; Tables S1 and S2). A minimum of three reference samples were needed to establish a reference envelope within a study area for that test organism (Table S3). A test sediment within a study area was classified as toxic to a test organism if the mean response of one or more toxicity end points (survival, weight, or total biomass) in that sample was less than the lowest mean for reference samples from the same study area. The mean responses of test organisms in each sediment sample were expressed as a percentage of the median responses of test organisms in reference sediments within a study area (Tables S2 and S3); this helps to account for the potential influence of physical characteristics of the sediment samples within a study area on the response of test organisms (e.g., TOC, grain size).

Statistical analyses were performed using SAS statistical software (SAS/STAT version 9.2; SAS Cary, NC). Differences in a toxicity end point among sites within a study area and within batch of sediments tested were determined by analysis of variance (ANOVA). Toxicity end point data were transformed before ANOVA to improve normality as indicated by Shapiro–Wilk test (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012). If transformations (arcsine square root for survival; square root or log for weight or total biomass) did not improve normality, data were rank-transformed before analysis (Conover and Iman 1981). Spearman rank correlation analysis was used to evaluate relationships between responses in the toxicity tests and the physical or chemical characteristics of sediments (Tables 2, 3). Statistical significance for the rank correlations was established at 0.05.

Probable effect concentrations (PECs) were used to evaluate relationships between sediment contamination and sediment toxicity (MacDonald et al. 2000). A PEC is a contaminant concentration above which adverse effects on sediment-dwelling organisms are expected to occur more often than not. The PEC quotient (PECQ) for a contaminant is the measured concentration divided by the PEC. The mean PECQ for a sample is the average of PECQ values for all contaminants in that sample. A higher likelihood of toxicity is predicted for samples with PECQ values >1 for individual contaminants or mean PECQ values of >0.1 to >0.2 for sample mixtures (Ingersoll et al. 2001, 2005, 2009).

The current study deviates from the originally published mean PECQ procedure as follows:

1. The PEC values from MacDonald et al. (2000) were assumed to apply to 1 %TOC in sediment (reflecting sediment levels typical of the data set used to derive PECs; Ingersoll et al. 2009). Hence, in the current study, contaminant concentrations were normalized by the measured concentration of TOC in the samples (express as µg/g [or µg/kg] dry weight at 1 %TOC before comparison with PECs).

Table 2 Spearman rank correlations among *H. azteca* 28 day toxicity end points and sediment characteristics and frequencies of observed toxicity for samples below and exceeding toxicity thresholds

Metric	Correlation coefficient			Sediment toxicity threshold	Samples above threshold (%)	Frequency of toxicity (% of samples in group)		Correct classification (%)[f]
	Survival	Weight	Total biomass			Samples below threshold	Samples exceeding threshold	
Toxicity end points								
Survival	**1.000**	0.077	**0.488**	NA	NA	NA	NA	NA
Weight	–	**1.000**	**0.810**	NA	NA	NA	NA	NA
Biomass	–	–	**1.000**	NA	NA	NA	NA	NA
Physical characteristics								
Total ammonia	−0.045	0.161	0.241	>32 mg N/L[a]	0	33	NA	67
Unionized ammonia	−0.182	−0.113	−0.058	>1.5 mg N/L[a]	0	33	NA	67
Sand	0.133	0.193	0.005	ND	NA	NA	NA	NA
Silt	−0.063	−0.053	−0.016	ND	NA	NA	NA	NA
Clay	−0.212	0.081	−0.008	ND	NA	NA	NA	NA
TOC (<2 mm)	−0.108	0.161	0.162	ND	NA	NA	NA	NA
TOC (<63 μm)	0.140	0.185	**0.293**	ND	NA	NA	NA	NA
Trace elements (@ 1 %TOC)								
As	−0.143	−0.150	−0.197	33.0 μg/g[b]	1	33	0	66
Cd	−0.188	−0.179	−0.059	4.98 μg/g[b]	0	33	NA	67
Cr	−0.143	−0.193	**−0.313**	111 μg/g[b]	0	33	NA	67
Cu	**−0.337**	−0.179	−0.178	149 μg/g[b]	0	33	NA	67
Pb	−0.191	−0.184	−0.076	128 μg/g[b]	0	33	NA	67
Ni	−0.131	−0.185	**−0.355**	48.6 μg/g[b]	0	33	NA	67
Zn	**−0.253**	−0.187	−0.136	459 μg/g[b]	0	33	NA	67
Mean trace element PEC-Q	**−0.246**	−0.137	−0.184	1.0	0	33	NA	67
Mean trace element PEC-Q (dw)	−0.048	−0.005	0.102	1.0	9	35	11	60
Organics (@ 1 %TOC)								
Fluorene	−0.120	**−0.248**	−0.182	0.536 μg/g[b]	0	33	NA	67
Anthracene	−0.146	−0.193	−0.137	0.845 μg/g[b]	0	33	NA	67
Benzo[a]anthracene	−0.133	−0.141	−0.072	1.05 μg/g[b]	0	33	NA	67
Benzo[a]pyrene	−0.152	−0.162	−0.105	1.45 μg/g[b]	0	33	NA	67
Chrysene	−0.152	−0.128	−0.065	1.29 μg/g[b]	1	32	100	68
Fluoranthene	−0.145	−0.148	−0.072	2.23 μg/g[b]	2	32	50	67
Naphthalene	−0.037	−0.125	−0.098	0.561 μg/g[b]	0	33	NA	67
Phenanthrene	−0.141	−0.141	−0.071	1.17 μg/g[b]	0	33	NA	67
Pyrene	−0.151	−0.158	−0.082	1.52 μg/g[b]	2	32	50	67
Total PAH	−0.140	−0.174	−0.098	22.8 μg/g[b]	0	33	NA	67
ESBTU	−0.138	−0.187	−0.122	1.0[c]	20	27	55	69
Mean OC PECQ	−0.080	**−0.238**	−0.186	1.0[d]	0	33	NA	67
Total PCB	−0.044	**−0.277**	**−0.206**	0.676 μg/g[b]	0	33	NA	67
Total PAH (dw)	**−0.211**	−0.035	0.025	22.8 μg/g[b]	6	33	33	65
Mean OC PECQ (dw)	**−0.218**	−0.042	0.027	1.0[d]	1	33	0	67
Total PCB (dw)	−0.174	−0.085	0.030	0.676 μg/g[b]	0	33	NA	67
Bifenthrin	**−0.583**	**−0.249**	**−0.427**	0.49 ng/g[e]	14	25	79	76
Permethrin	**−0.242**	−0.015	**−0.217**	19.6 ng/g[e]	1	32	100	68
ΣPyrethroid toxicity quotient	**−0.536**	**−0.241**	**−0.408**	1.0	15	24	80	77
Bifenthrin (dw)	**−0.612**	**−0.207**	**−0.411**	0.49 ng/g[e]	29	15	75	82

Table 2 continued

Metric	Correlation coefficient			Sediment toxicity threshold	Samples above threshold (%)	Frequency of toxicity (% of samples in group)		Correct classification (%)[f]
	Survival	Weight	Total biomass			Samples below threshold	Samples exceeding threshold	
PEC quotients (@ 1 %TOC)								
Mean PECQ-3	**−0.230**	−0.197	−0.183	0.1	19	28	53	68
Mean PECQ-4	**−0.199**	**−0.230**	**−0.207**	0.1	18	29	50	67
Mean PECQ-5B	**−0.570**	**−0.307**	**−0.457**	0.1	36	11	68	83
Mean PECQ-5M	**−0.473**	**−0.273**	**−0.388**	0.1	20	23	70	76
Mean PECQ-5P	**−0.541**	**−0.302**	**−0.447**	0.1	37	13	67	80
Mean PECQ-3 (dw)	−0.113	−0.017	0.107	0.1	84	50	29	33
Mean PECQ-4 (dw)	−0.130	−0.038	0.093	0.1	76	33	32	41

Bolded values indicate significant correlations at $p < 0.05$

ND not determined, *NA* not applicable, *dw* dry weight ($n = 98$ samples except for ammonia where $n = 32$)

[a] Nile Kemble (unpublished data) *H. azteca* 28 day water-only 20 % effect concentration (EC_{20}; unionized ammonia calculated for pH 8 at 23 °C)

[b] MacDonald et al. (2000) probable effect concentration

[c] USEPA (United States Environment Protection Agency 2003) and Dave Mount (USEPA, Duluth, MN, personal communication, August 2008)

[d] Current study

[e] Table 4

[f] Overall correct classification: percent not toxic below threshold + percent toxic above threshold

Table 3 Spearman rank correlations among *C. dilutus* 10 day toxicity end points and sediment characteristics as well as frequencies of observed toxicity for samples below and above toxicity thresholds

Metric	Correlation coefficient			Sediment toxicity threshold	Samples above threshold (%)	Frequency of toxicity (% of samples in group)		Overall correct classification (%)[f]
	Survival	Weight	Total biomass			Samples below threshold	Samples above threshold	
Toxicity end points								
Survival	**1.000**	−0.143	**0.247**	NA	NA	NA	NA	NA
Weight	–	**1.000**	**0.835**	NA	NA	NA	NA	NA
Biomass	–	–	**1.000**	NA	NA	NA	NA	NA
Physical characteristics								
Total ammonia	−0.127	−0.108	−0.090	93 mg N/L[a]	0	12	NA	88
Unionized ammonia	0.129	**−0.372**	−0.225	0.94 mg N/L[a]	0	12	NA	88
Sand	0.090	−0.017	−0.016	ND	NA	NA	NA	NA
Silt	−0.065	−0.083	−0.059	ND	NA	NA	NA	NA
Clay	−0.160	0.113	0.069	ND	NA	NA	NA	NA
TOC (<2 mm)	−0.040	0.051	0.053	ND	NA	NA	NA	NA
TOC (<63 μm)	0.142	0.075	0.145	ND	NA	NA	NA	NA
Trace elements (@ 1 %TOC)								
As	−0.023	−0.025	−0.110	33.0 μg/g[b]	1	12	0	87
Cd	−0.078	−0.101	−0.073	4.98 μg/g[b]	0	12	NA	88
Cr	**−0.232**	−0.079	**−0.216**	111 μg/g[b]	0	12	NA	88
Cu	−0.089	−0.132	−0.126	149 μg/g[b]	0	12	NA	88
Pb	0.072	−0.127	−0.028	128 μg/g[b]	0	12	NA	88

Table 3 continued

Metric	Correlation coefficient			Sediment toxicity threshold	Samples above threshold (%)	Frequency of toxicity (% of samples in group)		Overall correct classification (%)[f]
	Survival	Weight	Total biomass			Samples below threshold	Samples above threshold	
Ni	**−0.221**	−0.081	−0.193	48.6 μg/g[b]	0	12	NA	88
Zn	−0.010	−0.135	−0.077	459 μg/g[b]	0	12	NA	88
Mean trace element PEC-Q	−0.094	−0.095	−0.119	1.0	0	12	NA	88
Mean trace element PEC-Q (dw)	0.053	−0.008	0.069	1.0	9	11	22	83
Organics (@ 1 %TOC)								
Fluorene	0.050	**−0.208**	−0.099	0.536 μg/g[b]	0	12	NA	88
Anthracene	0.056	−0.187	−0.071	0.845 μg/g[b]	0	12	NA	88
Benzo[a]anthracene	0.052	−0.132	−0.020	1.05 μg/g[b]	0	12	NA	88
Benzo[a]pyrene	0.038	−0.143	−0.043	1.45 μg/g[b]	0	12	NA	88
Chrysene	0.041	−0.100	0.006	1.29 μg/g[b]	1	12	0	87
Fluoranthene	0.037	−0.119	−0.010	2.23 μg/g[b]	2	13	0	86
Naphthalene	−0.005	−0.137	−0.095	0.561 μg/g[b]	0	12	NA	88
Phenanthrene	0.058	−0.143	−0.029	1.17 μg/g[b]	0	12	NA	88
Pyrene	0.053	−0.118	−0.008	1.52 μg/g[b]	2	13	0	86
Total PAH	0.043	−0.136	−0.029	22.8 μg/g[b]	0	12	NA	88
ESBTU	−0.138	−0.187	−0.122	1.0[c]	20	9	25	78
Mean OC PECQ	−0.050	−0.085	−0.076	1.0[d]	0	12	NA	88
Total PCB	0.005	**−0.242**	**−0.221**	0.676 μg/g[b]	0	12	NA	88
Total PAH (dw)	0.031	−0.034	0.067	22.8 μg/g[b]	6	10	50	88
Mean OC PECQ (dw)	−0.011	0.042	0.102	1.0[d]	1	11	100	87
Total PCB (dw)	0.036	−0.031	0.024	0.676 μg/g[b]	0	12	NA	88
Bifenthrin	−0.005	−0.014	−0.025	0.49 ng/g[e]	14	11	21	80
Permethrin	−0.069	**0.225**	**0.223**	19.6 ng/g[e]	1	12	0	88
ΣPyrethroid toxicity quotient	0.012	−0.014	−0.016	1.0	15	11	20	79
Bifenthrin (dw)	−0.006	−0.057	−0.074	0.49 ng/g[e]	29	4	32	78
PEC quotients (@ 1 %TOC)								
Mean PECQ-3	0.012	−0.158	−0.099	0.1	19	11	16	75
Mean PECQ-4	0.007	−0.159	−0.105	0.1	18	10	22	78
Mean PECQ-5B	−0.032	−0.123	−0.116	0.1	36	3	29	72
Mean PECQ-5M	0.003	−0.125	−0.093	0.1	20	10	20	76
Mean PECQ-5P	−0.015	−0.117	−0.100	0.1	37	5	25	69
Mean PECQ-3 (dw)	0.095	0.002	0.094	0.1	84	13	12	25
Mean PECQ-4 (dw)	0.075	0.029	0.108	0.1	76	17	11	29

Bolded values indicate significant correlations at $p < 0.05$

ND not determined, *NA* not applicable, *dw* dry weight ($n = 98$ samples except for ammonia where $n = 32$)

[a] *C. dilutus* 10 day water-only median lethal effect concentration (LC_{50}; unionized ammonia calculated for pH 8 at 23 °C; Whiteman et al. 1996)

[b] MacDonald et al. (2000) probable effect concentration

[c] USEPA (United States Environment Protection Agency 2003) and Dave Mount (USEPA, Duluth, MN, personal communication, August 2008)

[d] Current study

[e] Table 4

[f] Overall correct classification: percent not toxic below threshold + percent toxic above threshold

2. The mean PECQ was expanded to include additional contaminants. Dieldrin and chlordane were added because their PECs come close to meeting published reliability criteria (MacDonald et al. 2000), and these compounds were frequently detected in the current study. This approach is consistent that of with other studies (Tao et al. 2010) in which chlordane and dieldrin were identified as contaminants of concern in field-collected sediments.

3. For pyrethroids, 28 day sediment toxicity thresholds for *H. azteca* were estimated based on the results of published *H. azteca* 10 day lethality tests conducted with spiked sediments (Table 4) by (a) dividing the 10 day lethal concentrations for pyrethroids by a factor of 2.5 to estimate sublethal effects (Amweg et al. 2005) and (b) further dividing by a factor of 2.0 to estimate 28 day effect thresholds from 10 day effect thresholds (Ingersoll et al. 2001). Although derived using the results of 10 day spiked-sediment toxicity tests conducted with *H. azteca*, these pyrethroid toxicity thresholds have a similar intent as PECs (i.e., as estimates of concentrations above which toxicity is expected to sediment-dwelling organisms), so a pyrethroid toxicity quotient is analogous to a PECQ value.

4. For all contaminants except pyrethroids, nondetections were assumed equal to half of the reporting level (detection limits ranged from 0.2 to 0.5 ng/g [dry weight; Table 4, Moran et al. 2012]), which is consistent with the original PECQ methodology (Ingersoll et al. 2001). For pyrethroids, nondetections were assumed equal to zero because pyrethroid reporting levels were relatively close to their toxicity thresholds (otherwise even samples with no detectable pyrethroids would have substantial pyrethroid toxicity quotient values; Moran et al. 2012).

5. The mean PECQ value equally weights three to five contaminant classes being studied (Ingersoll et al. 2001). For example, the PECQ-3 equally weights the contributions of: (a) trace elements (mean trace element PECQ of arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), nickel (Ni), and zinc (Zn), (b) PAHs (PECQ for total PAHs), and (c) PCBs (PECQ for total PCBs). The PECQ-4 equally weights the contributions of the following: (a) mean trace element PECQ, (b) PECQ for total PAHs, (c) PECQ for total PCBs, and (d) OC pesticides (mean PECQ for DDE, dieldrin, and total chlordane). The PECQ-5P equally weights the contributions of the: (a) mean trace-element PECQ, (b) PECQ for total PAHs, (c) PECQ for total PCBs, (d) mean PECQ for OC pesticides, and (e) pyrethroids (the sum of the toxicity quotients for bifenthrin, cypermethrin, cyhalothrin, and permethrin). The toxicity quotients for pyrethroids were summed because the pyrethroids share a common mode of action, so toxicity is expected to be additive (Trimble et al. 2009). However, results were similar when pyrethroids were represented by the mean toxicity quotient of these four pyrethroids (PECQ-5M) or by the toxicity quotient for bifenthrin alone (PECQ-5B).

Relationships between toxicity and concentrations of PAHs in sediment samples were also evaluated using mechanistically based chronic sum equilibrium-partitioning sediment benchmark toxic units (ΣESBTUs). The ΣESBTU approach integrates benchmarks for total PAHs (34 parent and alkylated PAHs) into a toxic unit model that accounts for the joint toxicity of various PAHs with the same mode of toxicity (i.e., nonpolar narcosis; USEPA United States Environment Protection Agency 2003). The ΣESBTU for the 34 PAHs was estimated from 18 parent

Table 4 Estimated pyrethroid whole-sediment toxicity thresholds for *H. azteca* in 28 day exposures

Compound	CAS no.	MDL (dw, ng/g)[a]	*H. azteca* 10 day LC_{50} (μg/g OC)	Source	*H. azteca* 10 day LC_{50} (dw ng/g at 1 %OC)	Estimated *H. azteca* 28 day threshold (dw ng/g at 1 %OC)[c]
Bifenthrin	82657–04–3	0.2	0.25 (9)[b]	Moran et al. (2012)	2.5	0.49
Cyfluthrin	68359–37–5	0.5	1.1 (2)[b]	Amweg et al. (2005)	11	2.2
Cyhalothrin	91465–08–6	0.2	0.44 (3)[b]	Moran et al. (2012)	4.4	0.87
Cypermethrin	52315–07–8	0.4	0.38 (4)[b]	Maund et al. (2002)	3.8	0.76
Deltamethrin	52918–63–4	0.2	0.79 (2)[b]	Amweg et al. (2005)	7.9	1.6
Esfenvalerate	66230–04–4	0.2	1.7 (4)[b]	Moran et al. (2012)	17	3.3
Permethrin	52645–53–1	0.2	9.8 (7)[b]	Moran et al. (2012)	9.8	19.6

Dw dry weight, *OC* organic carbon

[a] Minimum detection limits for USGS, Sacramento CA (reported in Moran et al. 2012)

[b] Estimated LC_{50} value based on lower 95 percentile of reported LC_{50}s (number of LC_{50}s in parentheses; Moran et al. 2012)

[c] The 28 day *H. azteca* sediment toxicity thresholds estimated from published *H. azteca* 10 day lethality tests conducted with spiked sediments by: (1) dividing the 10 day lethal concentrations for pyrethroids by a factor of 2.5 to estimate sublethal effects (Amweg et al. 2005); and (2) further dividing by a factor of 2.0 to estimate *H. azteca* 28 day effect thresholds from 10 day effect thresholds (Ingersoll et al. 2001). See Table 14 in Moran et al. (2012) for more detail

PAHs measured in the present study using methods described in USEPA (United States Environment Protection Agency 2003), including the application of a 50 % uncertainty factor (USEPA United States Environment Protection Agency 2003). The ΣESBTU approach was developed to account for the bioavailability of nonionic organic compounds in different sediments and incorporates select toxic effects concentrations in porewater (i.e., final chronic values [FCVs]). A ΣESBTU threshold of 1.0 was established based on an estimated critical body burden of approximately 2 μmol total PAHs/g lipid for 28 day weight of *H. azteca* in water-only exposures and an FCV of approximately 2.2 μmol total PAHs normalized to the fraction OC in sediment (Dave Mount, USEPA, Duluth, MN, personal communication). Using this approach, sediment samples with ΣESBTU values >1.0 were predicted to be toxic to *H. azteca* in 28 day sediment exposures to PAHs.

Results and Discussion

Sediment-Toxicity Tests

Mean survival and weight of amphipods or midge in the control sediment met test acceptability requirements (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012) for all of the batches of sediment samples (Table S1). Fifty of 98 samples across the study areas met the requirement of a reference sediment (low chemistry and low toxicity; Table 1 and Table S3).

A total of 37 % of the 98 sediment samples from the 7 study areas were identified as toxic to amphipods or midge (i.e., survival, weight, or total biomass were below the lower limit of a reference envelope value for the study area; Table 1 and Tables S2 and S3). More sediment samples were identified as toxic to amphipods (33 %) compared with midge (12 %). Of the 36 samples that were toxic to either amphipods or midge, 67 % were toxic to only amphipods, 11 % were toxic to only midge, and 22 % were toxic to amphipods and midge, suggesting that concentrations of contaminants in the sediments were more frequently toxic to amphipods compared with midge.

Survival was the most sensitive end point in the amphipod test (32 % toxic), whereas total biomass was the most sensitive end point in the midge test (11 % toxic). Samples from Dallas had the highest toxicity to amphipods (69 % toxic) and Boston samples the highest toxicity to midge (36 % toxic), whereas samples from Denver (23 % toxic) and Seattle (24 % toxic) had the lowest toxicity to amphipods or midge.

Comparisons of Sediment Characteristics and Sediment Toxicity

Significant rank correlations were infrequently observed between the water-quality characteristics measured in the overlying water or in the porewater relative to the survival, weight, or total biomass of amphipods or midge (data not shown). However, a significant negative correlation was observed between concentrations of unionized ammonia in porewater measured at the start of the exposures and midge weight (Table 3). None of the concentrations of unionized ammonia in porewater exceeded a 10 day median lethal effect concentration (LC_{50}) of 0.94 mg N/L established for *C. dilutus* in water-only exposures (Table 3). Hence, ammonia likely did not substantially contribute to the decreased weight of midge.

The grain size and TOC exhibited a broad range in sediment samples (e.g., sand-size particles ranged from 2 to 88 %, and TOC in the <2 mm size fraction ranged from 0.16 to 17 %; Moran et al. 2012). However, responses of amphipods or midge were not significantly correlated to grain size or to TOC measured in the <2 mm size fraction of sediment, and only a weak significant correlation ($r = 0.293$) was observed between amphipod biomass and TOC measured in the <63 μm fraction of sediment (Tables 2, 3). Therefore, the physical characteristics of the sediment samples likely did not substantially contribute to the responses of either test organism across the study areas. These results are consistent with reports by others that *H. azteca* and *C. dilutus* are relatively tolerant of wide ranges in these physical characteristics in sediment (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012).

Concentrations of metal, PAHs, OC pesticides, and PCBs in sediment infrequently exceeded PECs (Tables 2 and 3; Moran et al. 2012). Concentrations of pyrethroids were detected in 44 of 98 sediment samples. Pyrethroids were detected most often in the Dallas study area followed by the Salt Lake City and Atlanta study areas. The Seattle and Denver study areas had the fewest samples with pyrethroids detected. Of the 14 pyrethroids analyzed for, only 5 were detected; of these, bifenthrin was most commonly detected pyrethroid across all 7 of the study areas (at 40 of 98 sites) and within each of the 7 study areas (Moran et al. 2012). Bifenthrin was detected most often in samples from the Dallas and Salt Lake City study areas. Of the 14 sediment samples exceeding the calculated threshold for bifenthrin (Table 4), 6 were from the Dallas study area, 4 were from Salt Lake City study area, and 1 each was from the Atlanta, Denver, Boston and Seattle study areas.

Relationships to toxicity end points were initially evaluated relative to sediment-chemistry data expressed as (1)

dry weight concentrations, (2) TOC-normalized concentrations in the <2 mm size fraction of sediment, or (3) TOC-normalized concentrations in the <63 μm size fraction of sediment (Moran et al. 2012). Preliminary correlations between chemistry and toxicity end points were generally strongest when chemical concentrations were normalized to TOC measured in the appropriate sediment fraction (<2 mm fraction for organic contaminants and <63 μm fraction for trace elements) rather than on a dry-weight basis. Hence, these TOC-normalized values were the primary ones used in correlation analysis between sediment-chemistry and -toxicity end points (Tables 2 and 3), although the tables also include examples of correlations based on dry-weight concentrations.

Only a limited number of significant correlations were observed between toxicity end points for amphipods or midge and the concentrations of individual metals, individual or total PAHs, total PCBs, or mean PECQ for OC pesticides (normalized to 1 %OC or to dry weight), and these correlations were relatively weak. Moreover, concentrations of these contaminants infrequently exceeded individual PEC sediment-toxicity thresholds (Tables 2, 3). Weaker correlations were generally observed between sediment toxicity and sediment chemistry for midge compared with amphipods (Tables 2, 3).

Responses of amphipods or midge were not significantly correlated to concentrations of PAHs normalized to sediment OC using ΣESBTUs (Tables 2, 3, Fig. 1B). However, of the 20 samples that exceeded a ΣESBTU of 1.0, 50 % of them were toxic to amphipods, suggesting that PAH concentrations in some sediment samples may have contributed to the toxicity to amphipods based on exceedances of the ΣESBTU toxicity threshold of 1.0.

Responses of amphipods, but not midge, were weakly correlated to the mean PECQ-3 (calculated by averaging the quotients for mean trace elements, total PAHs, and total PCBs) and were weakly correlated to the mean PECQ-4 (which also included the mean PECQ for OC pesticides), and only 18–19 % of the samples exceeded a PECQ-3 or PECQ-4 toxicity threshold of 0.1 (Tables 2, 3). Of the samples that exceeded a PECQ-3 or PECQ-4 toxicity threshold of 0.1, a higher incidence of toxicity was observed to amphipods (50–53 % toxicity) compared with midge (16–22 % toxicity; Tables 2, 3; Figs. 1D, F). However, the frequency of toxicity below PECQ-3 or PECQ-4 thresholds (28–29 % for amphipods and 10–11 % for midge) was similar to the frequency of toxicity in samples above these thresholds. Hence, while PECQ-3 and PECQ-4 were correlated to toxicity, and component contaminants may have contributed to the toxicity, concentrations of these contaminants or groups of contaminants were only infrequently high enough to have solely caused the toxicity to amphipods or midge observed across the study areas.

Concentrations of bifenthrin, permethrin, and ΣPyrethroid toxic quotients were significantly negatively correlated to survival, weight, or total biomass of amphipods (Table 2, Fig. 1C) but not to midge (Table 3). A total of 14 % of the sediment samples exceeded the OC-normalized sediment toxicity threshold for bifenthrin; however, only one sediment sample exceeded the corresponding threshold for permethrin (Tables 2 through 4). Of the samples exceeding the OC-normalized bifenthrin toxicity threshold, 79 % were toxic to amphipods; however, only 25 % of the samples below this bifenthrin toxicity threshold were toxic to amphipods (Table 2; Fig. 1C). Similarly, of the samples exceeding the dry-weight bifenthrin toxicity threshold, 75 % were toxic to amphipods, whereas only 15 % of the samples below this bifenthrin toxicity threshold were toxic to amphipods (Table 2). Overall, correct classification of sediment samples as not toxic below, or as toxic above, the bifenthrin threshold was 76 % based on concentrations normalized to TOC or 82 % based on dry-weight concentrations (Table 2). Sediments from the Dallas study area exhibited the highest incidence of toxicity to amphipods (69 %; Table 1) and had some of the highest sediment concentrations of bifenthrin (Fig. 1C). Of the samples exceeding a ΣPyrethroid toxicity quotient threshold of 1.0, 80 % were toxic to amphipods compared with 24 % toxicity for samples below this toxicity threshold, and the overall correct classification of samples as not toxic to amphipods below, or as toxic above, the threshold was 77 % based on the ΣPyrethroid toxicity quotient (Table 2).

Including bifenthrin as a fifth contaminant class in the calculation of the mean PECQ-5B (or ΣPyrethroids as the fifth class in the mean PECQ-5P or mean pyrethroid toxicity quotients in the mean PECQ-5M) resulted in stronger negative correlations to responses of amphipods compared with the mean PECQ-3 or PECQ-4 (Table 2; Fig. 1E). However, correlations were not as strong for the mean PECQ-5M compared with the PECQ-5B or PECQ-5P. A total of 36–37 % of the samples exceeded a mean PECQ-5B or PECQ-5P toxicity threshold of 0.1. Above this threshold, 67–68 % of the samples were toxic to amphipods, whereas only 11–13 % of the samples below this threshold were toxic to amphipods (Table 2; Fig. 1C). In addition, more samples exceeded the mean PECQ-5B or mean PECQ-5P threshold of 0.1 (36–37 %) compared with the thresholds based solely on bifenthrin. Only 14 % of the samples exceeded the OC-normalized bifenthrin toxicity threshold, and only 29 % of the samples exceeded the dry-weight bifenthrin toxicity threshold (Table 3). Hence, including combined measures of trace elements, total PAHs, total PCBs, OC pesticides, and pyrethroid pesticides (bifenthrin in particular) in the calculation of the mean PECQs improved the correct classification of samples as toxic or not toxic to amphipods (76–83 %) compared with

the measures of individual classes of contaminants (66–69 %) or to mean PECQ-3 (68 %) or mean PECQ-4 (67 %; Table 3).

The toxicity of samples to midge was poorly predicted by the sediment-toxicity thresholds based on the trace metals and organic contaminants measured, including pyrethroids. The low incidence of toxicity to midge across the study areas (12 %, Table 1) may have resulted from other unmeasured contaminants in the sediment samples or from random chance. A minimum incidence of toxicity of approximately 20 % was observed in midge sediment-toxicity tests even at low sediment chemistry (Ingersoll et al. 2001). The finding that sediments were frequently more toxic to amphipods compared with midge is consistent with the pyrethroids being a primary cause of the toxicity. Spiked-sediment toxicity studies indicate that the 10 day weight of *H. azteca* was decreased at lower concentrations of bifenthrin (by a factor of 5–10) or permethrin (factor of 2) compared with concentrations that decreased the 10 day weight of *C. dilutus* (Amweg et al. 2005; Maul et al. 2008).

Weston et al. (2005), Amweg et al. (2006), and Hintzen et al. (2009) reported field-collected sediment samples to be lethal to *H. azteca* in 10 day laboratory exposures at sum pyrethroid toxic units (TUs) >1.0. The pyrethroid TUs in these previous studies were based on 10 day pyrethroid toxicity thresholds derived from spiked-sediment toxicity tests (Amweg et al. 2005). In contrast, lethality to *H. azteca* in 10 day exposures to field-collected sediments was observed at 10 day pyrethroid TUs greater than approximately 0.3 by Holmes et al. (2008), Ding et al. (2010), and Domagalski et al. (2010) In these previous studies, bifenthrin was a primary pyrethroid contributing to the 10 day *H. azteca* pyrethroid TUs in these field-collected sediment samples. Similarly, decreased populations of *H. azteca* in the field have been reported at 10 day pyrethroid TUs >0.6 (Weston et al. 2005).

In the present study, the estimated 28 day *H. azteca* ΣPyrethroid toxicity quotient threshold of 1.0 (Tables 2, 3) is numerically equivalent to a 10 day pyrethroid TU of 0.2 because the 28 day pyrethroid toxicity thresholds in the current study were derived by dividing 10 day *H. azteca* lethality data from spiked sediment-toxicity tests by a factor of 5 to account for longer-term exposure and sublethal effects (Table 4). Hence, the toxicity associated with pyrethroids to *H. azteca* in 28 day exposures based on survival or growth at 10 day pyrethroid TUs >0.2 (numerically equivalent to the 28 day pyrethroid TU of 1.0 in Table 2) were similar to the toxicity reported at 10 day pyrethroid TUs >0.3 for *H. azteca* in 10 day exposures based on survival (Holmes et al. 2008; Ding et al. 2010; Domagalski et al. 2010). However, these previous studies did not as extensively characterize concentrations of other contaminants of interest in the sediments (e.g., metals, PAHs, PCBs, other pesticides). Hence, other contaminants may have contributed to the toxicity observed in these previous 10 day lethality sediment-toxicity tests with *H. azteca*, which would decrease the apparent pyrethroid toxic threshold from exposure to pyrethroids alone.

Additional studies are needed to better establish chronic toxicity thresholds for pyrethroids in sediment based on longer-term exposures and including sublethal end points. The current study estimated 28 day pyrethroid toxicity thresholds for *H. azteca* on the basis of *H. azteca* 10 day lethality tests conducted with spiked sediments (Table 4). Given the uncertainties associated with these estimated pyrethroid toxicity thresholds, studies should be conducted using 28–42 day spiked sediment toxicity tests with *H. azteca* measuring effects on survival, weight, biomass, or reproduction to improve these estimates of chronic toxicity thresholds for pyrethroids in sediment (USEPA United States Environmental Protection Agency 2000; ASTM American Society for Testing and Materials International 2012). Moreover, there is some uncertainty from deriving a toxicity threshold using a combination of empirically based PECs (for contaminants other than pyrethroids) and pyrethroid toxicity thresholds estimated from mechanistically based spiked-sediment toxicity tests (10 day lethality tests with *H. azteca*; Table 4). Hence, it would be useful to develop empirically derived PECs for pyrethroids using matching chemistry and toxicity data for field-collected samples according to procedures outlined in MacDonald et al. (2000). Future studies should also include toxicity-identification evaluations and should further evaluate factors controlling the bioavailability of pyrethroids in sediment to better determine the contribution of pyrethroid to the overall toxicity observed in field-collected samples (e.g., Weston et al. 2008, You et al. 2008; Phillips et al. 2010).

Acknowledgments We thank Eric Brunson, Eugene Greer, Jamie Hughes, Chris Ivey, and Dave Whites for technical assistance in conducting the toxicity tests. Any use of trade, product, or firm names is for descriptive purposes only and does not imply endorsement by the United States Government. This study was a joint effort of the USGS National Water Quality Assessment Program, the USGS Toxic Substances Hydrology Program, and the USGS Environmental Health Program. This manuscript has been reviewed in accordance with USGS policy.

References

American Society for Testing and Materials International (2012) Standard test method for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (E1706-05 [2010]). Annual book of ASTM standards. Vol 11.06. ATSM, West Conshohocken, PA

Amweg EL, Weston DP, Ureda NM (2005) Use and toxicity of pyrethroid pesticides in the Central Valley, California, USA. Environ Toxicol Chem 24:966–972 (correction Environ Toxicol Chem 24:1300–1301)

Amweg EL, Weston DP, You J, Lydy MJ (2006) Pyrethroid insecticides and sediment toxicity in urban creeks from California and Tennessee. Environ Sci Technol 40:1700–1706

Besser JM, Brumbaugh WG, Hardesty DK, Hughes JP, Ingersoll CG (2009) Assessment of metal-contaminated sediments from the Southeast Missouri (SEMO) mining district using sediment toxicity tests with amphipods and freshwater mussels. Report submitted to Dave Mosby, United States Fish and Wildlife Service, Columbia

Conover WJ, Iman RL (1981) Rank transformations as a bridge between parametric and nonparametric statistics. Am Stat 35:124–129

Ding Y, Harwood AD, Foslund HM, Lydy MJ (2010) Distribution and toxicity of sediment-associated pesticides in urban and agricultural waterways from Illinois, USA. Environ Toxicol Chem 29:149–157

Domagalski JL, Weston DP, Zhang M, Hladik M (2010) Pyrethroid insecticide concentrations and toxicity in streambed sediments and loads in surface waters of the San Joaquin Valley California, USA. Environ Toxicol Chem 29:813–823

Gilliom RJ, Barbash JE, Crawford CG, Hamilton PA, Martin JD, Nakagaki N, et al. (2006) The quality of our nation's waters: pesticides in streams and ground water, 1992–2001. United States Geological Survey Circular 1291. USGS, Reston, VA

Hintzen EP, Lydy MJ, Belden JB (2009) Occurrence and potential toxicity of pyrethroids and insecticides in bed sediments of urban streams in central Texas. Environ Pollut 157:110–116

Holmes RW, Anderson BS, Phillips BM, Hunt JW, Crane DB, Mekebri A, Connor V (2008) Statewide investigation of the role of pyrethroid pesticides in sediment toxicity in California's urban waterways. Environ Sci Technol 42:7003–7009

Hunt JW, Anderson BS, Phillips BM, Newman J, Tjeerdema RS, Fairey R et al (2001) Evaluation and use of sediment toxicity reference sites for statistical comparisons in regional assessments. Environ Toxicol Chem 20:1266–1275

Ingersoll CG, Brunson EL, Dwyer FJ, Hardesty DK, Kemble NE (1998) Use of sublethal endpoints in sediment toxicity tests with the amphipod *Hyalella azteca*. Environ Toxicol Chem 17: 1508–1523

Ingersoll CG, MacDonald DD, Wang N, Crane JL, Field LJ, Haverland PS et al (2001) Predictions of sediment toxicity using consensus-based freshwater sediment quality guidelines. Arch Environ Contam Toxicol 41:8–21

Ingersoll CG, MacDonald DD, Brumbaugh WG, Johnson BT, Kemble NE, Kunz JL et al (2002) Toxicity assessment of sediments from the Grand Calumet River and Indiana Harbor Canal in northwestern Indiana. Arch Environ Contam Toxicol 43:153–167

Ingersoll CG, Bay SM, Crane JL, Field LJ, Gries TH, Hyland JL et al (2005) Ability of sediment quality guidelines to estimate effects of sediment-associated contaminants in laboratory toxicity tests or in benthic community assessments. In: Wenning RJ, Batley G, Ingersoll CG, Moore DW (eds) Use of sediment quality guidelines and related tools for the assessment of contaminated sediments. SETAC Press, Pensacola, pp 497–556

Ingersoll CG, Besser JM, Brumbaugh WG, Ivey CD, Kemble NE, Kunz JL, et al. (2008) Sediment chemistry, toxicity, and bioaccumulation data report for the United States Environmental Protection Agency–Department of the Interior sampling of metal-contaminated sediment in the Tri-state Mining District in Missouri, Oklahoma, and Kansas. Prepared by USGS, Columbia MO, and MacDonald Environmental Sciences Ltd., Nanaimo, BC, for the USEPA, Kansas City, MO; USEPA, Dallas, TX; and USFWS, Columbia, MO

Ingersoll CG, Kemble NE, Kunz JL, Brumbaugh WG, MacDonald DD, Smorong D (2009) Toxicity of sediment cores collected from the Ashtabula River in northeastern Ohio USA to the amphipod *Hyalella azteca*. Arch Environ Contam Toxicol 57:315–329; 826–827 [erratum]

Kemble NE, Brumbaugh WG, Brunson EL, Dwyer FJ, Ingersoll CG, Monda DP et al (1994) Toxicity of metal-contaminated sediments from the upper Clark Fork River, MT to aquatic invertebrates in laboratory exposures. Environ Toxicol Chem 13:1985–1997

Kuivila KM, Hladik ML, Ingersoll CG, Kemble NE, Moran PW, Calhoun DL et al (2012) Occurrence and potential sources of pyrethroid insecticides in stream sediments from seven U.S. metropolitan areas. Environ Sci Technol 46:4297–4303

Laskowski DA (2002) Physical and chemical properties of pyrethroids. Rev Environ Contam Toxicol 174:49–170

MacDonald DD, Ingersoll CG, Berger T (2000) Development and evaluation of consensus-based sediment quality guidelines for freshwater ecosystems. Arch Environ Contam Toxicol 39:20–31

Maul JD, Brennan AA, Harwood AD, Lydy ML (2008) Effect of sediment associated pyrethroids, fipronil, and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization, and survival. Environ Toxicol Chem 27:2582–2590

Maund SJ, Hamer MJ, Lane MCG, Farrelly E, Rapley JH, Goggin UM, Gentle WE (2002) Partitioning, bioavailability, and toxicity of the pyrethroid insecticide cypermethrin in sediments. Environ Toxicol Chem 21(1):9–15

Moran PW, Calhoun DL, Nowell LH, Kemble NE, Ingersoll CG, Hladik M, et al (2012) Contaminants in stream sediments from seven U.S. metropolitan areas—Data summary of a national pilot study. United States Geological Survey Scientific Investigations Report 2011-5092. USGS, Reston, VA. http://pubs.usgs.gov/sir/2011/5092/

Nowell LH, Moran PW, Gilliom RJ, Calhoun DL, Ingersoll CG, Kemble NE, et al (2012) Contaminants in stream sediments from seven United States metropolitan areas: Part I—Distribution in relation to urbanization. Arch Environ Contam Toxicol. doi: 10.1007/s00244-012-9813-0

Phillips BM, Anderson BS, Voorhees JP, Hunt JW, Holms RW, Mekebri A et al (2010) The contribution of pyrethroid pesticides to sediment toxicity in four urban creeks in California, USA. J Pestic Sci 35:302–309

Spurlock F, Lee M (2008) Synthetic pyrethroid use patterns, properties, and environmental effects. In: Gan J, Spurlock F, Hendley P, Weston D (eds) Synthetic pyrethroids: occurrence and behavior in aquatic environments. American Chemical Society Symposium Series 991. ACS, Washington, DC, pp 3–25

Tao J, Ingersoll CG, Kemble NE, Dias RJ, Morowchick JB, Welker G et al (2010) Sediment contamination of residential streams in the metropolitan Kansas City area, USA: Part II. Whole-sediment toxicity to the amphipod *Hyalella azteca*. Arch Environ Contam Toxicol 59:370–381

Trimble AJ, Weston DP, Belden JB, Lydy MJ (2009) Identification and evaluation of pyrethroid insecticide mixtures in urban sediments. Environ Toxicol Chem 28:1687–1695

United States Environment Protection Agency (2003) Procedures for the derivation of equilibrium partitioning sediment benchmarks (ESBs) for the protection of benthic organisms: PAH Mixtures. EPA-600-R-02-13. USEPA, Washington, DC

United States Environmental Protection Agency (2000) Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, 2nd ed. EPA/600/R-99/064, USEPA, Washington, DC

Weston DP, Holmes RW, You J, Lydy MJ (2005) Aquatic toxicity due to residential use of pyrethroid insecticides. Environ Sci Technol 39:9778–9784

Weston DP, Zhang M, Lydy MJ (2008) Identifying the cause and source of sediment toxicity in an agriculture-influenced creek. Environ Toxicol Chem 27:953–962

Whiteman FW, Ankley GT, Kahl MD, Rau DM, Balcher MD (1996) Evaluation of interstitial water as a route of exposure for ammonia in sediment tests with benthic macroinvertebrates. Environ Toxicol Chem 5:794–801

You J, Pehkonen S, Weston DP, Lydy MJ (2008) Chemical availability and sediment toxicity of pyrethroid insecticides to *Hyalella azteca*: application to field sediment with unexpectedly low toxicity. Environ Toxicol Chem 27:2124–2130