

**Survey of Micropollutants in Fluvial-Estuarine Sediments and Water from the Hunting
Creek Watershed and the Tidal Freshwater Potomac River**

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Introduction

A wide variety of micropollutants (MPs) exist that impact water quality in the Chesapeake Bay watershed. Some micropollutants are regulated through the Clean Water Act (CWA), which includes a list of 65 chemical constituents considered Toxic Pollutants (CWA section 307(a)(1), 40 CFR 401.15). Some of these Toxic Pollutants (e.g., PCBs) have Total Maximum Daily Loads established in the Potomac River watershed (Haywood and Buchanan 2007) because of extensive historical pollution problems. Other micropollutants such as pharmaceutical chemicals, personal care products and xenoestrogens are unregulated and widely used in commerce, but may pose a threat to ecological and environmental health (Luo et al. 2014). These chemicals of emerging concern have substantial emissions into the aquatic environment, but their sources, fate and risks are not well characterized. As such, a need exists to determine the sources, presence and ecological risks of these micropollutants in surface waters to assess potential impacts to human and ecological health.

Sediment and water samples collected from Hunting Creek (Alexandria, VA, USA) in 2018 (May through October) were analyzed for selected emerging micropollutants to characterize their presence, geospatial variability and distribution between water and river bed sediments in a Potomac River tributary receiving wastewater discharge. The present study is a continuation of the ongoing collaboration between the Potomac Environmental Research and Education Center (PEREC) at George Mason University and the Alexandria Renew Enterprises. Hunting Creek is a tidal embayment formed where Cameron Run meets the tidal Potomac River in northern Virginia.

Emerging micropollutants find their way into the aquatic environment primarily through stormwater runoff, agricultural practices and wastewater treatment plant (WTP) discharge or releases. When released into natural waters these chemicals accumulate in organic matter and fine-grained sediments or suspended sediment particles. Because storm runoff and wastewater discharge represent a sizable fraction of the annual surface water flow in urban regions, these sources are often sufficient to promote in-stream accumulation of micropollutants. Thus, the entire aquatic community may be exposed throughout entire life cycles and across generations to mixtures of biologically-active chemicals in urban areas. To better understand the implications of micropollutants in the Potomac River ecosystem, further ecological baseline investigations are needed because little is known regarding the fate, effects and distribution of emerging micropollutants in the aquatic environment. The 2018 Hunting Creek project was patterned on the long-running Gunston Cove Study, which PEREC has been conducting in partnership with the County of Fairfax Department of Public Works and Environmental Services since 1984.

Study Objectives

Water and fluvial-estuarine sediments collected in the vicinity of the Hunting Creek embayment of the Potomac River were analyzed for micropollutants normally associated with urban sources. The objectives of the present investigation were to

- characterize the identity, occurrence and concentrations of MPs in water and sediments (the Hunting Creek tributary and embayment provide an ideal geographic location to

study the sourcing and environmental behavior of MPs arising from reclaimed water);

- establish a baseline of the MPs in water and sediments that may be of future importance in public and ecological health and regulation; and
- identify spatial and temporal trends (i.e., status and trends) in the concentrations of MPs in water and sediments in Hunting Creek.

Materials and Methods

Study area

Hunting Creek is a tributary embayment of the Potomac River lying 8 km downstream of Washington, DC, immediately south of the city of Alexandria, VA. Hunting Creek exists at the stream confluence of Cameron Run and Hooffs Run prior to discharge into the mainstem Potomac River. The Cameron Run watershed, the largest of the two sub-sheds, is predominantly urban (95% residential). Jones Point (Alexandria, VA) forms the northern boundary and Dyke Marsh the southern boundary of Hunting Creek (FCPS 2007). Alexandria Renew Enterprises WTP is located north of the shoreline of upper Hunting Creek and discharges an average of 150,000 m³ of wastewater daily. The Hunting Creek sampling grid was divided into the Cameron Run (CR01) upstream zone (above the head of tide), upper Hunting Creek (UHC01) WTP discharge zone, lower Hunting Creek (AR03, AR03) and downstream zone and the mainstem Potomac River (AR04 and AR10) reference zones. A total of six sampling stations have been established (Fig. 1) for the micropollutant survey.

Water sampling

The Hunting Creek watershed and the Potomac River mainstem sampling locations are provided in Table 1 along with the sampling dates in 2018 (Table 2). The Potomac River/Lower Hunting Creek sites were accessed as part of the ecological survey sampling schedule by boat, and the Cameron Run and Upper Hunting Creek sites were accessed by wading from shore. At each site 20 L of water was collected from the river by pumping water using a submersible pump (Fultz Pumps Inc., Lewistown, PA) into stainless-steel Cornelius (soda) kegs (Midwest Home Brewing Supplies, Minneapolis, MN). Water samples were collected 0.3 m below the surface and shallow water (<2 m) and vertically-integrated in deeper water (>2m) by continuously moving the submersible pump head vertically in the water column during sampling. The kegs were sealed with an O-ring-lined lid, stowed and transported to the lab where they were



Figure 1. Sampling locations in the Hunting Creek watershed and Potomac River (shown by pins). WTP is the AlexRenew wastewater treatment plant.

briefly stored at 4 °C prior micropollutant analysis. River water was also collected in 1-L polypropylene bottles for determination of total suspended matter (TSM) concentrations. For the third sampling trip, the location of the Upper Hunting Creek station was moved from the south side of Upper Hunting Creek under the Route 1 superstructure to Hooffs Run near the Alex Renew outfall discharge (as noted in Table 1).

Table 1. Collection sites, labeling codes and coordinates for 2018 survey (water and sediment).

Site	Labeling Code	Sampling Coordinates (DD units)
Cameron Run	CR01	38.79861, -77.073197
Upper Hunting Creek	UHC01	38.792003, -77.056247
	UHC01 (relocated)	38.796071, -77.060465
Lower Hunting Creek	AR02	38.788849, -77.050063
	AR03	38.78022, -77.04811
Potomac River mainstem	AR04	38.78063, -77.03640
	AR10	38.79698, -77.03923

Sediment sampling

Riverbed sediments were obtained using a Petite Ponar grab sampler (Wildco, Saginaw, MI) tethered by rope. Once obtained from the riverbed in the Ponar, the sediments were lifted onboard and expelled as undisturbed as possible into a stainless-steel tray, where ~10 g of the top 2-4 cm (surficial layer) was placed directly into a cleaned, glass jar, and stowed on ice for transport to the laboratory. Riverbed sediments were obtained by boat according to the same locations established for water sampling (Table1) at the Lower Hunting Creek and the Potomac River sites. The sediment collection jars were sealed using a Teflon-lined lid and stored on ice for transportation to the laboratory, whereupon the samples were stored at -20°C until sample processing. Riverbed sediments were collected in triplicate using three separate grabs. Bottom sediment was not collected at stations CR01 and UHC01 because the river bottom was rocky and did not provide suitable fine-grained bottom sediment for analysis. At these latter locations, particles isolated from filtered water served as the source of the geosolids. A summary of the samples collected for water and sediment PPCP analysis in the 2018 survey is shown in Table 2.

Table 2. Water and sediment sampling summary for 2018. Water and sediment samples were analyzed in triplicate (N = 3).

Site	Sampling Dates (Replicate samples)
CR01 ^a	15 May (3), 16 July (3), 18 September (3)
UHC01 ^a	15 May (3), 16 July (3), 18 September (3) ^b
AR02	2 May (3), 12 July (3), 13 September (3)
AR03	2 May (3), 12 July (3), 13 September (3)
AR04	2 May (3), 12 July (3), 13 September (3)
AR10	2 May (3), 12 July (3), 13 September (3)

^aWater collection only, substrate at these upstream locations was rocky bottom; ^brelocated site.

Water filtration

Upon return to the laboratory, the 20-L river water samples were pressure filtered the day of collection through 150 mm diameter Whatman GF/F 0.7 μm (nominal pore diameter) glass fiber filters to separate particles from water (Fig. 2). Whatman GF/D 2.7 μm pre-filters were used to prevent clogging of the 0.7 μm filter. The filters were held in place using a 142 mm filter-holder stand (EMD Millipore, Billerica, MA). Ultra-high purity gaseous nitrogen was fitted to the inlet ball-lock valve of the keg. The keg's outlet valve was connected to the inlet port of the filter holder. The outlet of the filter holder was connected with tubing that was used to aliquot samples. Nitrogen pressure was held at 40 psi and gas flow was carefully controlled by a needle valve forcing the water through the filter set which collected suspended particles. The filtrate was aliquoted into 1 L amber glass bottles. Nine bottles of filtered water were collected from each keg and stored at 4 °C. The filter set was sealed in an aluminum pouch prior to extraction and micropollutant analysis and stored at -20 °C. The exact volume in each bottle and remaining keg water were measured for total sample volume. The 1 L water samples were vacuum filtered through pre-weighed 47 mm diameter GF/D and GF/F filter sets, air dried and weighed again for determining particle mass per liter (TSM).

Water extraction

The micropollutants are extracted from filtered water to which surrogate standards are added by using two solid phase extraction (SPE) techniques (Fig. 2). Oasis SPE cartridges are used at the Potomac Science Center (PSC). Oasis HLB cartridges are conditioned with 6 mL MTBE, equilibrated with 6 mL MeOH and 6 mL of ultra-high purity water (UHPW; Millipore, Milli-Q). The samples are loaded onto the cartridges using a Supelco vacuum manifold (Sigma-Aldrich, St. Louis, MO) and large volume sample tubing at a rate of 1-2 drops per second. Following extraction, the cartridges are washed with 95:5 UHPW:MeOH and eluted with 6 mL 10:90 MeOH-MTBE into 40 mL amber VOA bottles. The SPE extracts are concentrated by evaporation using a Turbo Vap evaporator (Zymark Corp., Hopkinton, MA) to 1 mL and quantitatively transferred to a 12 x 32 mm deactivated amber glass autosampler vial with PTFE lined septa for instrumental analysis. The combination SPE C18-ion exchange method is used for extracting micropollutants from water to maximum extraction efficiencies. Oasis MAX and MCX cartridges are conditioned with 6 mL of MeOH and equilibrated with 6 mL of UHP water. The cartridges are stacked with MAX on top, and 1 L samples with appropriate surrogate standards are added. Following extraction, the cartridges are separated and washed with 2 mL of 95:5 UHP-W:MeOH. The MAX and MCX cartridges are eluted separately with 6 mL of 69:29:2 MeOH:EtOAc:HCOOH and 6 mL

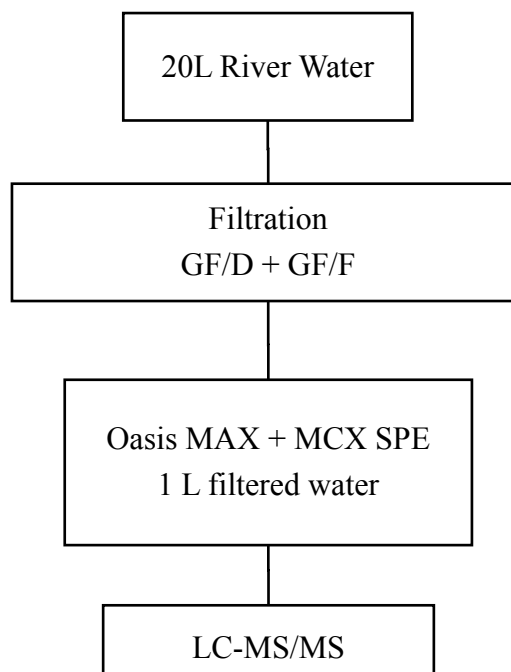


Figure 2. Water analysis method flow chart.

of 67.5:27.5:5 MeOH:EtOAc:NH₃, respectively. The extracts are combined and concentrated as per above.

Sediment extraction

Micropollutants were extracted from sediment using QuEChERS (**Quick-Easy-Cheap-Effective-Rugged-Safe**) protocol (Agilent Technologies, Santa Clara, CA) following previously reported procedures (Kachhawaha et al. 2017). QuEChERS protocol is essentially liquid-solid extraction followed by liquid-liquid extraction followed by sample extract cleanup (Fig. 3). The thawed wet sediment was pre-sieved (0.5 mm) to remove pebbles and other large particles. Sieved sediment was subsequently centrifuged for 10 minutes at 1500 rpm and the water was decanted to remove bulk water prior to extraction. An aliquot was reserved for determining moisture separately. About 2.0 g of dewatered sediment was placed in 50-mL polypropylene centrifuge tubes along with 10 mL of Optima grade acetonitrile. Internal standards and surrogate standard mixtures were added at this point to preserve the analyte-to-internal standard ratio prior to analyzing sample aliquots that avoid accidentally obtaining aqueous portions of the bottom phase. The tubes were vortexed for 1 min. Then 10 mL of ultra-high purity water was added to the slurry followed by vortexing again for 1 min. Packets containing 6 g of anhydrous magnesium sulfate and 1.5 g sodium acetate were added to affect a phase separation between water and acetonitrile and to partition the micropollutants into the organic phase. The mixture was again vortexed for 1 min. The tubes were subsequently centrifuged for 5 min at 4000 rpm, and the tubes were placed in a -20 °C freezer for 2 hours to aid in the separation of the organic and aqueous phases. An 8-9 mL aliquot was decanted into a 15-mL polypropylene sample clean-up tube containing 1.2 g MgSO₄ and 0.4 g of primary-secondary amine (PSA) to remove interfering matrix components. The tubes were vortexed and centrifuged for 10 min at 3000 rpm. Aliquots of supernatant were decanted into clean 40-mL amber glass vials. The volume in the vials was reduced to 1 mL in a TurboVap evaporator (using a vortex of N₂ gas) and transferred to a syringeless filter vial for LC-MS/MS analysis.

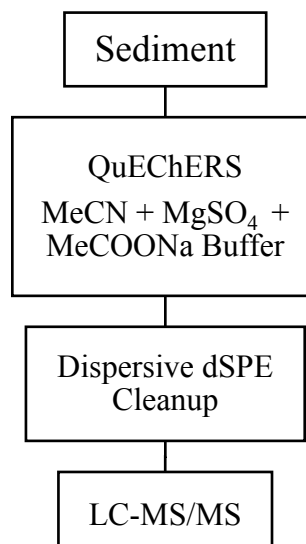


Figure 3. Sediment analysis method flow chart.

Filter extraction

Glass-fiber filters holding the filtered suspended matter were cut into strips with solvent-rinsed stainless-steel surgical scissors. They were placed into clean 250 mL Erlenmeyer flasks along with any remaining material on the foil pouches. Surrogate and internal standards were added along with 100 mL of 60:40 acetone:EtOAc. The flasks were sonicated in a water bath for 30 min and decanted into a 250 mL conical TurboVap tube. The extraction procedure was repeated. The combined solvent was evaporated in a TurboVap concentrator to 5 mL. The extracts were processed by the QuEChERS according to the above protocol.

Total suspended matter analysis

Water samples collected in the 1-L polypropylene bottles are warmed to room temperature and shaken rigorously to resuspend settled particles. The water is filtered through a stacked arrangement of a pre-weighed 47 mm glass fiber filters, a Whatman GF/D (pre-filter) overlaying a GF/F, under an applied vacuum of ~20 torr using a Millipore filtration apparatus. The volume of the filtered water is measured by graduated cylinder and the filters are dried for 24 hrs at 65 °C prior to gravimetric analysis. Total suspended matter (TSM) concentration (mg/L) is determined as mg of filtered particles ÷ sample volume (L).

Sediment moisture

Sediment moisture was determined by placing 1 g of wet sediment into a tared crucible and measuring mass. The crucible was placed in an oven for 24 hr at 65 °C and again gravimetrically evaluated. Moisture content (%M) was evaluated by the loss of mass loss upon heating: % M = [mass of water loss (g) ÷ mass of wet sediment (g)] x 100. %M was used to correct wet weight to dry weight (dw) for the sediment samples in the expression of MP sediment concentrations.

Sediment grain size

The thawed sediment samples were initially passed through a 500-micron stainless-steel sieve to remove the largest particles (e.g., large organics, shells, etc.) and then run through a centrifuge to remove excess water, giving the sediment the consistency of wet paste. Each sample was separated into triplicates and stored in three different plastic containers. One cubic centimeter (1 cm³) of sediment was collected from each container, placed in a test tube, and soaked for at least 30 minutes in 10 ml of class 1 deionized water with a 5% Calgon solution (Hexametaphosphate) to avoid flocculation during analysis. Each test tube was sonicated for 60 seconds to completely disaggregate the sediment and then the suspended sediment was poured into the aqueous liquid module of the Beckman-Coulter laser diffraction (LS 13320) particle size analyzer (PSA) for analysis. Once in the aqueous liquid module, the PSA conducted six separate runs of each sediment sample. The sediment sample was sonicated for an additional 90 seconds before each of the six runs (see Blott and Pye [2006] and Rodriguez and Uriarte [2009] for additional details). Overall, six data files were generated for every sediment subsample, and one additional data file, which was the average of the six runs.

Data generated by the Beckman-Coulter LS13320 were run through the Microsoft© Excel-based computer program GRADISTAT (Blott and Pye 2001) to calculate a complete suite of grain-size statistics (Fig. 4). The geometric Folk and Ward Method was used to calculate mean grain size and percent sand, silt, and clay, as well as to generate sand-silt-clay ternary diagrams (see Blott and Pye 2001).

Micropollutants monitored

The Status and Trends list of analytes (Appendix A) were analyzed via LC-MS/MS in all Hunting Creek Survey water and sediment samples. The list includes 91 pharmaceutical and personal care product target chemicals (hereafter referred to as PPCPs). The PPCPs were selected based on use statistics (i.e., most commonly prescribed drugs and over the counter medications) and those previously considered a high risk in the aquatic environment based on a review of published literature (Kaplan 2013). There exist over 5,000 PPCPs registered in commerce in the USA so the list represents a fraction of all known PPCP chemicals.

SAMPLE STATISTICS

SAMPLE IDENTITY: 2018-0803-T03-PS-SED-EM-201-SS2-R ANALYST & DATE: GLB, 07/31/18

SAMPLE TYPE: Bimodal, Very Poorly Sorted TEXTURAL GROUP: Sandy Mud
SEDIMENT NAME: Very Fine Sandy Medium Silt

	μm	ϕ	GRAIN SIZE DISTRIBUTION					
MODE 1:	13.63	6.199	SAND:	12.5%	GRAVEL:	0.0%	COARSE SAND:	0.0%
MODE 2:	0.432	11.16	SILT:	74.8%	SAND:	12.5%	MEDIUM SAND:	0.0%
MODE 3:			CLAY:	12.7%	MUD:	87.5%	FINE SAND:	3.4%
D ₁₀ :	1.350	3.785					V FINE SAND:	9.1%
MEDIAN or D ₅₀ :	12.92	6.274			V COARSE GRAVEL:	0.0%	V COARSE SILT:	14.4%
D ₉₀ :	72.53	9.533			COARSE GRAVEL:	0.0%	COARSE SILT:	17.8%
(D ₉₀ / D ₁₀):	53.73	2.518			MEDIUM GRAVEL:	0.0%	MEDIUM SILT:	19.0%
(D ₉₀ - D ₁₀):	71.18	5.748			FINE GRAVEL:	0.0%	FINE SILT:	14.9%
(D ₇₅ / D ₂₅):	7.152	1.582			V FINE GRAVEL:	0.0%	V FINE SILT:	8.7%
(D ₇₅ - D ₂₅):	29.24	2.838			V COARSE SAND:	0.0%	CLAY:	12.7%
			METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description		
	μm	μm	ϕ	μm	ϕ			
MEAN (\bar{x}):	26.95	11.06	6.439	12.19	6.359	Medium Silt		
SORTING (σ):	35.22	4.783	2.258	4.814	2.267	Very Poorly Sorted		
SKEWNESS (γ):	2.227	-0.690	0.690	-0.143	0.143	Fine Skewed		
KURTOSIS (α):	8.232	3.339	3.339	1.140	1.140	Leptokurtic		

Figure 4. Example of grain size statistics generated in GRADISTAT (Blott and Pye, 2001).

LC-MS/MS analysis

Quantitative analysis of the PPCPs in water, sediment and particle samples was performed using a Shimadzu 8050 LC-MS/MS (Shimadzu, Columbia, MD) with combined electrospray (positive and negative modes) and APCI ionization (DUIS) in the presence of a Corona needle. Chromatographic separations are performed using a 50 mm x 2.1 mm (id), 1.8 μm (particle dia.) Raptor C18 reversed-phase UHPLC column (Restek Corp., Bellefonte, PA) in conjunction with a binary mobile phase consisting of Milli-Q water (solvent A), and acetonitrile (solvent B) both containing 0.1%

formic acid mobile phase modifier,. The UHPLC gradient elution program is provided below for a total run time of 15 min. UHPLC retention times for the PPCPs are shown in Appendix A.

Total flow rate = 0.40 mL/min

30% B at 0 min

30% to 95% B 0-6 min

95% B 6-12 min

95% to 30% B 12-13 min

30% B 13-15 min

The MS/MS was operated in the multiple reaction monitoring (MRM) mode with quantifier and qualifier ion transitions and instrumental operating parameters determined experimentally (Appendix B). Quantitation was performed using isotopically (^2H or ^{13}C) labelled internal standards added prior to the extraction step (Appendix A). Ten-point calibration curves based on the primary product MRM ion for each micropollutant and standard are used to determine sample concentrations. Two qualifier product ions were used to confirm the chemical identity of the PPCP along with chromatographic retention times. Data analysis and quantitation was performed using LabSolutions software (ver. 5.91, Shimadzu, Columbia, MD). The LC-MS/MS methods developed at PSC closely parallel methods reported for the analysis of multiple classes of MPs in WTP discharge water and surface waters (Dasenaki and Thomaidis 2015; Matongo et al. 2015; Zhang and Fent 2018).

Quality assurance

The quality assurance (QA) protocol includes laboratory blanks, method detection limit evaluations, matrix spike recoveries, and surrogate recoveries. Surrogate spike recoveries are

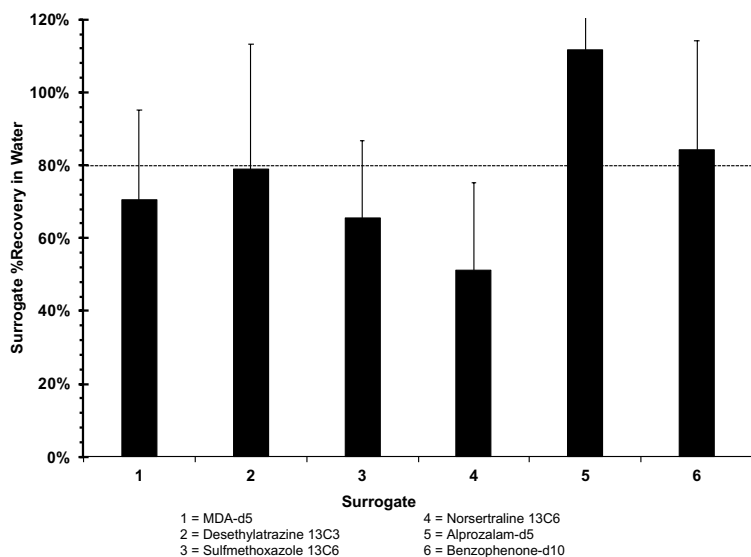


Figure 6a. Mean surrogate recoveries for all water samples collected (+ 1 SD).

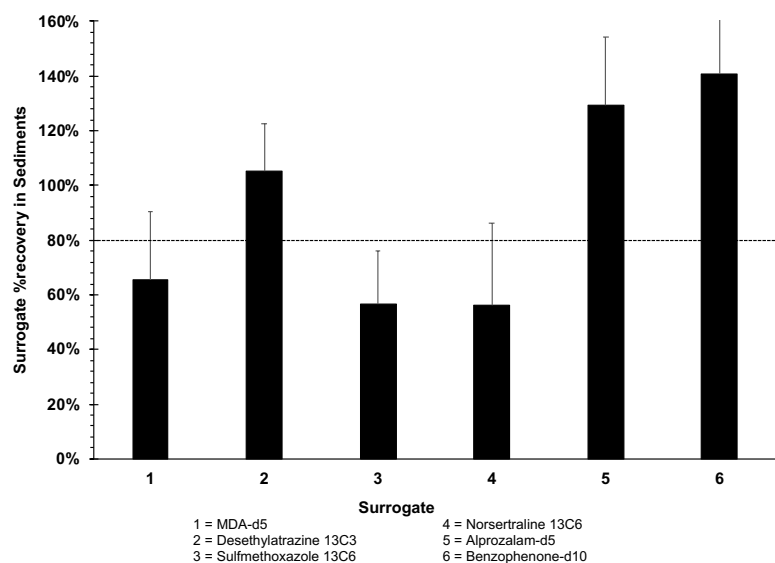


Figure 6b. Mean surrogate recoveries for all sediment samples collected (+ 1 SD).

summarized in Figs. 5a,b. All samples were spiked with surrogate (recovery) standards before beginning the extraction process in order to assess the performance associated with each individual analysis. Surrogate standards typically consist of isotopically labelled (deuterium or C-13) homologues of the target chemicals that mimic the analytical performance of the native contaminants. Three of the surrogate standards exceeded 80% recovery (high performance), including desethylatrazine-13C3, aprolalam-d5 and benzophenone-d10 in water and sediments. The final concentrations reported for the target chemicals were not corrected for surrogate recoveries.

Laboratory blanks included all reagents and containers in contact with the samples, but excluded the normal sample matrix (surface water, particles or riverbed sediment) to serve in determining any background signal responses contributed during method work up. In the lab blanks, Milli-Q Laboratory blanks included all reagents and

containers in contact with the samples, but excluded the normal sample matrix (surface water, particles or riverbed sediment) to serve in determining any background signal responses contributed during method work up. In the lab blanks, Milli-Q water (18 MΩ), clean GF/F filters and boat sand (pre-fired at 450 °C) simulated the sample matrices for water, particles and bed sediments, respectively. The laboratory blanks are used as a subtraction (correction) from the measured sample concentrations in cases where target chemicals were detected in the blanks. Four of the 91 target chemicals were detected in lab blanks at very low concentrations, including cis-tramadol (1.2 ng/L), DEET (1.6 ng/L), carbamazepine (0.23 ng/L) and diclofenac (1.2 ng/L). The detection limits for PPCPs ranged from 0.1 to 4.8 ng/L in water and 0.1 to 5.0 ng/g in

sediments and particles, with the DL defined by a signal-to-noise (S/N) ratio of 3 or by 3-times the lab blank S/N when present.

The results from matrix spike recoveries are provided in Appendix B. Matrix spikes included all target chemicals amended to collected water and sediment samples as a method performance evaluation. Matrix spikes were performed using 80 ng of each target chemical in 1-L of water or 2 g of wet sediment. The matrix spike recoveries in the surface water samples ranged from 0 to 175% with an overall mean of 77%. The 0% recovery values were observed for trans-3'-hydroxycotinine, acyclovir, albuterol, atenolol, 2-hydroxy ibuprofen, hydromorphone, Penicillin G, naloxone, codeine, ciprofloxacin, bupropion, naproxen, budesonide, lisinopril and tetracycline. The matrix spike recoveries in riverbed sediments ranged from 0 to 182% with an overall mean of 71%. The 0% recovery values were observed for acyclovir, atenolol, azithromycin, gabapentin, morphine, 2-hydroxy ibuprofen, hydromorphone, sulfathiazole, aspartame, penicillin G, sulfathiazole, aspartame, enrofloxacin, hydrocodone, MDEA, bupropion, naproxen, budesonide, atorvastatin, lisinopril, tetracycline and furosemide.

Results and Discussion

Potomac River and Cameron Run surface water discharge

The discharges of the Potomac River at Little Falls, MD (Sta. USGS 01646500) and Cameron Run at Alexandria, VA (Sta. USGS 01653000) recorded on the sampling dates are shown in Table 3 (www.usgs.gov). The discharge values represent average daily flow in meters per second.

Table 3. Average daily river discharge recorded on the day of sampling.

Date	Potomac River (m ³ /s)	Cameron Run (m ³ /s)
2-May-18	407	-
15-May-18	684	10.3
12-Jul-18	187	-
16-Jul-18	147	0.15
13-Sep-18	1,613	-
18-Sep-18	828	20.0

Total suspended matter

Total suspended matter (TSM) measured in the Hunting Creek Survey water samples at sites CR01 and UHC01 is summarized in Table 4. TSM was used for PPCP sediment analysis at these sites because no fine-grain bed material was present in the stream. The TSM values represent a single measurement for the entire 20-L sample collected.

Sediment grain size

A summary sandy-silt-clay ternary diagram with data points from every sampling station for each month (72 sediment samples total) shows the overall strong linear trend of the average of

mean grain sizes at these stations (Fig. 6). An analysis of grain size by Hunting Creek Survey station number is provided below.

Table 4. Total suspended matter measured in the water samples at sites CR01 and UHC01.

Site	Date	TSM (mg/L)
CR01-1	15 May 2018	9.42
CR01-2	16 Jul 2018	16.3
CR01-3	18 Sep 2018	NA
UHC01-1	15 May 2018	19.3
UHC01-2	16 Jul 2018	217
UHC01-3	18 Sep 2018	NA

NA = not available from the sampling period.

AR02

May 2018: For May 2018, mean grain size ranged from 24.25 μ m (medium silt) to 57.65 μ m (coarse silt) and the mean was 32.47 μ m (coarse silt); percent sand, silt, and clay ranged from 28.6%, 38.7%, and 5.3% to 55.9%, 64.4%, and 7.2%, respectively, and the mean percent for each was 36.3%, 57.3%, and 6.4%, respectively (Appendix F).

July 2018: For July 2018, mean grain size ranged from 15.65 μ m (medium silt) to 18.42 μ m (medium silt) and the mean was 16.96 μ m (medium silt); percent sand, silt, and clay ranged from 15.7%, 72.3%, and 8.1% to 19.6%, 75.2%, and 9.4%, respectively, and the mean percent for each was 17.2, 74.1, and 8.3, respectively (Appendix F).

Sept 2018: For September 2018, mean grain size ranged from 22.73 μ m (medium silt) to 32.67 μ m (coarse silt) and the mean was 26.63 μ m (medium silt); percent sand, silt, and clay ranged from 27.1%, 53.2%, and 6.8% to 39.9%, 64.8%, and 6.8%, respectively, and the mean percent for each was 32.3%, 60.0%, and 7.3%, respectively (Appendix F).

For all three months together, the mean grain size for station AR02 ranged from 16.96 μ m (medium silt) to 32.47 μ m (coarse silt), with an average of 25.36 μ m (medium silt). The mean percent sand, silt, and clay ranged from minimum values of 17.2%, 57.3%, and 6.4% to maximum values of 36.3%, 74.1%, and 8.3%, respectively, with average values of 28.6%, 63.8%, and 7.3%, respectively (Appendix F). The dominant sediment type at AR02 was sandy silt (Appendix E). A strong linear trend is evident in the sand-silt-clay ternary diagram (Appendix E).

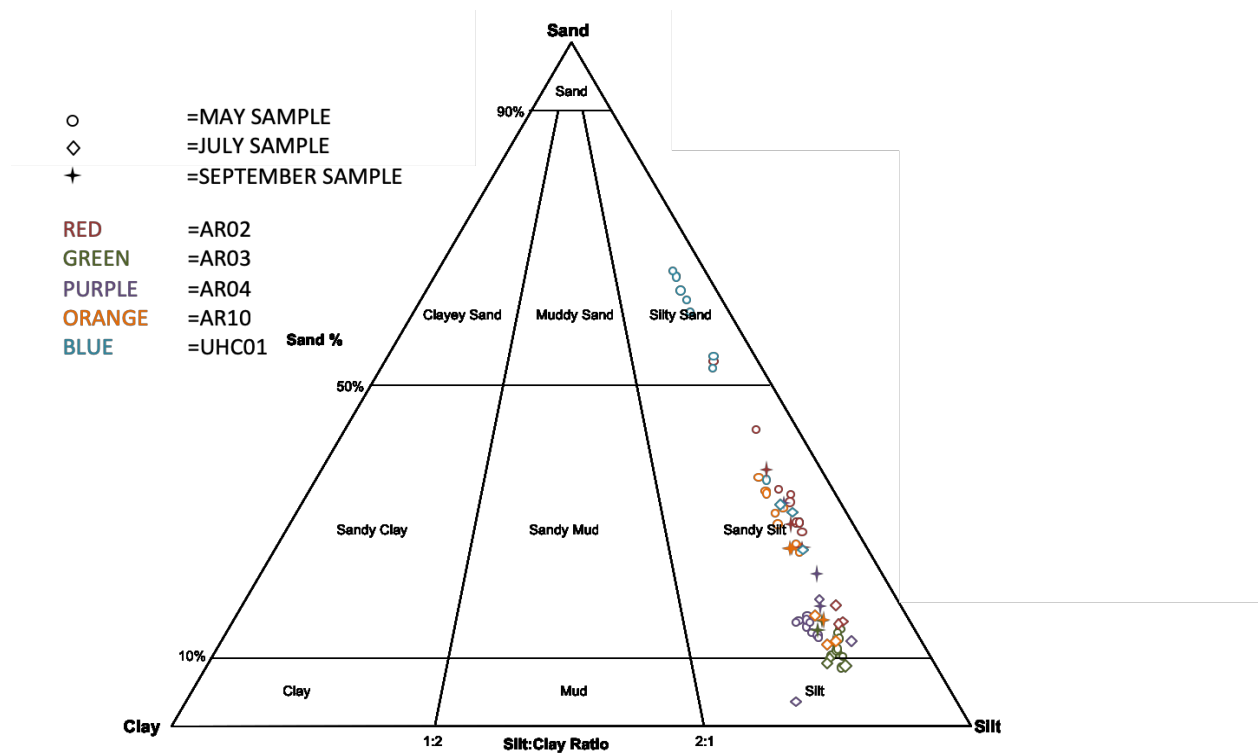


Figure 7. Summary sand-silt-clay ternary diagram for May, July, and September 2018 for all Hunting Creek stations (72 sediment samples total).

AR03

May 2018: For May 2018, mean grain size ranged from 11.63 μ m (fine silt) to 16.15 μ m (medium silt) and the mean was 13.59 μ m (fine silt); percent sand, silt, and clay ranged from 8.4%, 73.0%, and 9.4% to 17.3%, 79.2%, and 12.7%, respectively, and the mean percent for each was 12.3%, 76.6%, 11.1%, respectively (Appendix G).

July 2018: For July 2018, mean grain size ranged from 11.09 μ m (fine silt) to 12.72 μ m (fine silt) and the mean was 11.72 μ m (fine silt); percent sand, silt, and clay ranged from 9.2%, 74.8%, and 11.5% to 12.5%, 79.0%, and 13.8%, respectively, and the mean percent for each was 10.4%, 76.8%, and 12.1%, respectively (Appendix G).

Sept 2018: For September 2018, mean grain size ranged from 13.12 μ m (fine silt) to 15.01 μ m (fine silt) and the mean was 14.00 μ m (fine silt); percent sand, silt, and clay ranged from 13.0%, 71.5%, and 10.9% to 17.1%, 75.8%, and 12.5%, respectively, and the mean percent for each was 14.9%, 73.5%, and 11.6%, respectively (Appendix G).

For all three months together, the mean grain size for station AR03 ranged from 11.72 μ m (fine silt) to 14.00 μ m (fine silt), with an average of 13.10 μ m (fine silt). The percent sand, silt, and clay ranged from minimum values of 10.4%, 73.5%, and 11.1% to maximum values of 14.9%, 76.8%, and 12.1%, respectively, with average values of 12.6%, 75.6%, and 11.6% (Appendix G).

The sediment at AR03 was a mix of sandy silt and silt (Appendix E). No linear trend is present in the sand-silt-clay ternary diagram (Appendix E).

AR04

May 2018: For May 2018, mean grain size ranged from 12.02 μ m (fine silt) to 15.98 μ m (medium silt) and the mean was 13.22 μ m (fine silt); percent sand, silt, and clay ranged from 12.6%, 66.3%, and 12.0% to 21.2%, 74.3%, and 14.9%, respectively, and the mean percent for each was 15.7%, 71.1%, and 13.3%, respectively (Appendix H).

July 2018: For July 2018, mean grain size ranged from 5.92 μ m (very fine silt) to 19.84 μ m (medium silt) and the mean was 14.37 μ m (fine silt); percent sand, silt, and clay ranged from 3.7%, 69.6%, and 8.8% to 20.7, 77.9%, and 21.0%, respectively, and the mean percent for each was 12.6%, 74.3% and 12.4%, respectively (Appendix H).

Sept 2018: For September 2018, mean grain size ranged from 16.01 μ m (medium silt) to 31.09 μ m (coarse silt) and the mean was 23.50 μ m (medium silt); percent sand, silt, and clay ranged from 16.8%, 58.4%, 7.0% to 34.4%, 72.6%, 10.6% and the mean percent for each was 25.3%, 66.1%, 8.6%, respectively (Appendix H).

For all three months together, the mean grain size for station AR04 ranged from 13.22 μ m (fine silt) to 23.50 μ m (medium silt), with an average of 17.03 μ m (medium silt). The percent sand, silt, and clay ranged from minimum values of 12.6%, 66.1%, and 8.6% to maximum values of 25.3%, 74.3%, and 13.2%, respectively, with average values of 17.9%, 70.5%, and 11.4%, respectively (Appendix H). The dominant sediment type at AR04 was sandy silt (Appendix E). A weak linear trend is present in the sand-silt-clay ternary diagram (Appendix E).

AR10

May 2018: For May 2018, mean grain size ranged from 21.25 μ m (medium silt) to 28.56 μ m (medium silt) and the mean was 24.45 μ m (medium silt); percent sand, silt, and clay ranged from 25.9%, 52.9%, and 7.4% to 38.7%, 65.1% and 9.6%, respectively, and the mean percent for each was 31.9%, 59.4%, and 8.7%, respectively (Appendix I).

July 2018: For July 2018, mean grain size ranged from 12.63 μ m (fine silt) to 15.00 μ m (fine silt) and the mean was 13.90 μ m (fine silt); percent sand, silt, and clay ranged from 12.5%, 70.4%, and 10.6% to 18.1%, 76.3%, and 12.3%, respectively, and the mean percent for each was 14.6%, 73.8%, and 11.0%, respectively (Appendix I).

Sept 2018: For September 2018, mean grain size ranged from 14.60 μ m (fine silt) to 23.00 μ m (medium silt) and the mean was 19.65 μ m (medium silt); percent sand, silt, and clay ranged from 16.1%, 62.3%, and 9.5% to 27.9%, 73.1%, and 11.0%, respectively, and the mean percent for each was 23.6%, 66.3%, and 10.1%, respectively (Appendix I).

For all three months together, the mean grain size for station AR10 ranged from 13.90 μ m (fine silt) to 24.45 μ m (medium silt), with an average of 19.33 μ m (medium silt). The percent sand, silt, and clay ranged from minimum values of 14.6%, 59.4%, and 8.7% to maximum values of

31.9%, 73.8%, and 11.0%, respectively, with average values of 23.4%, 66.5%, and 9.9%, respectively (Appendix I). The sediment type at AR10 was sandy silt (Appendix E). A strong linear trend is evident in the sand-silt-clay ternary diagram (Appendix E).

UHC01

May 2018: For May 2018, mean grain size ranged from 31.85 μ m (coarse silt) to 95.16 μ m (very fine sand) and the mean was 75.33 μ m (very fine sand); percent sand, silt, and clay ranged from 36.7%, 27.5%, and 3.9% to 68.5%, 55.6%, and 7.7%, respectively, and the mean percent for each was 60.1%, 34.8%, and 5.0%, respectively (Appendix J).

July 2018: For July 2018, mean grain size ranged from 21.41 μ m (medium silt) to 27.27 μ m (medium silt) and the mean was 24.90 μ m (medium silt); percent sand, silt, and clay ranged from 26.7%, 57.3%, and 6.8% to 35.2%, 64.9%, and 8.4%, respectively, and the mean percent for each was 31.2%, 61.2%, and 7.3%, respectively (Appendix J).

For May and July 2018, the mean grain size for station UHC01 ranged from 24.90 μ m (medium silt) to 75.33 μ m (very fine sand), with an average of 50.12 μ m (coarse silt). The percent sand, silt, and clay ranged from minimum values of 31.2%, 34.9, and 5.0% to maximum values 60.1%, 61.2%, and 7.3%, respectively (Appendix J). The sediment at UHC01 was a mix of silty sand and sandy silt (Appendix E). A strong linear trend is evident in the sand-silt-clay ternary diagram (Appendix E).

PPCP detection frequencies

The detection frequencies of the 91-PPCP target chemicals in surface water, riverbed sediment and filtered particles are shown in Table 5. Overall, 39 of the PPCPs were detected in water, sediment or particles while 52 were undetected in any matrix. In Table 5 the PPCP target chemicals are grouped into high (>70%), moderate (>25%) and low (>0%) frequency categories. The high frequency PPCPs were commonly detected in all matrices, including water, sediment and particles. The moderate frequency PPCPs typically include those detected with high frequency in one matrix (water or sediment) only (with propoxyphene being the exception). The low frequency PPCPs were those detected <70% frequency in any single matrix.

Table 5. Percent detection frequencies (%DF) of the 39 target chemicals found in water, sediment and particle samples.

PPCP	%DF water	%DF sediment	%DF particles	Average %DF
High frequency				
DEET	100%	100%	100%	100%
cis-Tramadol	100%	100%	87%	96%
Venlafaxine	80%	100%	100%	93%
Diphenhydramine	83%	100%	91%	92%
Carbamazepine	89%	97%	83%	90%
Methadone	81%	100%	70%	84%
Desvenlafaxine	85%	100%	65%	83%
Triamterene	83%	100%	57%	80%

Dextromethorphan	78%	100%	43%	74%
Metoprolol	74%	97%	48%	73%

Moderate frequency

Propranolol	56%	100%	43%	66%
Diltiazem	59%	92%	43%	65%
Fexofenadine	89%	42%	52%	61%
Fluoxetine	44%	100%	35%	60%
Sertraline	7%	100%	70%	59%
Escitalopram	7%	100%	48%	52%
Nicotine	100%	50%	0%	50%
Verapamil	0%	100%	48%	49%
Amitriptyline	7%	97%	39%	48%
Fentanyl	0%	100%	35%	45%
10,11-Carbamazepine epoxide	85%	0%	22%	36%
Caffeine	100%	0%	0%	33%
Triclocarban	0%	100%	0%	33%
Propoxyphene	0%	69%	26%	32%
Bupropion	91%	0%	0%	30%
Sulfamethoxazole	85%	0%	0%	28%
Nortriptyline	0%	81%	0%	27%

Low frequency

Diclofenac	67%	0%	0%	22%
Hydrochlorothiazide	59%	0%	0%	20%
Azithromycin	35%	8%	0%	15%
Paroxetine	0%	25%	0%	8%
Metformin	24%	0%	0%	8%
Celecoxib	20%	0%	0%	7%
Atorvastatin	17%	0%	0%	6%
Perfluorooctanoic Acid	4%	11%	0%	5%
Ranitidine	11%	0%	0%	4%
Gabapentin	7%	0%	0%	2%
Temazepam	7%	0%	0%	2%
Warfarin	6%	0%	0%	2%

PPCP concentrations

Concentrations of the 39 PPCPs detected in the Hunting Creek Survey water samples (i.e., those listed in Table 5) were expressed as total concentrations (Σ^{39} PPCP) in Fig. 7a. The Σ^{39} PPCP concentrations provide a concise way to display the geospatial trends in PPCP concentrations among sampling sites. The lowest Σ^{39} PPCP concentrations were found at the end-member stations, CR01 (upstream reference) and AR04/10 (Potomac River reference sites), and the highest total concentrations were found at upper and lower Hunting Creek stations (UHC01 and AR02/03). In terms of temporal variation, the upper and lower Hunting Creek and Potomac River stations showed the lowest overall concentrations in the September samples when river

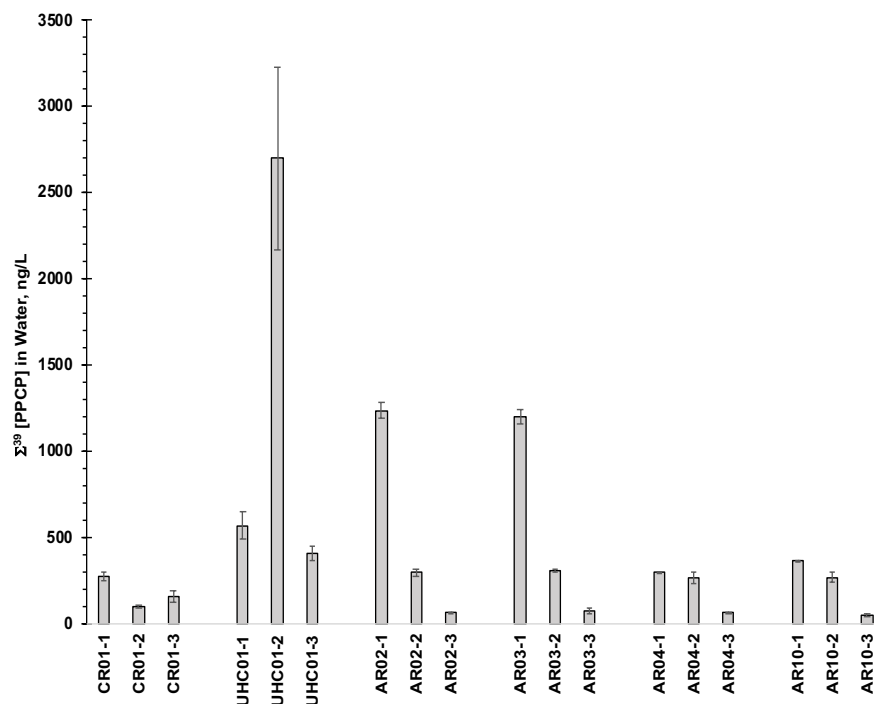


Figure 7a. Concentrations of total PPCPs (Σ^{39} PPCP) in surface water samples by sampling station (± 1 SD).

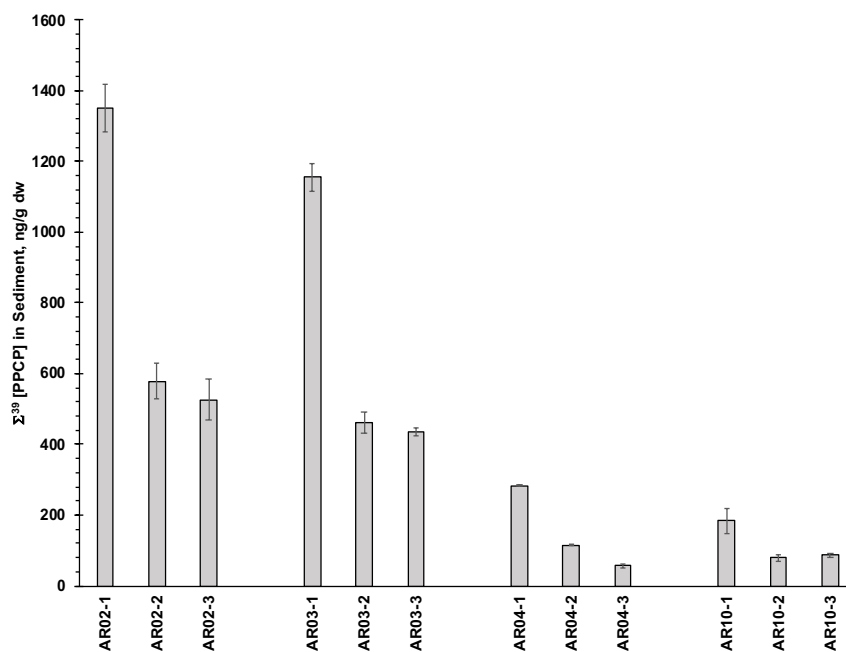


Figure 7b. Total concentrations of PPCPs (Σ^{39} PPCP) in riverbed sediments by sampling station (± 1 SD).

flows (Table 3) were the greatest (i.e., maximum dilution of the discharged PPCPs). For the upstream CR01 station, July showed the lowest observed Σ^{39} PPCP concentration 1 water sample had the highest observed Σ^{39} PPCP concentration overall ($>2,500$ ng/L), followed by AR02 and

AR03 in May 2018 (1,100 ng/L). There was variability in the temporal concentrations among the sampling stations, where maxima in Σ^{39} PPCP concentrations did not coincide.

Concentrations of the Σ^{39} PPCP in the Hunting Creek Survey sediments are shown in Fig. 7b. Only the Lower Hunting Creek and Potomac River stations are illustrated because the upstream stations did not have fine-grained bed sediments present. The Σ^{39} PPCP concentrations were greater in the Lower Hunting Creek stations (AR02/03) relative to Potomac River stations (AR04/10) for all sampling dates. The relative seasonal (May-Sep) abundances of the Σ^{39} PPCP concentrations were similar across all the sampling sites (highest in May and lower in July and September). Sites AR02/03 were both located in the Lower Hunting Creek channel cut through the head delta of Lower Hunting Creek, which can be observed via aerial photographs. The temporal maxima in Σ^{39} PPCP correlates with the lowest water column temperature observed during the May sampling period. The degradation rates of environmental pollutants are kinetically slowest at the lowest environmental temperatures based on activation energy effects on rates (Tinsley 2011).

The following charts display the individual 39 PPCPs detected in water and sediment samples for each sampling site. The individual scales of the charts vary by site so the low concentrations of certain PPCPs can be more easily visualized.

The measured concentrations of the PPCPs in water samples collected from Cameron Run in 2018 are illustrated in Fig. 8a. The prominent PPCPs in Cameron Run water included DEET (insect repellent), nicotine and caffeine, with lower concentrations of carbamazepine and cis-tramadol. This site represents the background PPCPs in the Hunting Creek watershed above the head of tide that are delivered to the tidal river.

The measured concentrations of the PPCPs in water samples collected at UHC01 in 2018 are illustrated in Fig. 8b, where the concentrations in water were found at much greater concentrations than Cameron Run. The prominent PPCPs include fexofenadine (Allegra), cis-tramadol, desvenlafaxine, caffeine, metoprolol, nicotine, sulfamethoxazole, dextromethorphan, venlafaxine, DEET, and hydrochlorothiazide. The greatest concentrations generally occurred in the July samples, but some PPCPs, including caffeine and nicotine, showed the highest concentrations in September.

The measured concentrations of the PPCPs in water samples collected at AR02 in 2018 are illustrated in Fig. 9a. The concentrations of PPCPs at AR02 were lower relative to UHC01, although fexofenadine was present at concentration approaching that of UHC01. The PPCP profile was similar to UHC01 in chemical composition and relative concentrations among the observed chemicals.

Site AR03 closely resembled AR02 in PPCP composition and relative abundance (Fig. 9b), with the exception of cis-tramadol and fexofenadine. Both were present at relatively high

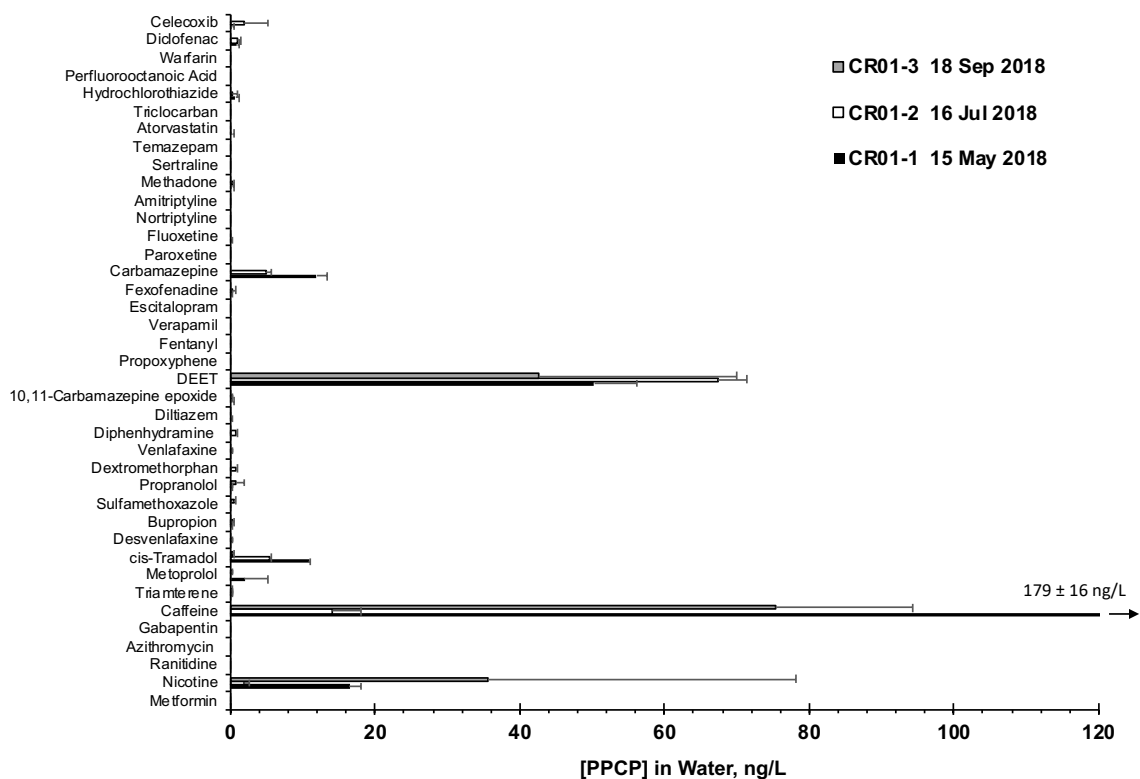


Figure 8a. Concentrations of PPCPs measured in water from site CR01 (+1 SD).

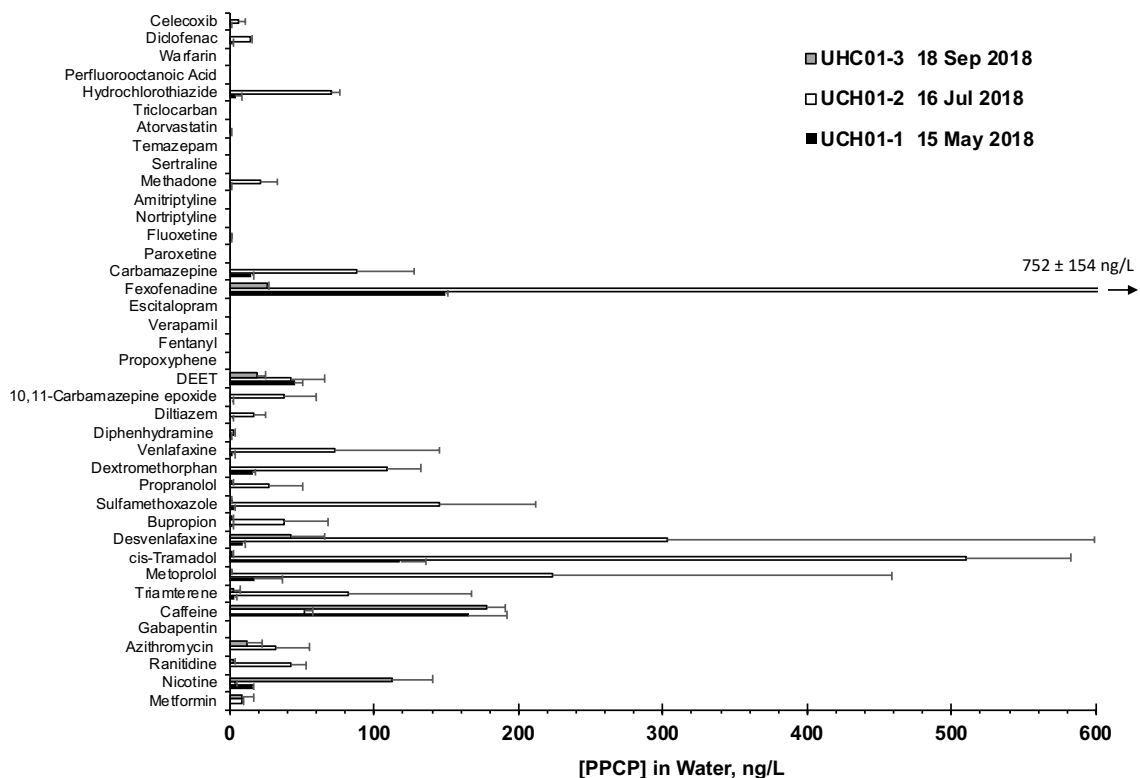


Figure 8b. Concentrations of PPCPs in water collected from site UHC01 (+1 SD).

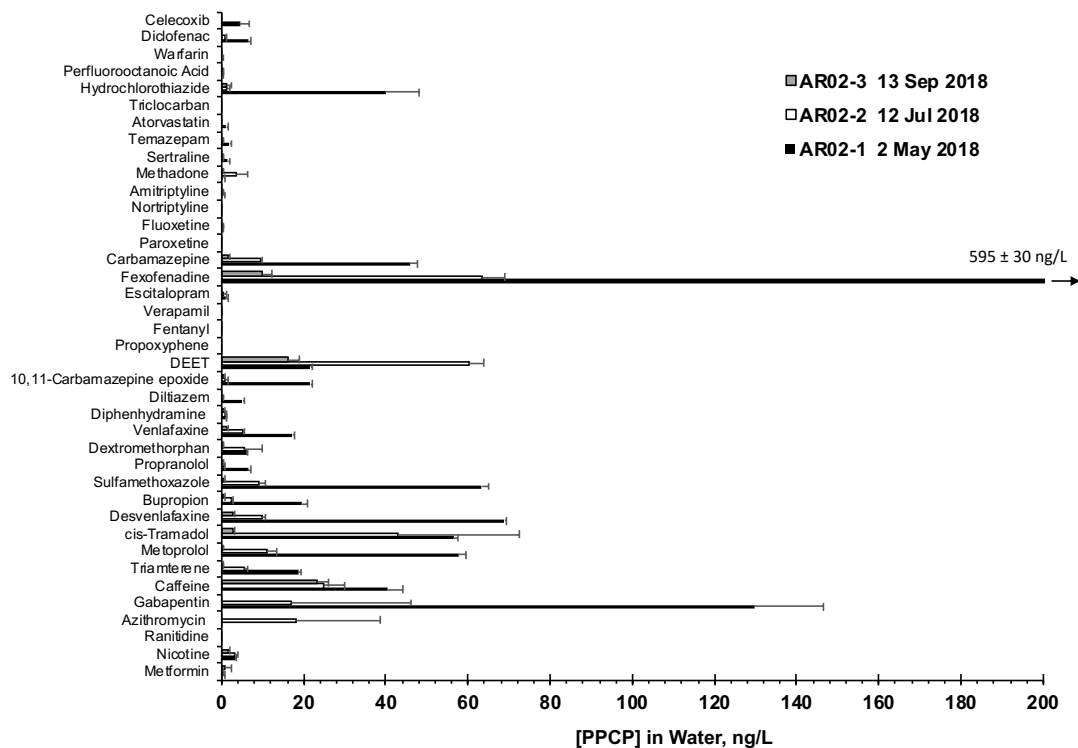


Figure 9. Concentrations of PPCPs in water collected from site AR02 (+1 SD).

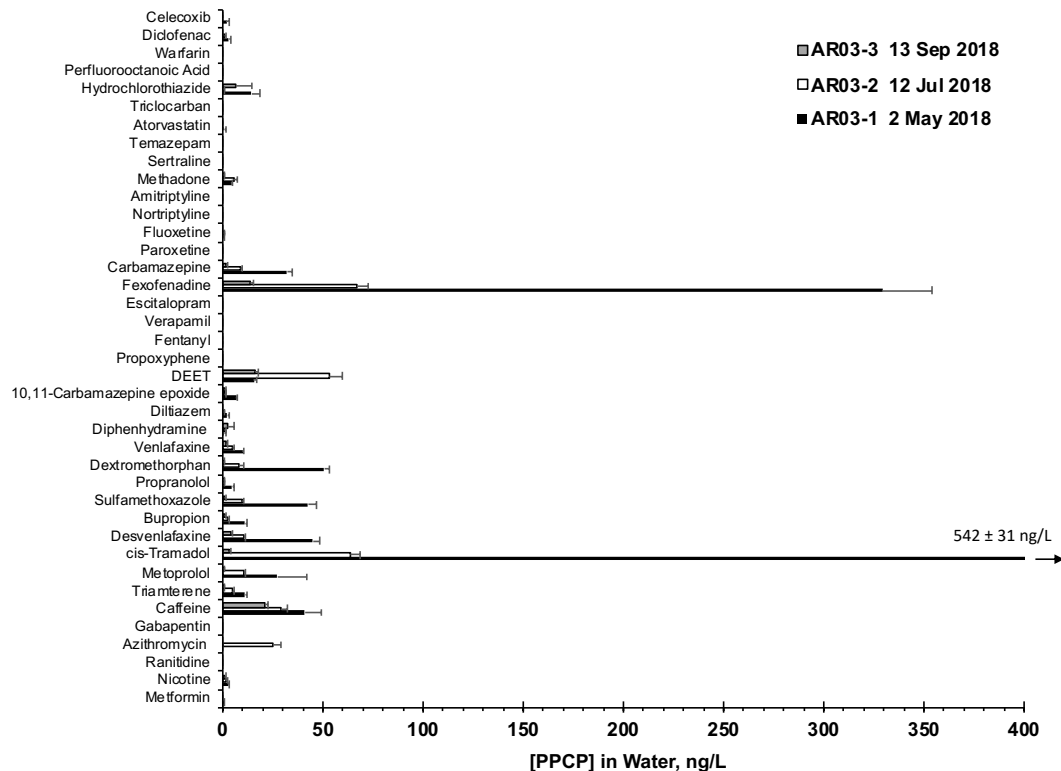


Figure 9. Concentrations of PPCPs in water collected from site AR03 (+1 SD).

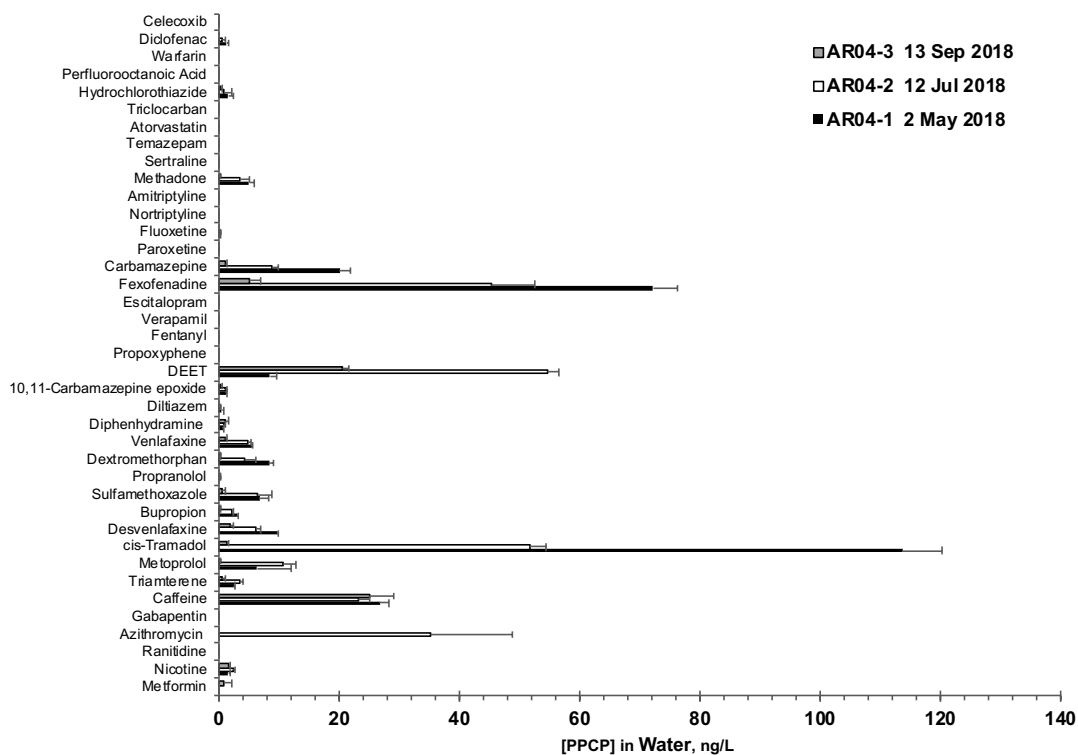


Figure 11. Concentrations of PPCPs in water collected from site AR04 (+1 SD).

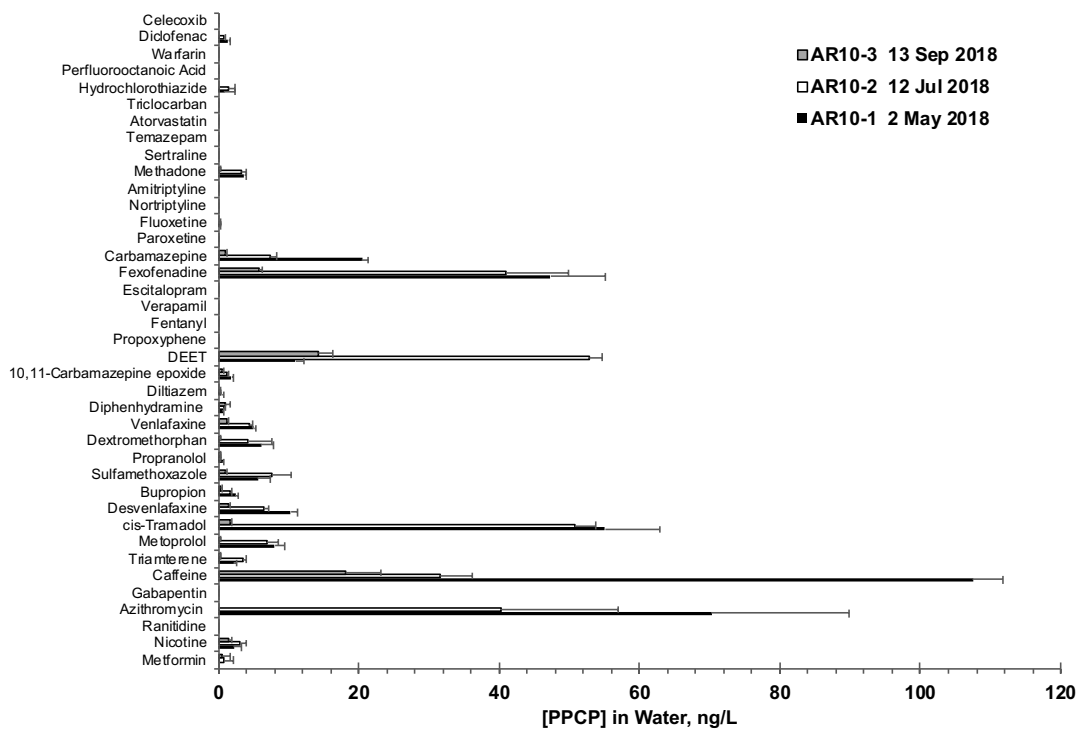


Figure 11. Concentrations of PPCPs in water collected at site AR10 (+1 SD).

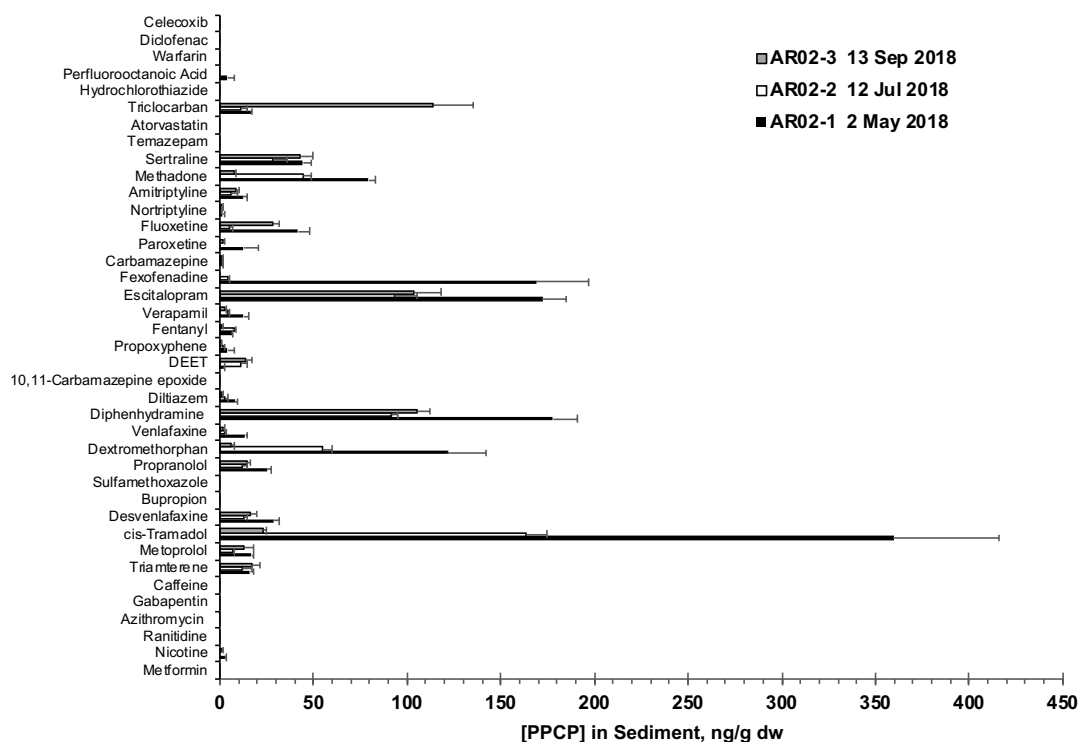


Figure 13. Concentrations of PPCPs in sediment collected from site AR02 (+1 SD).

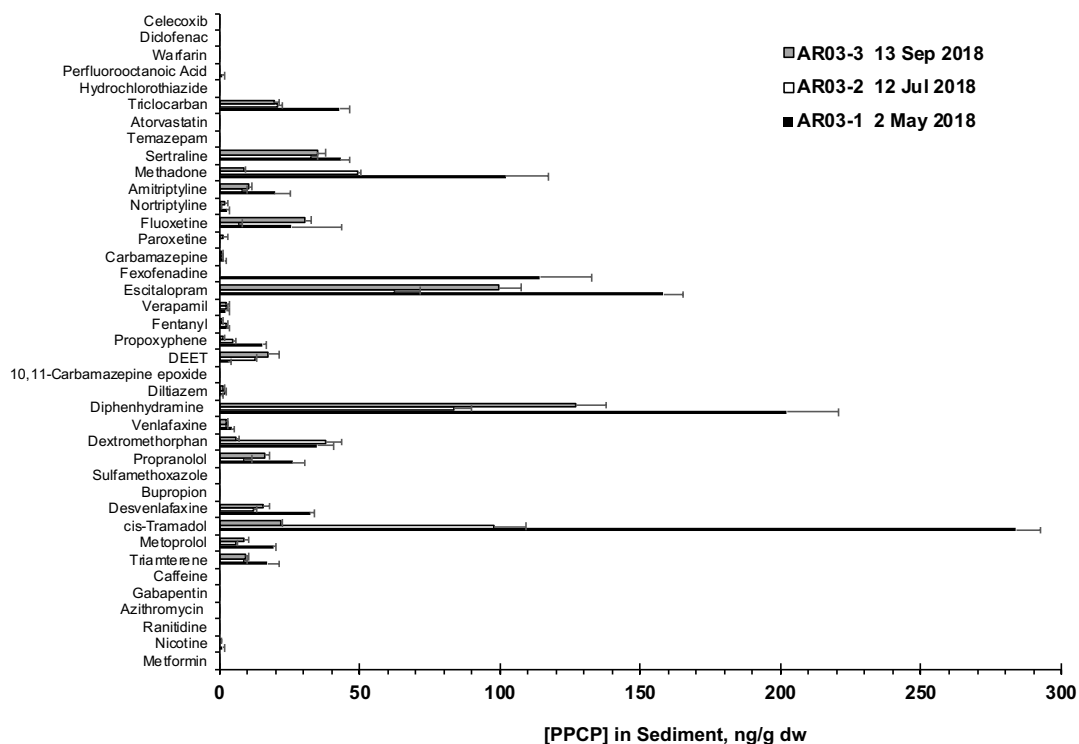


Figure 13. Concentrations of PPCPs in sediment from site AR03 (+1 SD).

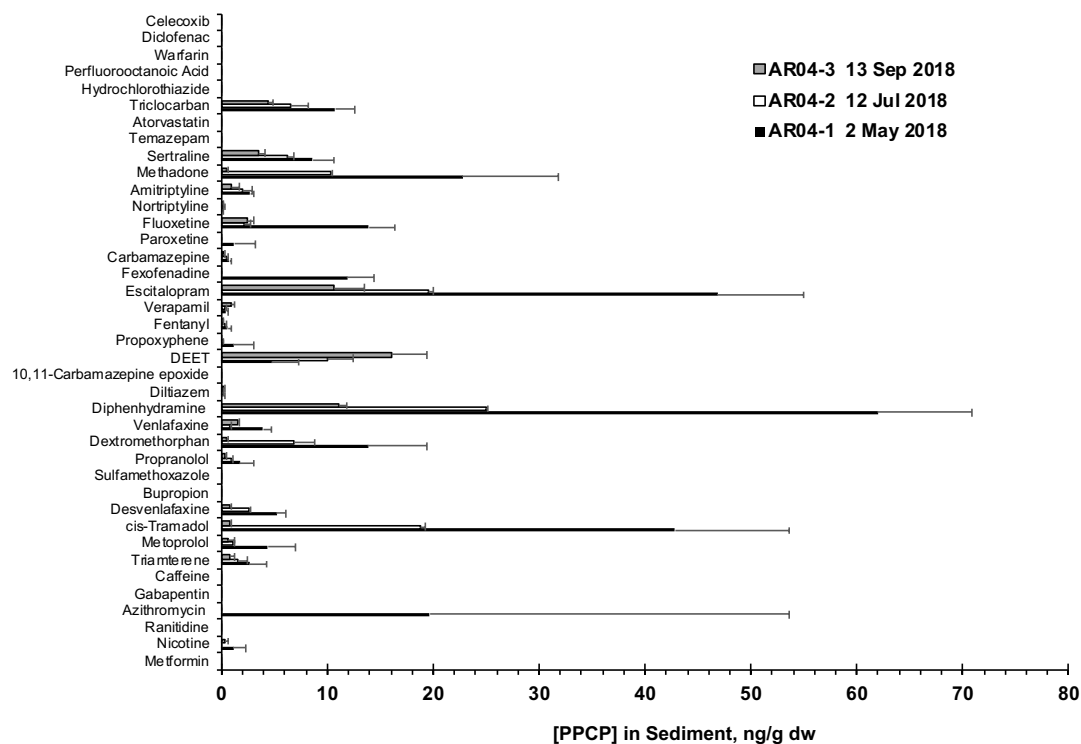


Figure 15. Concentrations of PPCPs in sediment collected from site AR04 (+1 SD).

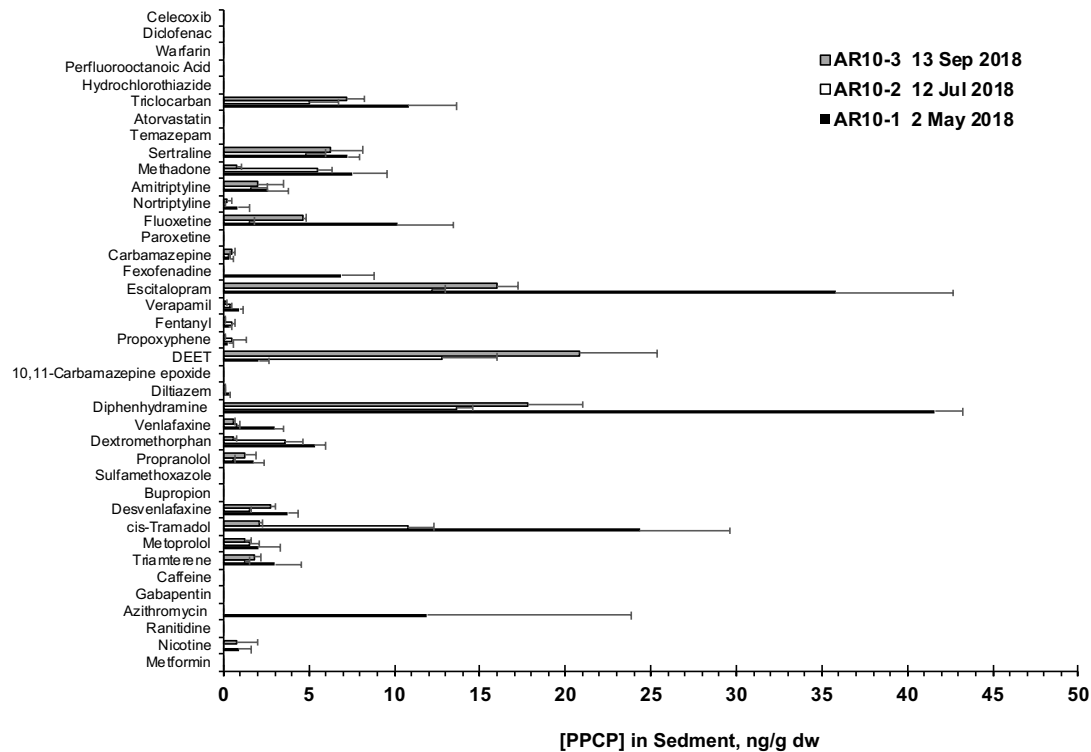


Figure 15. Concentrations of PPCPs in sediments collected from site AR10 (+1 SD).

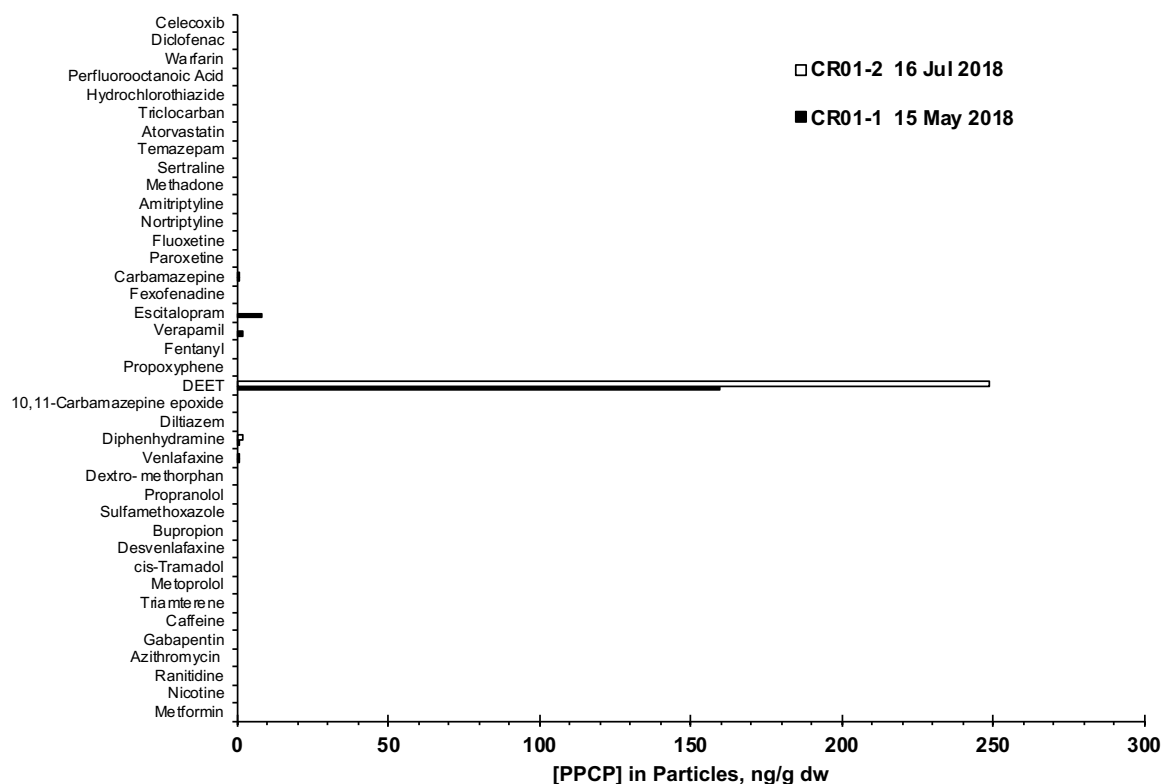


Figure 17. Concentrations of PPCPs in particles collected from site CR01.

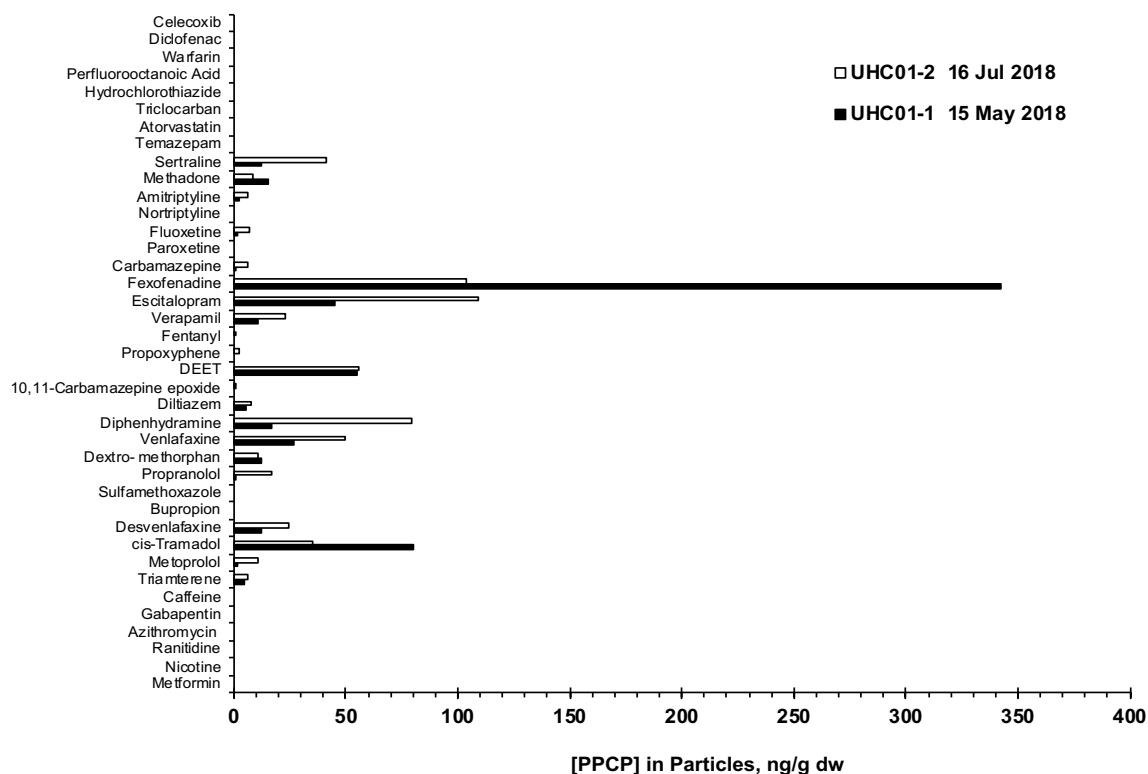


Figure 17. Concentrations of PPCPs in particles collected from site UHC01.

concentrations but the relative concentrations of these two chemicals were reversed between the two sites. Other differences were more subtle.

PPCP concentrations at sites AR04 and AR10 were similar in composition and relative abundance (Figs 10a,b). PPCP concentrations at these sites were lower than AR02/03. Sites AR04 and AR10 were located within the mainstem of the Potomac River where dilution is undoubtedly much greater than in Hunting Creek.

A few aspects regarding PPCP concentrations at site AR10 were unique. AR10 showed the highest concentrations of azithromycin, and the caffeine concentration was comparable to that seen at UHC01.

Riverbed sediments showed distinct profiles of the PPCPs in comparison to water Figs. 11-12. PPCPs that were more concentrated in sediments or occurred more frequently relative to water included fluoxetine, sertraline, fentanyl, escitalopram, diphenhydramine, methadone, amitriptyline, nortriptyline, and triclocarban. The PPCPs detected in sediment are characterized by having larger K_{ow} values (Appendix D) than those that predominate in water. A larger K_{ow} favors partitioning into sediment organic matter (OM), that is, the larger K_{ow} PPCPs are more soluble in non-polar phases like natural OM and concentrate there.

The concentrations of PPCPs in river particles (filtered solids) are shown in Figs. 13a,b. DEET was the only predominant PPCP in CR01 particles, while fexofenadine, sertraline, escitalopram, DEET, diphenhydramine, venlafaxine, desvenlafaxine and cis-tramadol were the most abundant PPCPs observed in particles from UHC01.

Summary and Conclusions

- In the 2018 Hunting Creek Survey, 39 selected PPCP micropollutants were detected in water, sediments and suspended particles out of a total 91 PPCP micropollutants that were monitored. The most frequently detected PPCPs included DEET, cis-tramadol, venlafaxine, diphenhydramine, carbamazepine, methadone, desvenlafaxine, triamterene, dextromethorphan, and metoprolol.
- The best approach in assessing the sources of micropollutants is to compare the upstream versus downstream presence and concentrations of PPCPs in streams and rivers relative to the point of WTP discharge. Cameron Run served as the upstream reference in this study, where caffeine, nicotine and DEET were prominent chemicals (>50 ng/L) in stream waters above the head of tide discharging into the tidal freshwater Potomac River, followed by lower concentrations of cis-tramadol, metoprolol and carbamazepine (1-20 ng/L).
- Water collected from Upper Hunting Creek (UHC01), the WTP discharge zone, showed the presence of many PPCPs at much higher concentrations than that found in the Cameron Run water samples. PPCPs present at a concentration >200 ng/L included cis-tramadol, metoprolol, fexofenadine, and desvenlafaxine; PPCPs >100 ng/L at UHC01 included nicotine, caffeine, sulfamethoxazole, dextromethorphan, and venlafaxine; and PPCPs >20 ng/L

included ranitidine, azithromycin, triamterene, bupropion, propanol, carbamazepine, 10,11-carbamazepine epoxide, diltiazem, DEET, methadone and hydrochlorothiazide.

- The concentrations of PPCPs in water decreased downstream of UHC01 at stations AR02 and AR03. The chemical profile of PPCPs was similar to UHC01 at these stations, but present at lower concentrations. Fexofenadine and cis-tramadol were the only PPCPs present equal to or greater than 200 ng/L. Gabapentin was detected at AR02 and not at UHC01 making it unique to these stations. Site AR03 showed generally lower concentrations of PPCPs except for cis-tramadol and fexofenadine, with a similar profile as station AR02.
- The concentrations of PPCPs in water at sites AR04 and AR10 were much lower than that found in Lower Hunting Creek but with generally the same chemical profile (relative abundance) as the Lower and Upper Hunting Creek stations. Thus, these PPCPs are common to the main stem Potomac River as well.
- PPCPs were also frequently detected in sediments or particles except for site CR01, where DEET was the only PPCP detected with a significant concentration. Many more PPCPs at higher concentrations were detected in particles from UHC01 relative to Cameron Run. Also, along with cis-tramadol and fexofenadine, PPCPs that were more prominent in sediment relative to water included diphenhydramine, triclocarban, sertraline, methadone, amitriptyline, fluoxetine, escitalopram, verapamil, fentanyl, and dextromethorphan. PPCP concentrations were greatest in sediments at sites AR02 and decreased in concentration at sites AR03, AR04 and AR10 showing a clear downstream gradient in Hunting Creek leading to the main stem Potomac River.
- The ternary diagrams regarding surficial sediment grain size tend to show a distinct linear trend in the average of the mean grain sizes, which is explained by a widely ranging sand percentage in the sediment compared to the relatively constant ratio of silt to clay.
- The average of the mean sediment grain sizes and average percent sand, silt, and clay for each sampling station are as follows:
 - AR02: 25.36 μ m (medium silt); 28.6%, 63.8%, and 7.3%
 - AR03: 13.10 μ m (fine silt); 12.6%, 75.6%, and 11.6%
 - AR04: 17.03 μ m (medium silt); 17.9%, 70.5%, and 11.4%
 - AR10: 19.33 μ m (medium silt); 23.4%, 66.5%, and 9.9%
 - UHC01: 50.12 μ m (coarse silt); 45.7%, 48.0%, and 6.1%

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Appendix A. List of PPCPs analyzed in the 2018 Hunting Creek Survey by LC-MS/MS.

Chemical	Type ^a	LC RT (min) ^b
<u>Internal Standards</u>		
Methamphetamine-d5	ISTD	1.954
Caffeine-13C3	ISTD	2.288
Oxycodone-d3	ISTD	2.242
Ciprofloxacin-d8	ISTD	2.869
Sulfomethazine-13C6	ISTD	3.103
Diazepam-d5	ISTD	6.361
<u>Surrogate Standards</u>		
MDA-d5	SSTD	1.910
Desethylatrazine-13C3	SSTD	2.888
Hydrocodone-d6	SSTD	2.477
Sulfomethoxazole-13C6	SSTD	3.789
Norsertaline-13C6	SSTD	5.675
Alprazolam-d5	SSTD	5.793
Benzophenone-d10	SSTD	6.836
<u>Target Chemicals</u>		
Metformin	Target	0.393
Nicotine	Target	0.454
trans-3'-Hydroxycotinine	Target	0.449
Acyclovir	Target	0.454
Cimetidine	Target	1.127
Cotinine	Target	0.458
Albuterol	Target	0.923
Atenolol	Target	1.024
Ranitidine	Target	0.463
Azithromycin	Target	2.146
Gabapentin	Target	1.506
Morphine	Target	0.933
Oxymorphone	Target	1.073
Clonidine	Target	1.784
2-Hydroxy Ibuprofen	Target	1.926
Hydromorphone	Target	1.000
Nadolol	Target	2.141
Caffeine	Target	2.284
Sulfathiazole	Target	2.457
Aspartame	Target	2.392
Penicillin G	Target	2.384
(±)-Amphetamine	Target	1.731
(±)-Methamphetamine	Target	2.001
Triamterene	Target	2.605
Naloxone	Target	1.888
MDA	Target	1.939
Codeine	Target	2.005

Ciprofloxacin	Target	2.903
Phentermine	Target	1.983
Metoprolol	Target	2.949
Sulfamethazine	Target	3.107
Naltrexone	Target	2.311
MDMA	Target	2.319
Enrofloxacin	Target	3.219
Formoterol	Target	3.301
Atrazine_Mercapturate	Target	3.324
Hydrocodone	Target	2.486
cis-Tramadol	Target	3.132
Desvenlafaxine	Target	2.656
MDEA	Target	2.655
Bupropion	Target	3.646
Sulfamethoxazole	Target	3.781
Enalapril	Target	4.235
Propranolol	Target	4.316
Meperidine	Target	3.536
Sulfadimethoxine	Target	4.518
Dextromethorphan	Target	4.549
Sulfaquinoxaline	Target	4.638
Venlafaxine	Target	3.812
Diphenhydramine	Target	4.701
Diltiazem	Target	5.137
10_11-Carbamazepine epoxide	Target	4.293
Promethazine	Target	5.169
DEET	Target	5.346
Propoxyphene	Target	5.512
Fentanyl	Target	5.250
Verapamil	Target	5.679
Escitalopram	Target	4.806
Benzotropine	Target	5.758
Buprenorphine	Target	5.800
Fexofenadine	Target	5.773
Carbamazepine	Target	5.059
Loratadine	Target	6.019
Naproxen	Target	6.101
Oxazepam	Target	5.295
Paroxetine	Target	5.289
Fluoxetine	Target	5.502
Nordiazepam	Target	5.365
Bezafibrate	Target	6.229
Nitrazepam	Target	5.332
(±)-Lorazepam	Target	5.427
Budesonide	Target	6.392
Nortriptyline	Target	5.514
Amitriptyline	Target	5.662
Methadone	Target	5.818
Clonazepam	Target	6.200
Alprazolam	Target	5.815
Sertraline	Target	5.885

Temazepam	Target	6.070
Flunitrazepam	Target	6.210
Diazepam	Target	6.417
Atorvastatin	Target	7.194
Triclocarban	Target	7.374
Lisinopril	Target	7.474
Tetracycline	Target	7.789
Hydrochlorothiazide	Target	2.171
Furosemide	Target	5.141
Perfluorooctanoic Acid	Target	5.220
Glipizide	Target	5.974
Warfarin	Target	6.644
Diclofenac	Target	7.139
Celecoxib	Target	7.222

^a ITSD = internal standard

SSTD = surrogate standard

Target = target unknown

^bUHPLC retention time in minutes

Appendix B. List of PPCP MRM ions and quadrupole voltages used in LC-MS/MS analysis.

Compound	MRM Ions (m/z) Precursor	Voltages (V)			
		M1 M2 M3	Q1	CE	Q3
Metformin	130.4	60.20	-10.0	-15.0	-10.0
		71.20	-11.0	-22.0	-12.0
		85.20	-10.0	-15.0	-15.0
Nicotine	163.3	130.30	-13.0	-22.0	-25.0
		117.30	-12.0	-28.0	-20.0
		132.30	-13.0	-19.0	-25.0
trans-3'-Hydroxycotinine	193.3	80.25	-15.0	-25.0	-15.0
		111.30	-14.0	-13.0	-20.0
		106.30	-15.0	-25.0	-22.0
Acyclovir	226.3	152.30	-10.0	-14.0	-27.0
		135.10	-10.0	-27.0	-26.0
		185.20	-17.0	-8.0	-17.0
Cimetidine	253.3	95.15	-10.0	-31.0	-17.0
		159.15	-10.0	-15.0	-10.0
		117.15	-10.0	-16.0	-21.0
Cotinine	177.3	80.20	-14.0	-26.0	-16.0
		98.25	-14.0	-30.0	-18.0
		136.20	-11.0	-13.0	-25.0
Albuterol	240.4	148.20	-10.0	-19.0	-28.0
		222.25	-10.0	-11.0	-14.0
		166.20	-10.0	-13.0	-17.0
Atenolol	267.3	145.25	-11.0	-26.0	-15.0
		190.25	-11.0	-20.0	-12.0
		225.20	-11.0	-18.0	-14.0
Ranitidine	315.3	176.25	-12.0	-18.0	-11.0
		130.20	-12.0	-26.0	-27.0
		102.20	-12.0	-35.0	-19.0
Azithromycin	591.5	116.10	-22.0	-35.0	-11.0
		158.40	-22.0	-31.0	-29.0
		186.50	-24.0	-37.0	-11.0
Gabapentin	172.4	154.30	-14.0	-14.0	-29.0
		137.30	-14.0	-20.0	-12.0
		95.20	-13.0	-23.0	-16.0
Morphine	286.4	152.20	-11.0	-51.0	-28.0
		201.20	-11.0	-25.0	-13.0
		165.20	-12.0	-40.0	-16.0
Oxymorphone	302.3	284.15	-12.0	-20.0	-19.0
		227.25	-12.0	-29.0	-14.0
		242.25	-12.0	-29.0	-16.0
Clonidine	230.2	44.20	-18.0	-25.0	-17.0
		213.15	-16.0	-26.0	-13.0
		160.25	-17.0	-34.0	-10.0
MDA d5	185.2	168.25	-14.0	-11.0	-11.0
		110.25	-13.0	-22.0	-22.0
		138.25	-14.0	-19.0	-14.0
2-Hydroxy Ibuprofen	221.3	180.25	-16.0	-10.0	-11.0
		121.20	-17.0	-29.0	-22.0
		139.15	-18.0	-19.0	-28.0

+/- Methamphetamine d5	155.2	92.15	-12.0	-21.0	-19.0
		91.15	-12.0	-21.0	-17.0
		121.20	-12.0	-14.0	-23.0
Hydromorphone	286.3	185.20	-12.0	-30.0	-11.0
		157.20	-12.0	-42.0	-15.0
		128.30	-12.0	-54.0	-23.0
Naldolol	310.4	254.35	-12.0	-19.0	-17.0
		201.30	-13.0	-23.0	-13.0
		236.20	-13.0	-21.0	-16.0
Caffeine	195.3	138.25	-15.0	-19.0	-26.0
		42.10	-15.0	-46.0	-14.0
		110.30	-14.0	-23.0	-21.0
Caffeine 13C3	198.1	140.20	-14.0	-19.0	-22.0
		112.20	-14.0	-23.0	-22.0
		43.15	-14.0	-35.0	-15.0
Oxycodone d3	319.2	301.2	-13.0	-20.0	-20.0
		244.20	-12.0	-29.0	-16.0
		259.20	-12.0	-26.0	-17.0
+/- Amphetamine	136.1	65.15	-13.0	-40.0	-26.0
		91.20	-13.0	-20.0	-20.0
		119.25	-13.0	-14.0	-23.0
MDA	180.4	163.25	-10.0	-12.0	-17.0
		105.15	-14.0	-21.0	-10.0
Naloxone	328.4	310.20	-13.0	-20.0	-22.0
		268.30	-13.0	-27.0	-12.0
Sulfathiazole	256.2	92.10	-10.0	-27.0	-16.0
		156.10	-10.0	-15.0	-10.0
		108.15	-10.0	-25.0	-20.0
Aspartame	295.3	120.35	-12.0	-28.0	-22.0
		180.30	-12.0	-15.0	-11.0
		235.25	-12.0	-15.0	-15.0
Penicillin G	335.3	289.15	-13.0	-27.0	-19.0
		128.10	-11.0	-28.0	-27.0
		91.20	-10.0	-42.0	-16.0
Hydrocodone d6	306.2	202.15	-12.0	-32.0	-20.0
		174.15	-12.0	-40.0	-18.0
		128.20	-12.0	-54.0	-23.0
Methamphetamine	150.0	91.20	-27.0	-25.0	-11.0
		65.20	-15.0	-40.0	-11.0
		119.25	-15.0	-15.0	-11.0
Triamterene	254.3	237.20	-10.0	-26.0	-16.0
		141.20	-10.0	-45.0	-13.0
		104.20	-10.0	-40.0	-18.0
Desethylatrazine 13C3	191.1	149.20	-14.0	-16.0	-14.0
		106.10	-14.0	-25.0	-20.0
		80.15	-14.0	-27.0	-16.0
Codeine	300.3	165.30	-12.0	-43.0	-10.0
		215.30	-12.0	-25.0	-13.0
		225.15	-23.0	-27.0	-15.0
Ciprofloxacin d8	340.1	322.25	-13.0	-22.0	-22.0
		235.15	-24.0	-38.0	-15.0
		296.25	-24.0	-19.0	-14.0
Ciprofloxacin	332.3	314.20	-13.0	-21.0	-14.0
		231.35	-13.0	-34.0	-15.0
		288.40	-13.0	-20.0	-13.0
Phentermine	150.0	91.20	-12.0	-35.0	-16.0

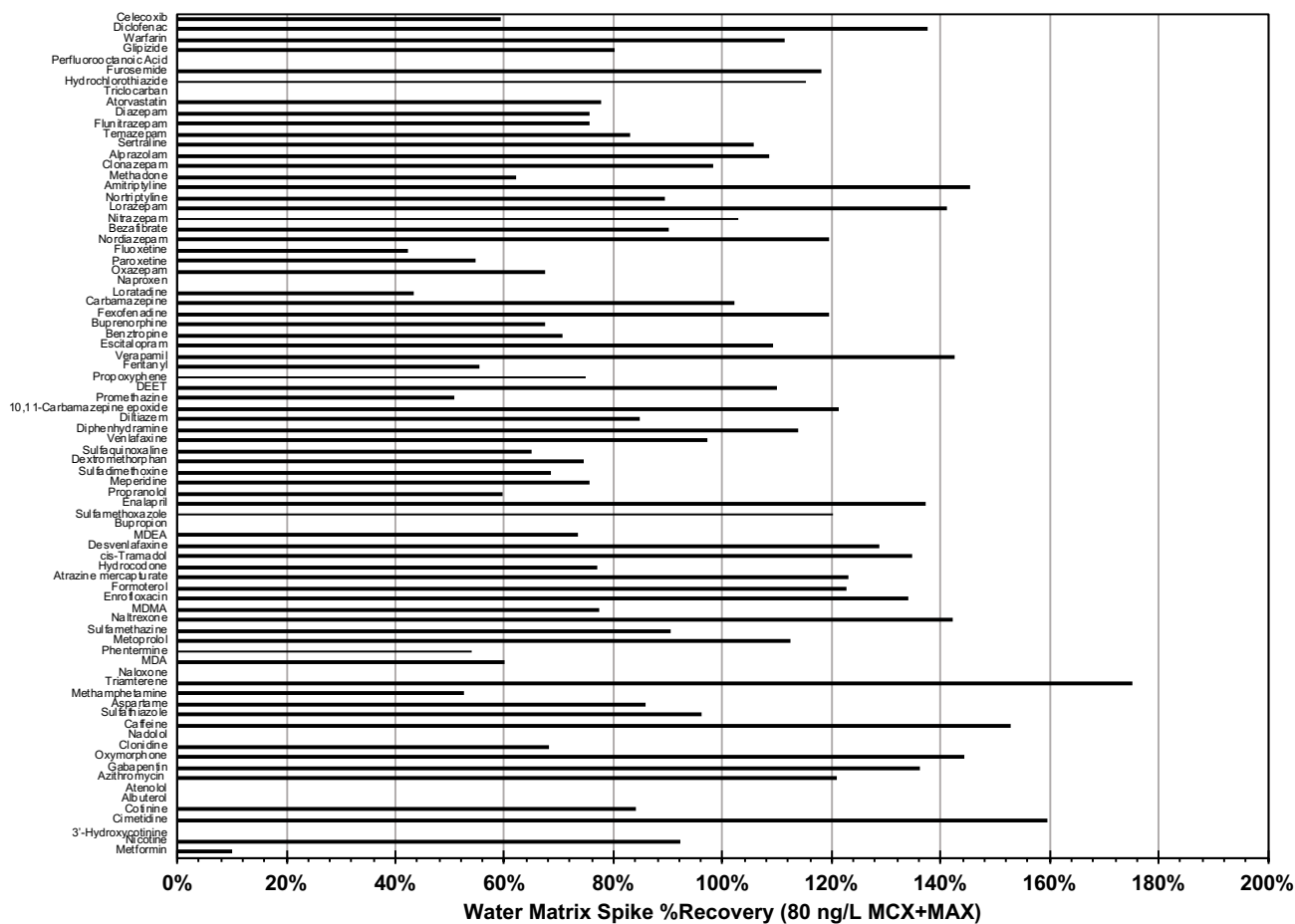
		65.10	-13.0	-41.0	-25.0
		39.20	-13.0	-50.0	-14.0
Metoprolol	268.4	116.20	-11.0	-20.0	-20.0
		56.20	-11.0	-29.0	-19.0
		133.25	-11.0	-25.0	-13.0
Sulfamethazine	279.3	186.20	-11.0	-19.0	-12.0
		92.20	-11.0	-31.0	-18.0
		124.20	-11.0	-22.0	-21.0
Sulfomethazine 13C6	285.1	186.10	-11.0	-19.0	-19.0
		124.20	-11.0	-24.0	-25.0
		98.15	-11.0	-29.0	-17.0
Naltrexone	342.4	324.20	-11.0	-23.0	-22.0
		270.30	-14.0	-27.0	-17.0
MDMA	194.4	163.35	-10.0	-14.0	-10.0
		105.15	-10.0	-23.0	-20.0
		135.20	-11.0	-20.0	-28.0
Enrofloxacin	360.3	316.40	-12.0	-20.0	-21.0
		342.35	-14.0	-26.0	-23.0
		245.15	-14.0	-29.0	-16.0
Formoterol	345.4	149.30	-13.0	-21.0	-28.0
		121.20	-11.0	-33.0	-22.0
		327.25	-14.0	-15.0	-23.0
Atrazine-Mercapturate	343.3	214.25	-14.0	-19.0	-14.0
		172.15	-13.0	-30.0	-11.0
		102.10	-14.0	-41.0	-19.0
Hydrocodone	300.3	199.20	-12.0	-29.0	-20.0
		171.15	-12.0	-39.0	-28.0
		128.20	-12.0	-54.0	-21.0
cis-Tramadol	264.0	58.20	-11.0	-22.0	-23.0
Desvenlafaxine	264.4	58.20	-11.0	-21.0	-10.0
		246.25	-11.0	-13.0	-16.0
		107.30	-11.0	-35.0	-20.0
MDEA	208.4	163.35	-11.0	-14.0	-10.0
		105.20	-11.0	-26.0	-18.0
		135.20	-10.0	-20.0	-26.0
Bupropion	240.3	184.20	-10.0	-13.0	-12.0
		131.20	-10.0	-25.0	-25.0
		130.25	-10.0	-40.0	-25.0
Sulfamethoxazole	254.3	92.10	-10.0	-30.0	-19.0
		65.10	-10.0	-44.0	-10.0
		108.25	-10.0	-22.0	-20.0
Sulfomethoxazole 13C6	260.1	162.10	-10.0	-15.0	-10.0
		98.10	-10.0	-27.0	-19.0
		114.10	-10.0	-23.0	-11.0
Ethyl Paraben 13C6	173.2	101.20	-13.0	-17.0	-19.0
		145.20	-13.0	-13.0	-15.0
		83.20	-13.0	-26.0	-16.0
Enalapril	377.4	234.2	-10.0	-20.0	-15.0
		91.15	-10.0	-54.0	-17.0
		117.30	-15.0	-38.0	-22.0
Propanolol	260.3	116.20	-11.0	-20.0	-22.0
		183.25	-11.0	-19.0	-18.0
		56.10	-11.0	-27.0	-22.0
Meripidine	248.4	174.20	-10.0	-20.0	-11.0
		220.35	-10.0	-22.0	-14.0
		70.20	-11.0	-30.0	-27.0

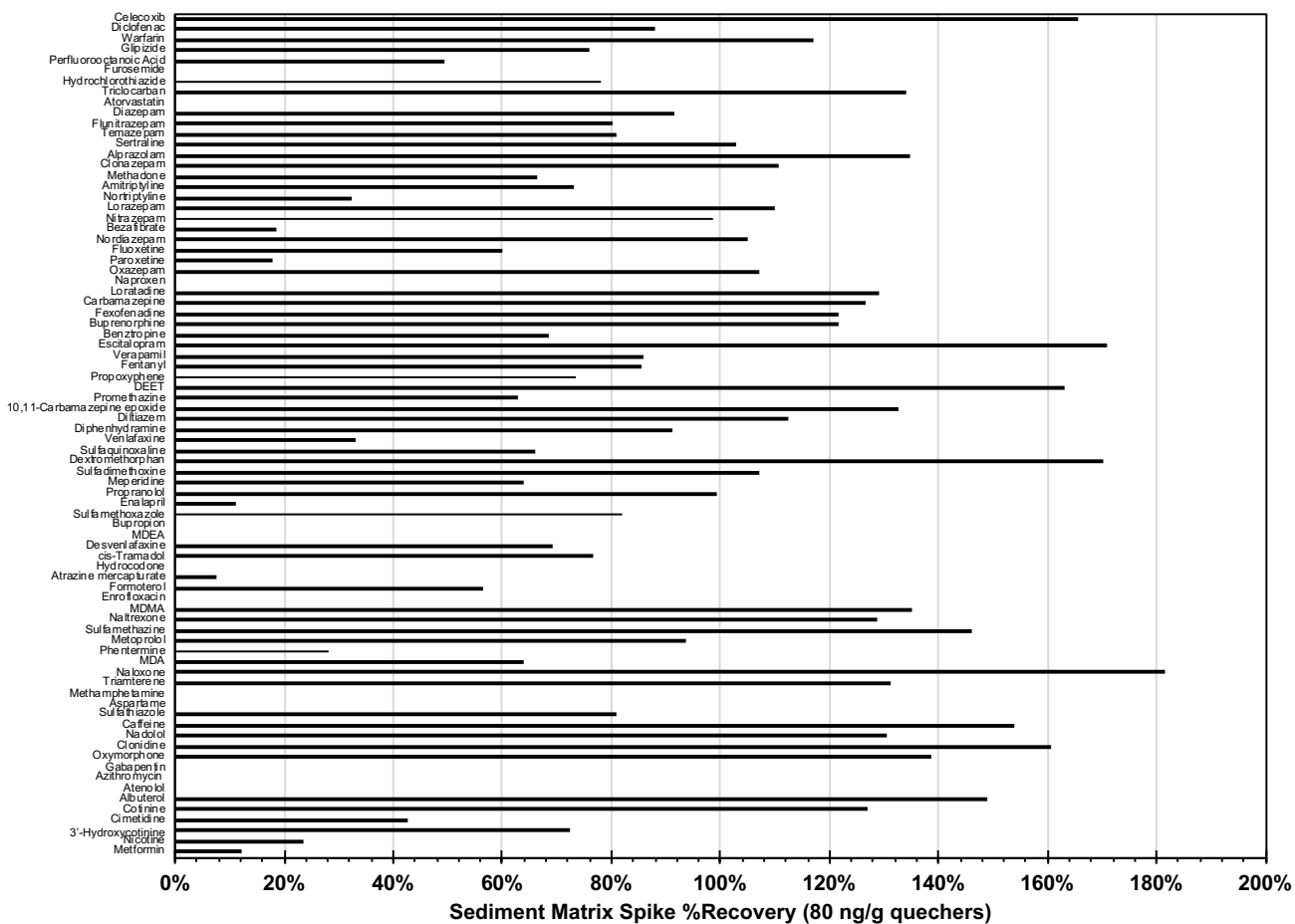
Sulfadimethoxine	311.3	156.25	-10.0	-21.0	-10.0
		92.10	-12.0	-36.0	-17.0
		108.20	-10.0	-33.0	-21.0
Dextromethorphan	272.4	215.25	-11.0	-24.0	-14.0
		171.20	-11.0	-39.0	-17.0
		147.20	-11.0	-29.0	-14.0
Sulfaquinoxaline	301.2	92.10	-12.0	-33.0	-17.0
		137.10	-12.0	-28.0	-26.0
		156.15	-10.0	-16.0	-15.0
Venlafaxine	278.4	58.25	-11.0	-21.0	-10.0
		260.30	-11.0	-13.0	-18.0
		121.20	-11.0	-29.0	-23.0
Diphenhydramine	256.3	167.20	-10.0	-19.0	-17.0
		152.20	-11.0	-36.0	-14.0
		165.20	-10.0	-40.0	-16.0
Diltiazem	415.3	178.20	-10.0	-25.0	-11.0
		150.20	-10.0	-45.0	-15.0
		109.25	-10.0	-55.0	-10.0
10,11-Carbamazepine Epoxide	253.3	180.30	-10.0	-30.0	-19.0
		236.20	-10.0	-11.0	-15.0
		210.15	-19.0	-14.0	-13.0
Promethazine	285.3	86.2	-11.0	-21.0	-16.0
		198.15	-11.0	-24.0	-20.0
		71.20	-11.0	-45.0	-12.0
DEET	192.3	91.20	-15.0	-32.0	-18.0
		119.25	-15.0	-20.0	-11.0
		89.60	-11.0	-19.0	-17.0
Propoxyphene	340.4	58.20	-13.0	-25.0	-10.0
		266.25	-13.0	-10.0	-18.0
		91.10	-14.0	-49.0	-17.0
Fentanyl	337.4	188.40	-14.0	-25.0	-12.0
		105.30	-14.0	-36.0	-20.0
		103.15	-14.0	-50.0	-19.0
Verapamil	455.4	165.30	-11.0	-28.0	-16.0
		150.35	-11.0	-41.0	-16.0
		303.25	-11.0	-28.0	-20.0
Escitalopram	325.4	109.10	-12.0	-28.0	-20.0
		262.20	-10.0	-21.0	-17.0
		234.10	-13.0	-29.0	-25.0
Norsertaline 13C6	281.0	159.05	-20.0	-20.0	-10.0
		123.10	-20.0	-44.0	-25.0
		89.15	-20.0	-54.0	-16.0
Benztropine	308.4	167.35	-12.0	-30.0	-10.0
		152.20	-12.0	-51.0	-15.0
		165.25	-12.0	-54.0	-16.0
Alprazolam d5	314.1	286.15	-12.0	-27.0	-19.0
		210.20	-12.0	-43.0	-21.0
		279.20	-12.0	-27.0	-19.0
Buprenorphine	468.5	396.30	-19.0	-41.0	-14.0
		55.25	-12.0	-47.0	-20.0
		414.35	-12.0	-35.0	-14.0
Fexofenadine	502.4	466.40	-20.0	-29.0	-16.0
		484.30	-20.0	-23.0	-17.0
		171.20	-20.0	-42.0	-11.0
Carbamazepine	237.3	194.25	-10.0	-19.0	-20.0
		192.25	-18.0	-22.0	-19.0

		193.25	-10.0	-32.0	-12.0
Loratadine	383.3	337.15	-15.0	-25.0	-24.0
		267.20	-14.0	-30.0	-18.0
		266.15	-15.0	-46.0	-17.0
Naproxen	185.3	170.20	-14.0	-18.0	-18.0
		141.20	-12.0	-30.0	-27.0
		153.10	-15.0	-21.0	-25.0
Oxazepam	287.2	241.10	-12.0	-23.0	-27.0
		269.10	-12.0	-17.0	-12.0
		104.15	-12.0	-35.0	-18.0
Paroxetine	330.3	192.40	-14.0	-22.0	-13.0
		70.25	-13.0	-29.0	-12.0
		44.20	-14.0	-23.0	-16.0
Fluoxetine	310.2	44.25	-13.0	-13.0	-16.0
		148.30	-13.0	-10.0	-15.0
		115.10	-13.0	-12.0	-16.0
Nordiazepam	271.2	140.25	-11.0	-29.0	-26.0
		226.25	-11.0	-28.0	-16.0
		165.20	-11.0	-28.0	-17.0
Bezafibrate	362.3	139.20	-15.0	-27.0	-13.0
		121.20	-12.0	-30.0	-11.0
		316.15	-14.0	-16.0	-20.0
Nitrazepam	282.3	236.20	-12.0	-25.0	-26.0
		180.25	-12.0	-37.0	-11.0
		207.30	-11.0	-35.0	-13.0
Diazepam d5	290.1	198.20	-11.0	-31.0	-21.0
		154.15	-11.0	-27.0	-16.0
		227.15	-11.0	-28.0	-10.0
Lorazepam	321.3	275.05	-12.0	-21.0	-19.0
		303.10	-13.0	-17.0	-14.0
		229.10	-13.0	-31.0	-15.0
Budesonide	431.4	413.30	-11.0	-13.0	-14.0
		237.35	-10.0	-31.0	-25.0
		173.40	-11.0	-29.0	-17.0
Nortriptyline	264.3	233.25	-11.0	-15.0	-15.0
		91.15	-11.0	-23.0	-17.0
		105.20	-11.0	-22.0	-21.0
Amitriptyline	278.4	233.30	-11.0	-19.0	-10.0
		91.15	-11.0	-28.0	-18.0
		117.30	-11.0	-22.0	-23.0
Methadone	310.4	265.25	-13.0	-16.0	-18.0
		105.25	-13.0	-29.0	-20.0
		77.20	-13.0	-54.0	-14.0
Clonazepam	316.3	270.10	-10.0	-26.0	-18.0
		241.10	-10.0	-36.0	-16.0
		214.25	-10.0	-38.0	-13.0
Alprazolam	309.3	281.15	-12.0	-27.0	-19.0
		205.30	-13.0	-41.0	-21.0
		274.25	-13.0	-26.0	-18.0
Sertraline	306.2	159.10	-12.0	-28.0	-16.0
		275.15	-12.0	-13.0	-12.0
Benzophenone d10	193.2	110.20	-14.0	-17.0	-20.0
		82.20	-14.0	-34.0	-15.0
		54.20	-15.0	-55.0	-21.0
Temazepam	301.2	255.20	-12.0	-23.0	-28.0
		283.15	-12.0	-14.0	-19.0

Flunitrazepam	314.2	177.15	-12.0	-40.0	-11.0
		268.30	-13.0	-27.0	-18.0
		239.15	-13.0	-34.0	-16.0
Diazepam	285.3	183.20	-24.0	-53.0	-18.0
		193.25	-12.0	-33.0	-12.0
		257.20	-12.0	-23.0	-17.0
Atorvastatin	559.3	154.20	-12.0	-28.0	-15.0
		250.00	-22.0	-48.0	-24.0
		440.40	-22.0	-24.0	-15.0
Triclocarban	315.2	380.15	-22.0	-31.0	-26.0
		127.10	-13.0	-29.0	-25.0
		93.15	-13.0	-40.0	-18.0
Lisinopril	406.4	128.15	-13.0	-20.0	-13.0
		84.25	-16.0	-30.0	-15.0
		365.10	-15.0	-16.0	-26.0
Tetracycline	445.2	245.40	-30.0	-30.0	-11.0
		341.10	-11.0	-19.0	-16.0
		429.15	-11.0	-15.0	-14.0
Hydrochlorothiazide	296.2	73.30	-11.0	-38.0	-13.0
		205.20	16.0	20.0	14.0
		121.20	15.0	30.0	11.0
Furosemide	329.0	269.10	16.0	17.0	13.0
		285.25	17.0	15.0	10.0
		205.15	17.0	23.0	21.0
Perfluorooctanoic Acid	413.0	126.15	17.0	32.0	27.0
		369.15	21.0	10.0	13.0
		169.20	21.0	18.0	11.0
Glipizide	444.2	119.20	22.0	24.0	15.0
		319.25	23.0	22.0	11.0
		170.20	23.0	30.0	17.0
Warfarin	307.1	161.25	15.0	19.0	11.0
		250.30	16.0	23.0	12.0
		117.20	16.0	35.0	12.0
Diclofenac	239.9	250.05	15.0	11.0	12.0
Celecoxib	380.1	316.30	19.0	23.0	11.0
		276.25	19.0	30.0	13.0
		296.30	19.0	25.0	14.0

Appendix C. Summary of matrix spike recoveries in water and sediments.



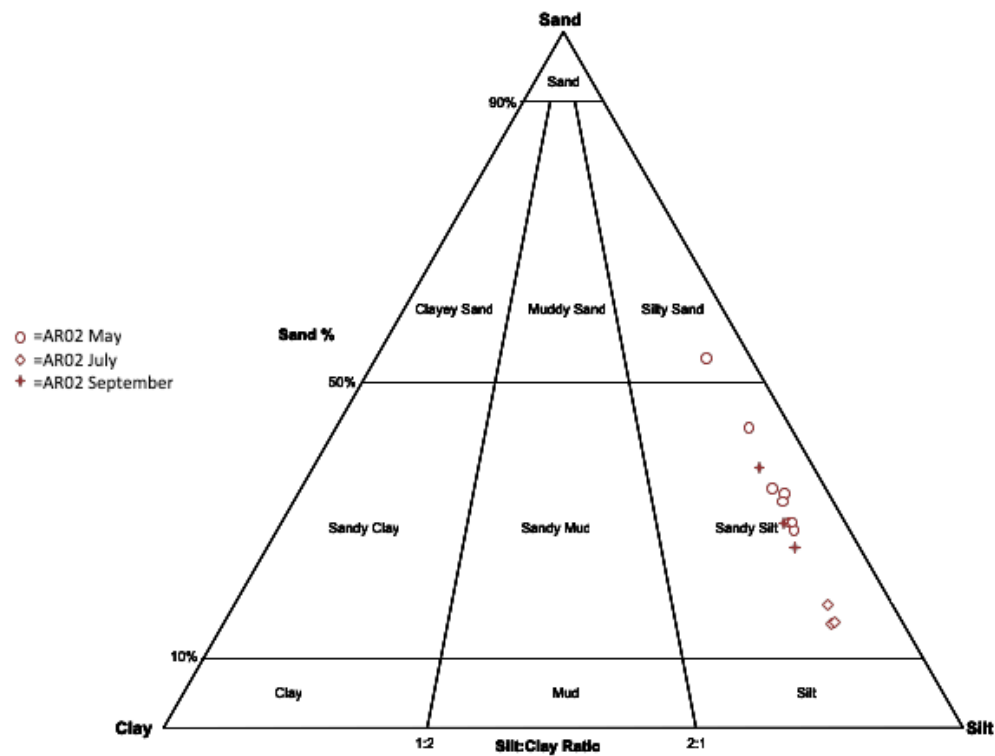


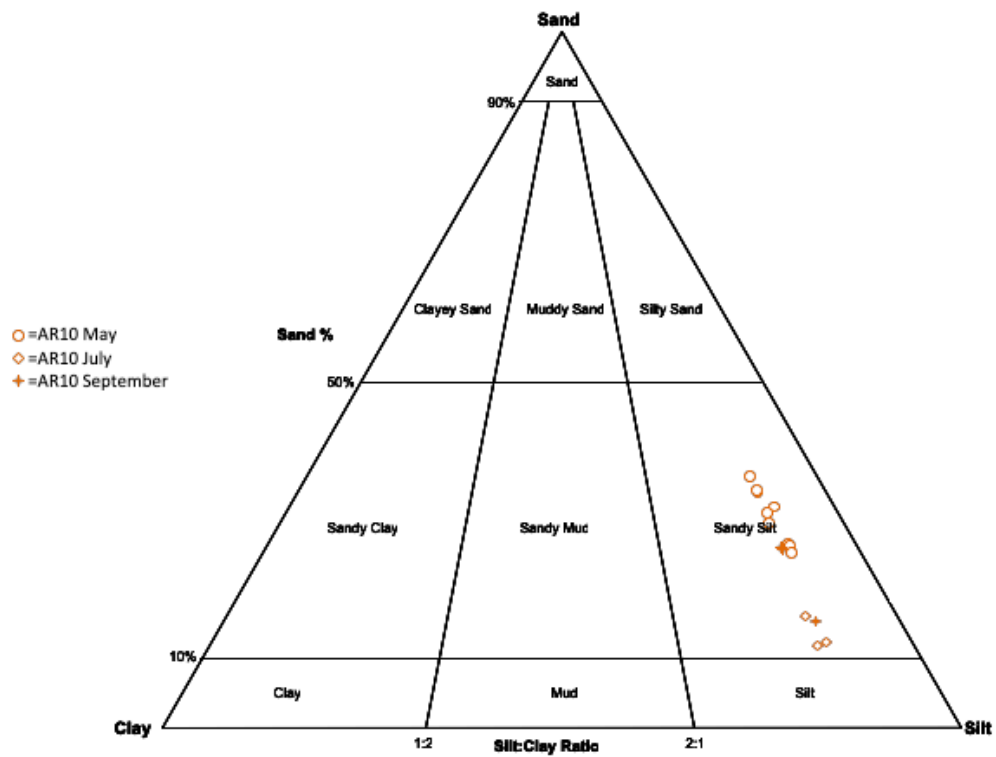
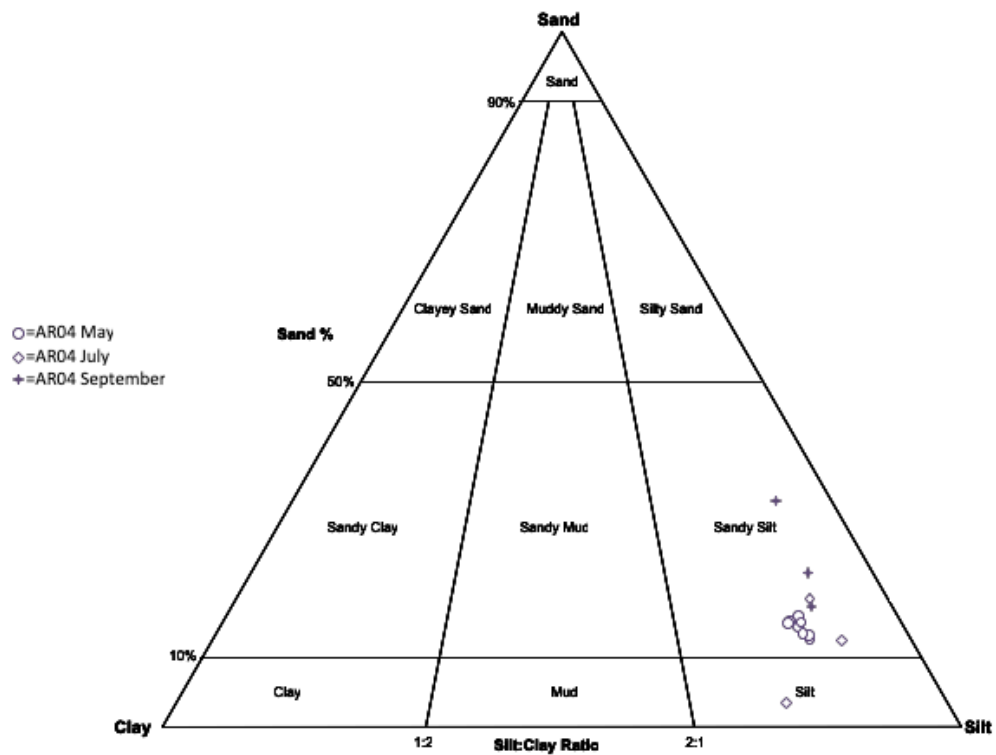
Appendix D. List of the log octanol/water partition coefficients (log K_{ow}) for the 39 PPCPs. (Source of K_{ow} values was www.Chemspider.com.)

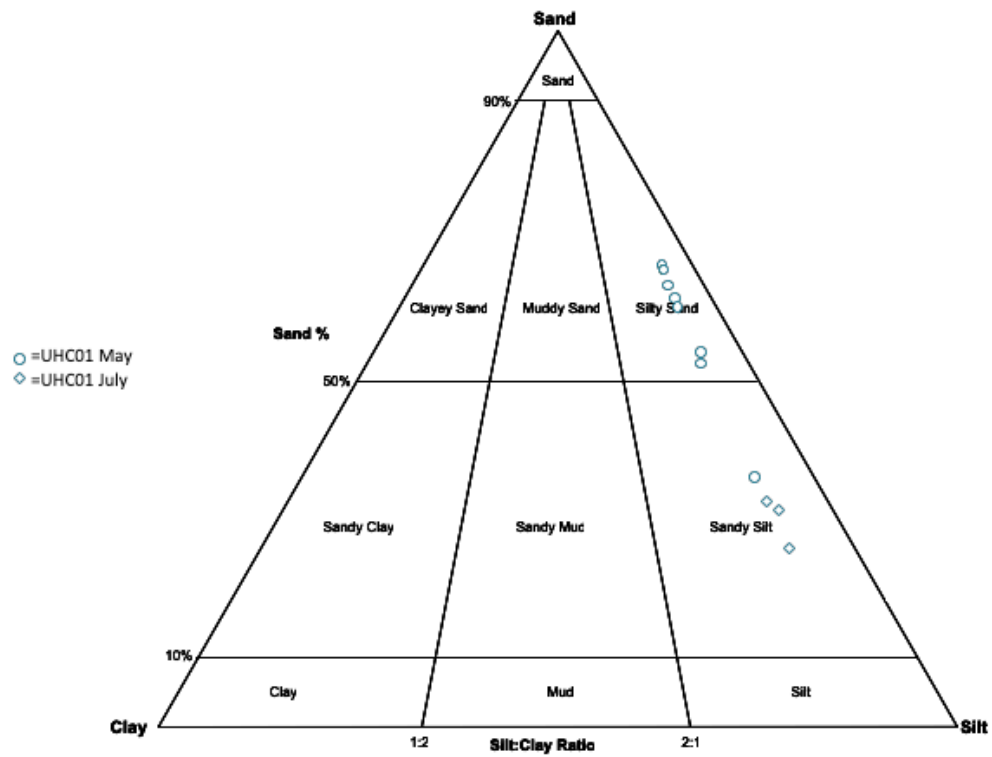
PPCP	CASN ^a	log K _{ow} ^b	log K _{ow} ^c
Amitriptyline	50-48-6	4.92	4.95
Atorvastatin	134523-00-5	na ^d	6.36
Azithromycin	83905-01-5	4.02	3.24
Caffeine	58-08-2	-0.07	0.16
Carbamazepine	298-46-4	2.45	2.25
10,11-Carbamazepine epoxide	36507-30-9	na	0.95
Celecoxib	169590-42-5	na	3.47
Desvenlafaxine	93413-62-8	na	2.72
Dextromethorphan	6700-34-1	na	3.60
Diazepam	439-14-5	2.82	2.70
Diclofenac	15307-79-6	4.51	4.02
Diltiazem	33286-22-5	2.70	2.80
Diphenhydramine	147-24-0	na	1.59
Escitalopram	128196-01-0	na	3.74
Fentanyl	437-38-7	4.05	3.89
Fexofenadine	83799-24-0	na	2.43
Fluoxetine	56296-78-7	3.82	4.65
Gabapentin	60142-96-3	-1.10	-1.37
Hydrochlorothiazide	58-93-5	-0.07	-0.10
Methadone	76-99-3	3.93	4.17
Metoprolol	56392-17-7	1.88	1.69
DEET	134-62-3	2.18	2.26
Nortriptyline	72-69-5	4.51	4.74
Paroxetine	061869-08-7	na	3.95
PFOA	335-67-1	na	4.81
Propranolol	318-98-9	na	0.74
Propoxyphene	469-62-5	4.18	5.27
Rantidine	66357-59-3	na	-1.22
Sertraline	79559-97-0	na	2.18
Sulfamethoxazole	723-46-6	na	0.48
Temazepam	846-50-4	na	2.15
Tramadol	27203-95-2	2.51	3.01
Triamterene	396-01-0	0.98	0.80
Triclocarban	101-20-2	na	4.90
Trimethoprim	738-70-5	0.91	0.73
Venlafaxine	093413-69-5	na	3.28
Verapamil	52-53-9	3.79	4.80
Warfarin	81-81-2	2.70	2.33

^aCAS registry number; ^bexperimental value; ^cpredicted value using Kowwin (EPI Suite); ^dnot available.

Appendix E. Five sand-silt-clay ternary diagrams synthesizing sediment texture for each sampling station for the months of May, July, and September 2018. Diagrams are presented in order of stations AR02, AR03, AR04, AR10 and UHC01.







Appendix F. Grain size statistics (mean and percent sand, silt, and clay) for AR02 for May, July, and September 2018.

			Sub Sample 1						Sub Sample 2						Sub Sample 3						May Avg.	Avg.	
			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6			
May	HC-0518-AR02-R01	Mean(μm)	25.68	25.20	25.02	25.24	24.33	24.25	26.39	26.06	26.11	26.09	25.83	25.86	26.06	26.06	26.11	26.09	25.83	25.86	25.67	32.47	
		Sand:	31.1%	29.7%	29.4%	29.6%	28.6%	28.7%	31.6%	31.0%	31.0%	30.9%	30.6%	30.6%	31.0%	31.0%	31.0%	30.9%	30.6%	30.6%	30.4%		
		Silt:	61.6%	63.1%	63.5%	63.4%	64.4%	64.2%	61.8%	62.3%	62.3%	62.4%	62.6%	62.7%	62.3%	62.3%	62.3%	62.4%	62.6%	62.7%	62.7%		
		Clay:	7.2%	7.2%	7.1%	7.0%	7.1%	7.1%	6.6%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.7%	6.8%		
	HC-0518-AR02-R02	Mean(μm)	31.07	30.19	30.16	30.12	28.33	30.14	32.35	30.50	29.40	31.86	31.40	31.82	30.21	29.89	29.44	29.36	28.01	29.30	30.20		36.3%
		Sand:	37.3%	36.0%	36.0%	35.9%	34.2%	35.9%	36.7%	34.7%	33.4%	35.7%	35.2%	35.4%	35.2%	34.6%	33.9%	33.8%	32.7%	33.8%	35.0%		
		Silt:	56.0%	57.2%	57.2%	57.4%	58.8%	57.2%	57.8%	59.6%	60.8%	58.7%	59.1%	59.1%	58.6%	59.2%	59.7%	59.8%	60.8%	59.9%	58.7%		
		Clay:	6.7%	6.8%	6.9%	6.8%	7.1%	6.9%	5.5%	5.7%	5.8%	5.6%	5.6%	5.6%	6.2%	6.2%	6.3%	6.3%	6.6%	6.4%	6.3%		
	HC-0518-AR02-R03	Mean(μm)	27.17	26.04	25.84	26.00	26.18	26.12	42.59	42.50	42.35	42.20	41.86	42.64	57.65	55.00	56.70	55.89	55.67	55.47	41.55		64%
		Sand:	32.7%	30.7%	30.3%	30.5%	30.6%	30.9%	45.1%	44.7%	44.6%	44.5%	44.3%	45.1%	55.9%	54.4%	55.1%	54.7%	54.7%	54.6%	43.5%		
		Silt:	60.3%	62.2%	62.5%	62.3%	62.2%	61.9%	49.7%	50.0%	50.1%	50.2%	50.3%	49.5%	38.7%	40.0%	39.4%	39.7%	39.8%	39.8%	50.5%		
		Clay:	7.0%	7.2%	7.2%	7.2%	7.2%	7.2%	5.3%	5.3%	5.3%	5.3%	5.4%	5.3%	5.5%	5.6%	5.6%	5.6%	5.5%	5.6%	6.0%		
July	HC-0718-AR02	Mean(μm)	16.93	16.93	17.42	16.88	16.84	16.90	16.19	15.96	15.75	15.74	15.65	15.71	18.39	17.95	18.42	17.85	17.83	17.92	16.96		
		Sand:	16.4%	16.4%	17.6%	16.3%	16.1%	16.3%	16.8%	16.3%	15.9%	15.9%	15.7%	15.8%	19.6%	18.7%	19.4%	18.5%	18.5%	18.7%	17.2%		
		Silt:	74.9%	75.0%	73.8%	75.1%	75.2%	75.1%	74.0%	74.5%	74.7%	74.7%	74.9%	74.8%	72.3%	73.1%	72.5%	73.2%	73.2%	73.1%	74.1%		
		Clay:	8.6%	8.6%	8.6%	8.6%	8.6%	8.6%	9.1%	9.2%	9.4%	9.4%	9.4%	9.4%	8.2%	8.3%	8.1%	8.3%	8.3%	8.3%	8.3%		
Sept.	HC-0918-AR02	Mean(μm)	23.05	22.73	22.80	23.09	22.76	22.84	25.37	24.92	25.03	24.92	24.94	25.01	32.67	31.98	31.64	31.89	31.17	32.61	26.63		
		Sand:	27.7%	27.1%	27.2%	27.6%	27.1%	27.2%	31.6%	30.6%	30.7%	30.5%	30.5%	30.6%	39.9%	38.7%	38.3%	38.5%	38.1%	39.7%	32.3%		
		Silt:	64.2%	64.7%	64.7%	64.3%	64.8%	64.6%	60.5%	61.5%	61.4%	61.6%	61.6%	61.6%	53.2%	54.4%	54.7%	54.5%	54.9%	53.3%	60.0%		
		Clay:	8.1%	8.1%	8.1%	8.1%	8.1%	8.1%	7.9%	7.9%	7.9%	7.9%	7.9%	7.9%	6.8%	7.0%	7.0%	7.0%	7.1%	6.9%	7.3%		
Site Avg,	Mean(μm)																				25.36		
	Sand:																				28.6%		
	Silt:																				63.8%		
	Clay:																				7.3%		

Appendix G. Grain size statistics (mean and percent sand, silt, and clay) for AR03 for May, July, and September 2018.

			Sub Sample 1						Sub Sample 2						Sub Sample 3							
			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	May Avg.	Avg.
May	HC-0518-AR03-R01	Mean	15.94	15.64	15.51	15.73	15.60	15.69	16.15	15.16	14.84	14.80	14.68	14.78	15.63	14.72	14.47	14.39	14.30	14.47	15.14	
		Sand:	15.6%	15.0%	14.7%	15.4%	14.9%	15.1%	17.3%	14.8%	14.2%	14.0%	13.7%	14.0%	16.0%	13.9%	13.3%	13.1%	13.0%	13.3%	14.5%	
		Silt:	75.0%	75.5%	75.7%	75.0%	75.7%	75.4%	73.0%	75.1%	75.7%	75.8%	76.1%	75.8%	74.0%	75.9%	76.4%	76.5%	76.7%	76.5%	75.6%	
		Clay:	9.4%	9.5%	9.6%	9.5%	9.4%	9.5%	9.8%	10.0%	10.1%	10.1%	10.2%	10.2%	9.9%	10.2%	10.3%	10.3%	10.4%	10.2%	9.9%	
	HC-0518-AR03-R02	Mean(μm)	13.60	13.20	13.17	13.00	12.76	12.74	14.29	13.78	13.58	13.13	13.14	13.13	13.80	13.26	12.79	12.72	12.68	12.63	13.19	13.59
		Sand:	12.5%	11.4%	11.3%	10.9%	10.1%	10.1%	14.2%	13.1%	12.6%	11.2%	11.3%	11.2%	13.5%	12.1%	10.7%	10.6%	10.6%	10.4%	11.6%	12.3%
		Silt:	76.5%	77.5%	77.5%	77.9%	78.6%	78.7%	74.8%	75.8%	76.2%	77.4%	77.3%	77.4%	75.4%	76.5%	77.8%	77.9%	77.9%	78.0%	77.2%	76.6%
		Clay:	11.0%	11.1%	11.2%	11.2%	11.2%	11.2%	10.9%	11.1%	11.3%	11.4%	11.4%	11.4%	11.1%	11.3%	11.5%	11.5%	11.5%	11.5%	11.3%	11.1%
	HC-0518-AR03-R03	Mean(μm)	12.90	12.02	11.94	11.71	11.63	11.67	13.54	12.62	11.86	12.03	12.31	11.72	14.42	13.54	12.80	12.53	12.59	12.28	12.45	
		Sand:	12.2%	9.5%	9.4%	8.6%	8.4%	8.6%	14.5%	12.2%	9.9%	10.5%	11.4%	9.4%	15.8%	13.5%	11.4%	10.7%	11.0%	10.1%	10.9%	
		Silt:	76.0%	78.3%	78.4%	79.1%	79.2%	79.0%	73.6%	75.4%	77.5%	76.9%	76.1%	77.9%	73.3%	75.3%	77.1%	77.7%	77.3%	78.1%	77.0%	
		Clay:	11.8%	12.2%	12.2%	12.3%	12.4%	12.4%	11.9%	12.4%	12.7%	12.6%	12.5%	12.7%	10.9%	11.3%	11.5%	11.6%	11.7%	11.8%	12.0%	
July	HC-0718-AR03	Mean(μm)	12.72	12.11	12.37	12.05	12.02	11.97	12.19	11.90	11.48	11.45	11.53	11.46	11.59	11.21	11.09	11.16	11.58	11.09		11.72
		Sand:	11.6%	9.7%	10.6%	9.5%	9.4%	9.2%	12.5%	11.3%	10.4%	10.5%	10.5%	10.8%	11.1%	9.9%	9.7%	9.9%	11.4%	9.7%		10.4%
		Silt:	76.9%	78.7%	77.8%	78.9%	78.9%	79.0%	74.8%	76.1%	76.5%	76.5%	76.5%	76.2%	75.5%	76.4%	76.5%	76.3%	75.0%	76.5%		76.8%
		Clay:	11.5%	11.7%	11.6%	11.7%	11.7%	11.7%	12.7%	12.6%	13.0%	13.0%	13.1%	13.0%	13.5%	13.7%	13.8%	13.7%	13.6%	13.8%		12.1%
Sept.	HC-0918-AR03	Mean(μm)	15.01	14.57	14.79	14.62	14.75	14.64	14.21	13.66	13.63	13.61	13.67	13.64	14.31	13.61	13.12	13.15	13.46	13.53		14.00
		Sand:	17.1%	16.1%	16.6%	16.2%	16.5%	16.2%	14.3%	13.1%	13.0%	13.0%	13.1%	13.0%	16.7%	15.4%	14.1%	14.2%	14.7%	15.3%		14.9%
		Silt:	71.8%	72.6%	72.2%	72.7%	72.4%	72.7%	74.7%	75.6%	75.7%	75.8%	75.6%	75.7%	71.5%	72.2%	73.4%	73.3%	73.1%	72.3%		73.5%
		Clay:	11.1%	11.2%	11.1%	11.2%	11.1%	11.1%	10.9%	11.3%	11.3%	11.3%	11.3%	11.3%	11.9%	12.4%	12.5%	12.5%	12.2%	12.4%		11.6%
	Site Avg.	Mean(μm)																				13.10
Sand:																					12.6%	
Silt:																					75.6%	
Clay:																					11.6%	

Appendix H. Grain size statistics (mean and percent sand, silt, and clay) for AR04 for May, July, and September 2018.

			Sub Sample 1						Sub Sample 2						Sub Sample 3						May Avg.	Avg.	
			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6			
May	HC-0518-AR04-R01	Mean(μm)	13.81	12.59	12.65	12.44	12.63	12.52	13.87	13.07	12.77	13.14	12.76	12.98	14.94	13.96	13.26	13.10	13.51	13.20	13.18		
		Sand:	17.2%	14.2%	14.6%	13.9%	14.4%	13.7%	17.4%	15.5%	14.7%	15.9%	14.8%	15.2%	19.6%	17.3%	15.6%	15.3%	16.1%	15.6%	15.6%		
		Silt:	69.9%	72.3%	72.1%	72.6%	72.1%	73.0%	69.5%	71.0%	71.6%	70.6%	71.5%	71.5%	67.9%	69.8%	71.2%	71.5%	71.1%	71.2%	71.1%		
		Clay:	12.9%	13.4%	13.4%	13.5%	13.4%	13.2%	13.1%	13.5%	13.6%	13.5%	13.7%	13.4%	12.5%	12.9%	13.2%	13.3%	12.9%	13.2%	13.3%		
	HC-0518-AR04-R02	Mean(μm)	14.24	13.11	12.85	12.76	12.78	12.68	15.98	14.24	14.19	13.47	13.38	13.25	13.45	13.02	12.43	12.34	12.59	12.14	13.27		13.22
		Sand:	17.3%	14.3%	13.8%	13.6%	13.7%	13.4%	21.2%	17.5%	17.7%	15.8%	15.6%	15.4%	15.8%	14.7%	13.1%	13.0%	13.9%	12.6%	15.1%		15.7%
		Silt:	70.5%	73.0%	73.4%	73.5%	73.4%	73.7%	67.2%	70.0%	69.8%	71.3%	71.5%	71.7%	71.7%	72.5%	73.9%	74.0%	73.2%	74.3%	72.1%		71.1%
		Clay:	12.3%	12.7%	12.8%	12.9%	12.9%	12.9%	11.6%	12.5%	12.5%	12.9%	12.9%	12.9%	12.5%	12.7%	13.0%	13.0%	13.0%	13.1%	12.7%		13.2%
	HC-0518-AR04-R03	Mean(μm)	14.57	12.99	12.75	12.58	12.67	12.70	14.46	13.41	12.49	12.49	12.20	12.02	15.61	14.09	13.39	13.15	12.99	13.22	13.21		
		Sand:	20.0%	16.4%	15.9%	15.4%	15.4%	15.6%	19.9%	17.0%	15.5%	15.2%	14.9%	14.4%	20.4%	17.2%	15.4%	14.8%	14.5%	15.1%	16.3%		
		Silt:	66.6%	69.5%	69.9%	70.3%	70.5%	70.3%	66.3%	68.8%	69.8%	70.3%	70.2%	70.7%	67.6%	70.2%	71.7%	72.2%	72.4%	71.8%	70.0%		
		Clay:	13.4%	14.1%	14.2%	14.3%	14.1%	14.1%	13.8%	14.2%	14.6%	14.4%	14.8%	14.9%	12.0%	12.6%	12.9%	13.0%	13.1%	13.0%	13.8%		
July	HC-0718-AR04	Mean(μm)	18.13	17.52	17.50	17.54	17.41	17.45	19.84	19.18	19.07	19.11	19.06	19.43	6.99	6.08	6.29	6.22	5.94	5.92		14.37	
		Sand:	14.8%	13.2%	13.3%	13.3%	13.1%	13.6%	20.7%	19.4%	19.3%	19.4%	19.3%	20.4%	7.1%	4.3%	4.8%	3.9%	3.7%	3.7%		12.6%	
		Silt:	76.5%	77.9%	77.8%	77.7%	77.9%	77.4%	69.6%	70.6%	70.8%	70.6%	70.7%	69.6%	73.4%	75.0%	74.7%	75.6%	75.4%	75.3%		74.3%	
		Clay:	8.8%	8.9%	8.9%	8.9%	9.0%	8.9%	9.8%	10.0%	10.0%	10.0%	10.0%	10.0%	19.5%	20.7%	20.5%	20.5%	20.9%	21.0%		12.4%	
Sept.	HC-0918-AR04	Mean(μm)	23.36	23.34	23.37	23.43	22.95	23.05	17.01	18.11	16.01	16.12	16.72	15.70	31.09	30.45	30.52	30.38	30.69	30.64		23.50	
		Sand:	24.0%	23.5%	23.8%	23.6%	22.8%	23.0%	19.6%	20.9%	17.4%	17.9%	18.7%	16.8%	34.4%	33.7%	33.9%	33.7%	34.4%	33.7%		25.3%	
		Silt:	67.7%	68.3%	67.9%	68.2%	68.9%	68.7%	70.1%	69.1%	72.2%	71.7%	71.1%	72.6%	58.6%	59.2%	59.1%	59.2%	58.4%	59.1%		66.1%	
		Clay:	8.3%	8.2%	8.3%	8.2%	8.4%	8.4%	10.2%	10.0%	10.5%	10.4%	10.2%	10.6%	7.0%	7.2%	7.1%	7.1%	7.2%	7.2%		8.6%	
	Site Avg.	Mean(μm)																				17.03	
		Sand:																				17.9%	
		Silt:																				70.5%	
		Clay:																				11.4%	

Appendix I. Grain size statistics (mean and percent sand, silt, and clay) for AR10 for May, July, and September 2018.

			Sub Sample 1						Sub Sample 2						Sub Sample 3							
			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	May Avg.	Avg.
May	HC-0518-AR10-R01	Mean(μm)	28.56	27.65	27.59	27.67	27.45	27.58	27.54	26.48	26.35	26.09	25.92	26.04	27.46	26.22	25.83	25.65	25.35	25.47	26.72	
		Sand:	38.7%	37.5%	37.5%	37.9%	37.3%	37.4%	37.1%	35.7%	35.8%	35.2%	35.0%	35.1%	35.2%	33.5%	32.9%	32.6%	32.7%	32.3%	35.5%	
		Silt:	52.9%	53.9%	53.9%	53.6%	54.1%	54.1%	54.5%	55.7%	55.6%	56.1%	56.2%	56.2%	57.4%	58.9%	59.4%	59.6%	59.6%	59.9%	56.2%	
		Clay:	8.4%	8.6%	8.6%	8.5%	8.6%	8.5%	8.4%	8.6%	8.6%	8.7%	8.7%	8.7%	7.4%	7.6%	7.7%	7.8%	7.8%	7.8%	8.3%	
	HC-0518-AR10-R02	Mean(μm)	22.98	22.85	22.20	22.24	22.32	22.06	23.55	22.32	22.31	21.84	22.18	21.69	22.94	21.90	21.42	21.29	21.27	21.25	22.14	24.45
		Sand:	28.6%	28.2%	27.4%	27.7%	27.5%	27.2%	29.7%	27.4%	27.3%	26.6%	27.4%	26.5%	28.6%	26.9%	26.1%	25.9%	25.9%	25.9%	27.3%	31.9%
		Silt:	62.8%	63.2%	63.8%	63.5%	63.9%	64.0%	62.0%	64.0%	64.2%	64.7%	63.9%	64.7%	62.9%	64.3%	64.9%	65.1%	65.1%	65.1%	64.0%	59.4%
		Clay:	8.5%	8.6%	8.7%	8.8%	8.6%	8.8%	8.3%	8.6%	8.5%	8.7%	8.7%	8.8%	8.5%	8.8%	8.9%	9.0%	9.0%	9.0%	8.7%	8.7%
	HC-0518-AR10-R03	Mean(μm)	23.22	23.21	22.57	22.53	22.54	22.45	26.95	26.95	25.78	25.67	26.06	26.06	25.53	24.43	24.42	24.39	23.92	24.06	24.49	
		Sand:	31.5%	31.6%	30.4%	30.4%	30.4%	30.3%	36.4%	36.4%	34.9%	34.8%	35.7%	35.7%	34.3%	32.2%	32.1%	32.1%	31.5%	31.6%	32.9%	
		Silt:	59.1%	59.0%	60.0%	60.1%	60.0%	60.2%	55.0%	55.0%	56.3%	56.3%	55.5%	55.5%	56.9%	58.7%	58.9%	58.9%	59.2%	59.3%	58.0%	
		Clay:	9.4%	9.4%	9.6%	9.5%	9.6%	9.6%	8.6%	8.6%	8.8%	8.9%	8.8%	8.8%	8.8%	9.1%	9.0%	9.0%	9.2%	9.0%	9.1%	
July	HC-0718-AR10	Mean(μm)	14.95	14.55	14.47	14.53	14.58	14.45	13.39	12.86	12.71	12.69	12.63	12.63	15.00	14.55	14.14	14.06	14.04	13.98		13.90
		Sand:	18.1%	17.2%	17.1%	17.2%	17.3%	17.0%	14.4%	13.1%	12.8%	12.7%	12.5%	12.5%	15.1%	14.3%	13.1%	12.9%	12.9%	12.7%		14.6%
		Silt:	70.4%	71.1%	71.2%	71.1%	71.0%	71.2%	73.6%	74.6%	74.9%	75.0%	75.1%	75.2%	74.3%	74.9%	75.9%	76.1%	76.2%	76.3%		73.8%
		Clay:	11.5%	11.7%	11.7%	11.7%	11.7%	11.7%	12.0%	12.3%	12.3%	12.3%	12.3%	12.3%	10.6%	10.8%	10.9%	11.0%	11.0%	11.0%		11.0%
Sept.	HC-0918-AR10	Mean(μm)	21.44	21.47	21.73	21.44	21.90	21.37	23.00	22.73	22.48	22.70	22.26	22.01	15.20	15.06	14.70	14.69	14.87	14.60		19.65
		Sand:	26.8%	27.2%	27.6%	26.9%	27.9%	26.7%	27.6%	27.7%	26.9%	27.3%	26.7%	26.3%	17.2%	16.8%	16.1%	16.3%	16.6%	16.3%		23.6%
		Silt:	63.3%	63.0%	62.6%	63.2%	62.3%	63.4%	62.9%	62.9%	63.5%	63.1%	63.6%	64.0%	72.2%	72.6%	73.1%	72.8%	72.5%	72.7%		66.3%
		Clay:	9.8%	9.8%	9.8%	9.9%	9.8%	9.9%	9.5%	9.5%	9.6%	9.6%	9.7%	9.7%	10.6%	10.6%	10.7%	10.9%	10.8%	11.0%		10.1%
Site Avg.	Site Avg.	Mean(μm)																				19.33
		Sand:																				23.4%
		Silt:																				66.5%
		Clay:																				9.9%

Appendix J. Grain size statistics (mean and percent sand, silt, and clay) for UHC01 for May and July 2018.

			Sub Sample 1						Sub Sample 2						Sub Sample 3						May Avg.	Avg.
			Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6		
May	HC-0518-UHC01-R01	Mean(μm)	93.84	94.39	91.16	92.83	91.95	92.62	94.29	93.80	94.44	94.64	94.50	93.14	78.99	80.58	79.38	80.39	80.49	79.86	88.96	
		Sand:	67.7%	67.8%	67.0%	67.3%	67.1%	67.3%	68.5%	68.1%	68.2%	68.2%	68.2%	67.8%	62.2%	62.5%	61.9%	62.1%	62.0%	62.2%	65.9%	
		Silt:	28.2%	28.2%	28.9%	28.7%	28.8%	28.6%	27.5%	27.8%	27.7%	27.6%	27.7%	28.0%	32.9%	32.7%	33.2%	33.0%	33.0%	32.9%	29.7%	
		Clay:	4.0%	4.1%	4.1%	4.0%	4.1%	4.1%	4.0%	4.1%	4.1%	4.1%	4.1%	4.1%	4.9%	4.9%	4.9%	4.9%	4.9%	4.9%	4.4%	
	HC-0518-AR10-R02	Mean(μm)	84.98	84.11	84.52	85.49	84.79	84.64	94.34	94.99	94.94	94.28	95.05	95.16	84.50	84.32	85.15	84.81	84.60	84.61	88.07	75.33
		Sand:	64.4%	63.9%	63.9%	64.2%	63.9%	63.8%	67.7%	67.6%	67.5%	67.4%	67.6%	67.6%	65.6%	65.2%	65.4%	65.3%	65.1%	65.3%	65.6%	60.1%
		Silt:	31.3%	31.6%	31.7%	31.5%	31.7%	31.8%	28.3%	28.5%	28.5%	28.7%	28.5%	28.5%	29.9%	30.3%	30.0%	30.2%	30.3%	30.2%	30.1%	34.9%
		Clay:	4.4%	4.5%	4.4%	4.3%	4.4%	4.4%	4.0%	4.0%	4.0%	4.0%	3.9%	3.9%	4.5%	4.5%	4.5%	4.5%	4.5%	4.5%	4.3%	5.0%
	HC-0518-AR10-R03	Mean(μm)	59.98	58.61	61.31	60.72	60.44	62.10	54.70	53.59	54.01	54.70	53.31	53.54	33.46	32.07	31.85	31.95	32.19	32.66	48.96	
		Sand:	55.4%	54.9%	55.8%	55.6%	55.5%	56.0%	54.2%	53.6%	53.9%	54.3%	53.5%	53.8%	38.6%	36.9%	36.7%	36.7%	37.1%	37.9%	48.9%	
		Silt:	39.3%	39.8%	39.0%	39.1%	39.2%	38.9%	39.7%	40.2%	39.9%	39.6%	40.3%	40.0%	53.9%	55.4%	55.6%	55.6%	55.2%	54.5%	44.7%	
		Clay:	5.3%	5.3%	5.2%	5.2%	5.3%	5.1%	6.1%	6.2%	6.2%	6.2%	6.2%	6.1%	7.5%	7.7%	7.7%	7.7%	7.7%	7.6%	6.4%	
July	HC-0718-AR10	Mean(μm)	27.24	26.59	26.53	27.36	27.27	26.44	27.25	26.33	25.75	25.65	25.66	26.43	22.09	21.48	21.51	21.50	21.41	21.79		24.90
		Sand:	33.4%	32.6%	32.5%	32.5%	32.4%	32.4%	35.2%	34.0%	33.3%	33.2%	33.2%	34.0%	27.8%	26.8%	26.9%	26.9%	26.7%	27.3%		31.2%
		Silt:	59.8%	60.6%	60.5%	60.6%	60.7%	60.7%	57.3%	58.3%	58.9%	58.9%	58.9%	58.3%	63.9%	64.8%	64.7%	64.7%	64.9%	64.5%		61.2%
		Clay:	6.8%	6.8%	7.0%	6.9%	6.9%	7.0%	7.5%	7.7%	7.8%	7.9%	7.8%	7.7%	8.3%	8.4%	8.4%	8.4%	8.4%	8.2%		7.3%
	Site Avg.	Mean(μm)																				50.12
		Sand:																				45.7%
		Silt:																				48.0%
		Clay:																				6.1%