Hudson River Foundation Final Report:
Currently and Recently-Used Persistent Bioaccumulative Toxic Contaminants in Water and Biota from the Passaic River

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1. Introduction

The extensive production and usage of persistent organic pollutants has led to widespread contamination of the environment with serious consequences for aquatic ecosystems. The lower Passaic River is a highly industrialized tributary of the Hudson River system that has been subjected to heavy industrialization and urbanization, resulting in the severe degradation of local aquatic life. Previous studies have found elevated concentrations of polychlorinated dibenzo-p- dioxins and furans (PCDD/Fs) and polychlorinated biphenyls (PCBs) in sediment, water, and biota.[1,2] Bioaccumulation and biomagnification of persistent, hydrophobic contaminants can lead to elevated levels in upper trophic levels, threatening the health of wildlife as well as humans who consume fish from the region.

While a number of studies have contributed valuable data on the occurrence of legacy pollutants in the Passaic River, there is a paucity of information about currently- and recently-manufactured emerging contaminants in the lower Passaic. It is crucial that we enhance our understanding of new pollutants that are currently being emitted to this fragile ecosystem. Even after remediation of historical pollution, emerging contaminants may be continued stressors to wildlife. They may also exacerbate the effects of other stressors in this degraded environment via chemosensitization or synergism.

Here, extracts from biota collected on the Passaic River were retrospectively analyzed for two groups of currently used organic contaminants, the polycyclic musks (PCMs) and novel halogenated flame retardants (NHFRs). Extracts from PEs deployed in surface waters of the river during the same time period were also analyzed. The primary objectives of this project were to: (i) provide baseline concentrations of PCMs and NHFRs in river water and biota from the lower Passaic River, (ii) calculate lipid-normalized bioaccumulation factors (BAFs) for these compounds, (iii) measure trophic magnification factors (TMFs).

2. Methods

2.1. Sample Collection and Preparation

Sampling of biota and passive sampler deployments was conducted by a team from the Lohmann Laboratory from 2011 to 2012. Sites of PE deployment are marked in Figure 1 as PE-1, PE-2, and PE-3, and details of the sampling campaign are listed in Table S1. Biota samples were collected during several sampling campaigns during August-November, 2011 at three sites along the Passaic River: an upriver site above the Dundee Dam (Station A), a tidal freshwater site at river kilometer 16.1 (Station B), and a brackish site near the NYNJ Port Authority Rail Maintenance Yard (river km 7.4;

Station C), near Newark Bay. Sampling sites are marked in Figure 1 and details of biota samples are listed in Table S2. More details on collection and preparation of these samples can be found in Khairy et al. 2014.[3]

2.1.1 Biota Collection and Preparation

Biota were collected using minnow traps, eel pots, crab traps, seines, and otter trawls. As most fish species are quite mobile, little difference was expected between specimens collected at the two downriver locations. Most fish collection was restricted to young-of-year individuals to avoid confounding factors such as movement in and out of the estuary and age-related differences in exposure. However, *Morone saxatilis* were 1 and 2-year-old juveniles, which may be more mobile. Because fish were generally quite small, specimens were pooled for chemical analysis. For some species, different lengths of organisms were divided into subgroups and analyzed separately, as noted in Table S2.

Biota samples were frozen until analysis at -20°C. Samples were then thawed and skinned, and tissue was pooled and homogenized in a blender with the head, bones, and gut removed. Some species (*lepomis macrochirus, hybognathus regius, fundulus diaphanus, menidia menidia,* and *fundulus heteroclitus*) were too small to be skinned and so skin was included in the homogenate. Composited homogenized samples were stored in amber glass jars at -20°C until extraction.

Biota extracts retrospectively analyzed for PCMs were originally prepared and extracted for analysis of PAHs, as detailed by Khairy et al.[3] Briefly, tissues were prepared based on a procedure described by Wretling et al.[4] in which tissues were saponified with aqueous methanolic potassium hydroxide and shaken for one hour and then transferred to a separatory funnel with deionized water and cyclohexane. After vigorous shaking, the organic layer was removed and the liquid-liquid extraction was repeated two more times. The organic layers were combined and dried over anhydrous sodium sulfate to remove water, and were then concentrated to 1 mL. Further cleanup was performed using muffled sea sand, deactivated neutral alumina, deactivated silica gel, and anhydrous sodium sulfate. The extract was eluted with 1:1 hexane:DCM, concentrated to 50 µL, and p-terphenyl-d_{14} was added as an injection standard before analysis.

Samples retrospectively analyzed for NHFRs were originally prepared for analysis of PBDEs and PCBs. These samples were loaded into precleaned thimbles and Soxhlet extracted with 1:1 hexane:acetone for 48 h. Extracts were then concentrated and dried over anhydrous sodium sulfate. An aliquot of each extract was removed and evaporated in a preweighed aluminum dish to determine lipid content gravimetrically. Extracts were then further concentrated and lipids were removed using a multilayer silica column and eluted with 70:30 hexane:DCM.

Data from stable isotope analysis ($\delta^{13}C$ and $\delta^{15}N$) are also included in Table S2. For this analysis, tissue subsamples were freeze-dried to a constant weight, ground to a fine powder, and stored in precombusted glass vials at -20°C. Samples were analyzed on a Micromass Isochrom Continuous Flow Stable Isotope Mass Spectrometer with a Carla Erba Elemental Analyzer (CHNS-O EA1 108).

2.1.2 Passive Sampler Deployment and Extraction

Passive samplers were retrospectively analyzed for PCMs and NHFRs. PEs had been extracted twice for 24 h each, first with dichloromethane then with hexane and were concentrated under nitrogen. Extracts were passed through an activated carbon column (0.8 cm i.d. filled with 2 cm of activated carbon/celite mixture) and eluted with 5 mL of DCM/cyclohexane (1:1, v:v).

Sampling rates, which were determined from loss of performance reference compounds (PRCs) over deployment, were calculated in Khairy et al.[5] and were used here to calculate ambient water concentrations from PE concentrations by first using Equation 1 to determine the percent equilibration (*f*) reached by each target compound during deployment, where K_{PEW} is the PE-water partitioning coefficient, *t* is the length of the deployment time, V_{PE} is the volume of the PE sampler determined from its mass and known density (0.91 kg/L), and R_s is the best fit value for the *in situ* sampling rate. Equation 2 was then used to convert concentration in the PE to a dissolved water concentration, where C_{PE} is the concentration in the polyethylene and C_w is the truly dissolved concentration in the surrounding water. K_{PEW} values used to derive truly dissolved concentrations for PCMs were calculated from aqueous solubility, and for NHFRs were calculated from octanol-water partitioning coefficients (K_{OW}). The values used are listed in Table S3.

$$f = 1 - e^{\frac{-R_S \cdot t}{K_{PEW} \cdot V_{PE}}} \qquad \text{Equation 1}$$

$$C_w = \frac{C_{PE}}{K_{PEW} \cdot f} \qquad \text{Equation 2}$$

2.2 GC/MS Analysis

Extracts were analyzed by two separate methods for PCMs and NHFRs, as detailed below. Target compound abbreviations and full names are given in Table S3. Final concentrations for biota samples were calculated using the wet weight converted to lipid weight using the percent lipid content of the tissues. For all PE and biota samples, concentrations were blank-subtracted using the mean of all available laboratory blanks and the detection limit was defined as three times the standard deviation of the lab blanks. Concentrations below the detection limit were replaced with half the detection limit.

2.2.1. Retrospective Analysis of PCMs

Fish extracts were analyzed on an Agilent 6890 GC coupled with an Agilent 5973 MSD in electron ionization select ion monitoring mode (EI SIM) for seven PCMs. More information about these compounds, including full names, abbreviations, and CAS numbers, are available in Table S3. Concentrations were corrected for internal standard recoveries using phenanthrene-d_{10} and chrysene-d_{12} as internal standards. Extracts were quantified using a 9-point calibration curve ranging from 1-500 pg/uL. Extracts from PEs were analyzed on an Agilent 7890 GC coupled with an Agilent 5977 MSD,

again in EI SIM, using the same calibration curve and internal standards as the fish samples.

All curves displayed satisfactory linearity with $r^2 > 0.995$. Calibration checks were analyzed after every 10 samples were within 75-130% of the expected concentration. For both analytical methods, the GCs were equipped with Agilent J&W DB-5 columns (30 m x 250 µm x 0.25 µm). 1 µL extracts were injected via splitless injection at 280°C. Helium gas flow was maintained at 1.5 mL/min. The oven program began at 90°C, was held for 3 minutes, then ramped to 100 °C at 6 °C/min, held for 2 minutes, then ramped to 320°C at 8 °C/min, and finally held for 6 minutes. The transfer line was held at 250°C. The EI source was held at 230°C with quadrupole at 150°C. Ions monitored are listed in the SI (Table S4).

2.2.2. Retrospective Analysis of NHFRs

1 µL of extract was injected via splitless injection at 280°C onto an Agilent 7890 GC equipped with an Agilent J&W DB-5 column (30 m x 250 µm x 0.25 µm) coupled to an Agilent 5977 MSD in negative chemical ionization (NCI) mode with methane reagent gas to quantify NHFRs. More information about the target compounds, including full names, abbreviations, and CAS numbers, is again available in Table S3. Helium gas flow was maintained at 1 mL/min. The oven temperature program began at 90°C, was held for 2 minutes, then ramped to 225°C at 8°C/min, held for another 2 minutes, ramped to 260°C by 4°C/min, then to 320°C at 8°C/min, and held for 8 minutes. The transfer line was held at 280 °C and the CI source and quadrupole were held at 150°C.

BDEs 35, 77, and128 were used as injection standards. Concentrations were not corrected for recovery, as no appropriate internal standards had been spiked prior to extraction for this retrospective analysis. Ions monitored are listed in the SI (Table S4). Calibration checks run every 10 samples were within 80 – 140% of expected values for all NHFR target compounds.

2.2.3. Calculation of Bioaccumulation Factors (BAFs)

BAFs were calculated using both lipid weight and wet weight concentrations from biota to facilitate comparison with other studies, which have used both types of data. Biota concentrations (pg/kg) were divided by PE-derived surface water concentrations (pg/L) for the nearest PE deployment site, so that Site A was paired with PE-1, Site B with PE-2, and Site C with PE-3 (see Figure 1).

3. Results and Discussion

3.1. PCMs in Biota from the Lower Passaic River

Concentrations of total PCMs (Σ_7PCM) in biota ranged from 60 ng/g lipid weight (ng/g lw) in large American eel (110 cm) from Station B to 10484 ng/g lw in Eastern silvery minnow, also from Station B. HHCB and AHTN were detected above detection limits in all samples. The remainder of polycyclic musks (ADBI, AHMI, and ATII) were detected in >65% of samples while the nitromusks (MUK and MUX) had lower frequency of detection (25% and 35%, respectively).

Σ_7PCM was dominated by HHCB and AHTN, which made up >70% of Σ_7PCM for all samples. All concentrations are shown in Table 1. No clear delineation between trophic groups is readily observable, as omnivores had greater concentrations of Σ_7PCM than carnivores at Station A and B, and there was no discernible difference between Σ_7PCM concentrations in carnivores and planktivores at Station C. Samples grouped and averaged based on trophic group and separated by location are displayed in Figure 2, which shows that Station A, which was above Dundee Dam and expected to have lower concentrations of organic contaminants, had similar or greater concentrations of Σ_7PCM than the downriver sites.

Reiner and Kannan measured much lower HHCB concentrations by 2-3 orders of magnitude in biota collected in June, 2006 from the Upper Hudson River.[6] Only the liver was analyzed for most biota in their study, but concentrations were lipid-normalized, facilitating comparison. Concentrations in this study were similar to those measured by O'Toole et al. in Hamilton Harbor, Lake Ontario (141 ng/g ww for gizzard shad; 84 ng/g ww for white perch)[7] and were generally much lower than in biota from the pond of a sewage treatment plan in Germany (4600-160000 ng/g lw).

3.2. NHFRs in Biota from the Lower Passaic River

Concentrations of total NHFRs (Σ_7NHFR) ranged from 1.3 ng/g lw in striped bass from Station C to 47 ng/g lw for gizzard shad from Station C. Elevated concentrations in the gizzard shad sample were dominated by BTBPE. Total concentrations averaged by trophic group are shown in Figure 3, and concentration in all biota samples are summarized in Table 2. PBT was not detected above detection limits in any samples and so is omitted from further discussion. Detection frequency for the other bromobenzenes (pTBX, PBBz, PBEB, and PBEB) ranged from 16% for PBBz to 63% for HBBz, while detection frequency of the larger NHFR molecules (BTBPE, ADP, and SDP) was >90%. Composition of NHFRs varied widely between locations and species.

There was no clear difference in Σ_7NHFR in carnivore species based on location, concentrations in omnivores were greater at downriver sites than the upriver locations. Unexpectedly, concentrations in carnivores were similar or lower than other trophic groups at all locations. Tomy et al. also measured lower concentrations of the DP isomers in higher trophic levels in a study of Lake Ontario and Lake Winnipeg, with greatest lipid-normalized concentrations in mussels and zooplankton.[8] However, there is no evidence of this phenomenon for the other NHFRs in previous work. Samples grouped and averaged based on trophic group and separated by location are displayed in Figure 3. As with PCM concentrations, no clear difference is apparent between above-dam and below-dam biota.

The Dechloranes (*anti* and *syn* isomers summed) ranged from 0.31 ng/g lw in banded killifish from Station B to 21 ng/g lw in American eel (10 cm) from Station B. Total Dechlorane Plus (DP) concentrations in biota from the lower Passaic were similar to total DP concentrations in biota from urban industrial rivers in Korea (0.17 – 30 ng/g lw).[9] Concentrations measured here were greater than those in fish from San Francisco Bay (<1 – 3.7 ng/g lw) and from Lake Winnipeg and Lake Ontario (0.02 – 4.4 ng/g lw).[8,10]

The fraction of *anti*-DP isomer (f_{anti}) in this study was highly variable, ranging from 11% in blue crab to 94% in the American eel (110 cm), and was not always <75% (the f_{anti} of the technical product). This contradicts other studies which have observed that the *syn*-isomer is more bioaccumulative than the *anti*-isomer.[9,11] However, some studies have noted that isomer composition can be distinct in biota from different environments.[8] This may, in part, be due to the interplay of numerous sources of DP, including diffusion from surrounding water, diet, differences in food web dynamics,[11] and sediment ingestion for some species.

Total concentrations of the four bromobenzenes that were detected (pTBX, PBBz, PBEB, and HBBz) ranged from 0.37 ng/g lw in Eastern silvery minnow from Station C to 25 ng/g lw in blue crab from Station B. The concentration of bromobenzenes in blue crab was elevated to about five times greater levels than all other species, which is similar to what was observed by Khairy et al. for PBDEs and OCPs, where it was hypothesized that these elevated concentrations may be due to the species' direct ingestion of sediment (Khairy et al., *in prep.*). Data from previous studies on concentrations of bromobenzenes in biota are sparse. HBBz, PBEB, and BTBPE were not found above detection limits in fish from San Francisco Bay collected in 2007.[10] Concentrations reported here were generally lower than what was measured in studies of biota from aquatic environments impacted by e-waste recycling facilities in China, which ranged from 40-518 ng/g lw for BTBPE, 197-3100 ng/g lw for HBBz, and 4-256 ng/g lw for PBEB.[12]

3.3. PCMs and NHFRs Dissolved in Passaic River Water

3.3.1. Dissolved PCMs in the lower Passaic River

Concentrations of PCMs in river surface water were dominated by HHCB and AHTN, with HHCB making up more than 85% of total PCMs (Σ_7PCM) in all samples. The polycyclic musks (ADBI, AHMI, ATII, HHCB, and AHTN) were found above detection limits in all PE extracts, while frequency of detection for the nitromusks was lower (36% and 54% of samples for MUX and MUK, respectively).

HHCB was greater at all locations during the final sampling period (October to November of 2012), ranging from 90-107 ng/L, compared to the remaining sampling seasons (14-94 ng/L). Composition of PCMs dissolved in water is shown in Figure 4 and total concentrations of AHTN and HHCB derived for each deployment are shown in Figure 5. Concentrations at all sites for all deployments are listed in Table 3.

3.3.2. Dissolved NHFRs in the lower Passaic River

NHFRs were present in the 5-60 pg/L range and were dominated by PBBz, which made up >90% of Σ_7NHFR at downriver sites (Sites 2 and 3) and >40% of Σ_7NHFR at Site 1. The bromobenzenes pTBX, PBT, and HBBz, as well as BTBPE, were found in less than half of PE extracts, while both DP isomers were above detection limits in all samples. Concentrations of dissolved NHFRs at all sites for all deployments are listed in Table 3. Few studies were available for comparison of concentrations for NHFRs dissolved in water. Concentrations of HBBz were similar or lower than what was seen in the Great Lakes (<DL - 0.7 pg/L), while PBBz and PBEB were greater than in the Great Lakes (<DL – 0.5 pg/L and <DL – 32 pg/L, respectively).[13]

Total concentrations of DP ranged from 0.02 pg/L above Dundee Dam during the first deployment period (12/2011-3/2012) – 0.1 pg/L at Site 3 (River km 9.6) during 6-8/2012. Few studies have reported concentrations of DP in the water column for comparison, and no data on dissolved concentrations are available. Concentrations calculated here were lower than those reported in previous active sampling studies of seawater in the Arctic and Antarctic region (0.05 – 4.2 pg/L)[14] and in the Great Lakes (0.1 – 14 pg/L)[13], likely because these concentrations include the particulate and/or colloidal phase, where the majority of the highly hydrophobic DP was expected to be found. Concentrations were also lower than those measured in surface waters of the Songhua River in northeastern China (170 – 550 pg/L).[15] f_{anti} was fairly consistent between locations and deployment periods, with an average of 31±4%, which is lower than the f_{anti} value for the technical product from OxyChem (0.64-0.80),[16] possibly due to changes in composition due to fractionation with other phases.

3.4. Bioaccumulation Factors based on PE-Derived Water Concentrations

Bioaccumulation factors (BAFs) were calculated as a measure of enrichment of the target compounds in biota versus the surrounding river surface waters, with units of [ng target compound/kg biota]/[ng target compound/L water]. BAFs are presented in Table 4 and Table 5 as lipid-normalized and wet weight-based values for PCMs and NHFRs, respectively. The wide range of values for the same species at different sites suggests that location-specific differences in sediment concentrations and other factors may have caused variation that was not captured by using only dissolved water concentrations to calculate BAFs.

While lipid-normalizing BAFs makes them more easily comparable to specimens with variable fat content, most other data available are for wet weight-based values, so here, values are compared to other wet weight-based BAFs from literature. Wet weight-based BAFs for HHCB in biota from the lower Passaic ranged from 19 for American eel (110 cm) to 4853 for Eastern silvery minnow from Station B. The wet weight-based BAF for bluegill was similar to the value calculated by Balk and Ford via a bench-scale study for the same species (1580 in that study, versus 2640 in this study),[17] and were generally similar or lower than those provided by Reiner et al. for biota in the upper Hudson River (18-371) and by Gatermann et al. in a sewage treatment pond in Germany (60-580).[6,18]

3.5. Trophic Magnification Factors based on PE-Derived Water Concentrations

Trophic magnification factors (TMFs) were calculated as in Fisk et al.[19] and are listed in Table S5. A significant relationship between the natural log of tissue concentration and the trophic position was not observed for most target chemicals. This was somewhat expected for PCMs, as they are metabolized to more polar products in many fish species with differing efficiencies.[20] While some studies of DP isomers in food webs have found evidence of trophic magnification, others have seen no significant trend.[8] Here, all compounds showed an absence of significant biomagnification, and the DP isomers, as well as BTBPE, showed some evidence of trophic dilution.

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Figures and Tables

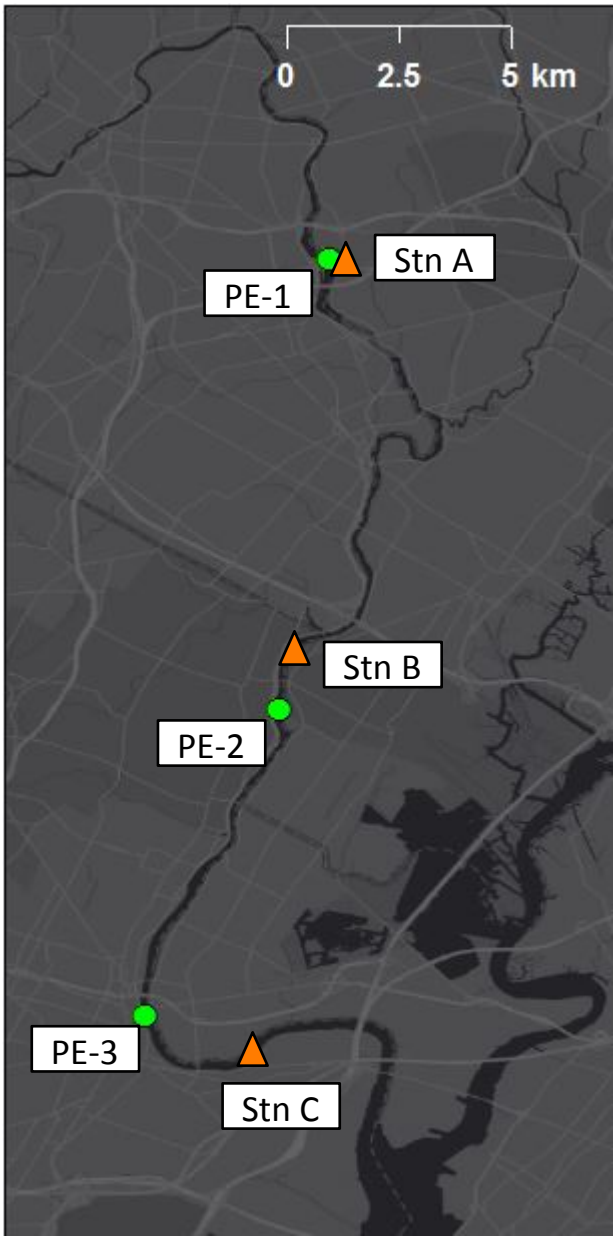

Figure 1. Sampling locations for biota (orange triangles) and PE deployments (green circles) along the Passaic River. Stn A/PE-1 are above Dundee Dam and intended as a reference/control location.

Species	ADBI		AHMI		ATII		HHCB		AHTN		MUX		MUK	
	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww	ng/g lipid	ng/g ww
Station A (Above Dundee Dam)														
Banded killifish	6.88	0.138	13.3	0.267	141	2.82	3288	65.8	359	7.17	3.31	0.066	0.078	0.002
Bluegill	11.5	0.381	9.32	0.308	22.2	0.733	6311	208	448	14.8	0.333	0.011	4.70	0.155
Eastern silvery minnow; benthic	31.8	1.11	28.2	0.988	18.9	0.662	8482	297	448	15.7	0.317	0.011	0.039	0.001
Pumpkinseed fish	3.49	0.084	2.34	0.056	5.20	0.125	1316	31.6	136	3.26	0.476	0.011	2.16	0.052
Redfin pickerel	22.9	0.229	6.70	0.067	14.4	0.144	9159	91.6	217	2.17	1.08	0.011	0.133	0.001
Station B (16.1 km)														
American eel (10 cm)	16.5	0.165	23.59	0.236	19.1	0.191	2597	26.0	284	2.84	22.3	0.223	0.312	0.003
American eel (110 cm)	2.26	0.136	0.034	0.002	13.4	0.807	23.0	1.38	21.5	1.29	0.191	0.011	0.023	0.001
Banded killifish	7.12	0.142	8.02	0.160	9.77	0.195	2609	52.2	115	2.29	3.45	0.069	14.6	0.293
Blue crab	14.8	0.340	0.175	0.004	12.4	0.285	724	16.7	65.5	1.51	4.38	0.101	20.6	0.475
Eastern silvery minnow	46.1	1.61	21.3	0.744	19.7	0.689	9972	349	425	14.9	0.467	0.016	0.057	0.002
Pumpkinseed fish	11.8	0.283	4.44	0.106	10.7	0.257	1759	42.2	161	3.88	13.8	0.331	0.195	0.005
Striped bass	7.79	0.125	11.6	0.186	11.3	0.182	2521	40.3	203	3.25	2.38	0.038	0.292	0.005
White perch	33.3	1.33	15.1	0.602	19.8	0.791	5027	201	221	8.85	0.260	0.010	18.1	0.722
Station C (7.4 km)														
Atlantic silverside (7.8-9.4 cm)	9.97	0.239	29.9	0.718	27.5	0.661	2142	51.4	244	5.85	2.12	0.051	0.053	0.001
Eastern silvery minnow	39.9	1.40	42.3	1.48	17.2	0.603	2679	93.8	214	7.49	2.08	0.073	0.057	0.002
Gizzard shad	0.241	0.003	72.8	0.801	55.5	0.610	5627	61.9	242	2.66	1.49	0.016	0.182	0.002
Mummichog	14.3	0.157	19.3	0.213	6.08	0.067	2123	23.4	132	1.45	1.04	0.011	20.2	0.222
Striped bass (10 cm)	40.0	0.400	38.0	0.380	70.0	0.700	9103	91.0	400	4.00	7.98	0.080	0.201	0.002
Striped bass (20 cm)	11.2	0.180	4.40	0.070	10.5	0.168	1922	30.8	89.3	1.43	6.51	0.104	0.175	0.003
Striped bass (30 cm)	11.5	0.196	17.8	0.302	8.02	0.136	2702	45.9	93.1	1.58	0.673	0.011	0.083	0.001
White perch	12.2	0.487	8.59	0.343	15.1	0.604	2585	103	120	4.82	1.23	0.049	4.22	0.169

Table 1. Summary of PCM concentrations in biota from the lower Passaic River in ng/g lipid weight and ng/g wet weight.

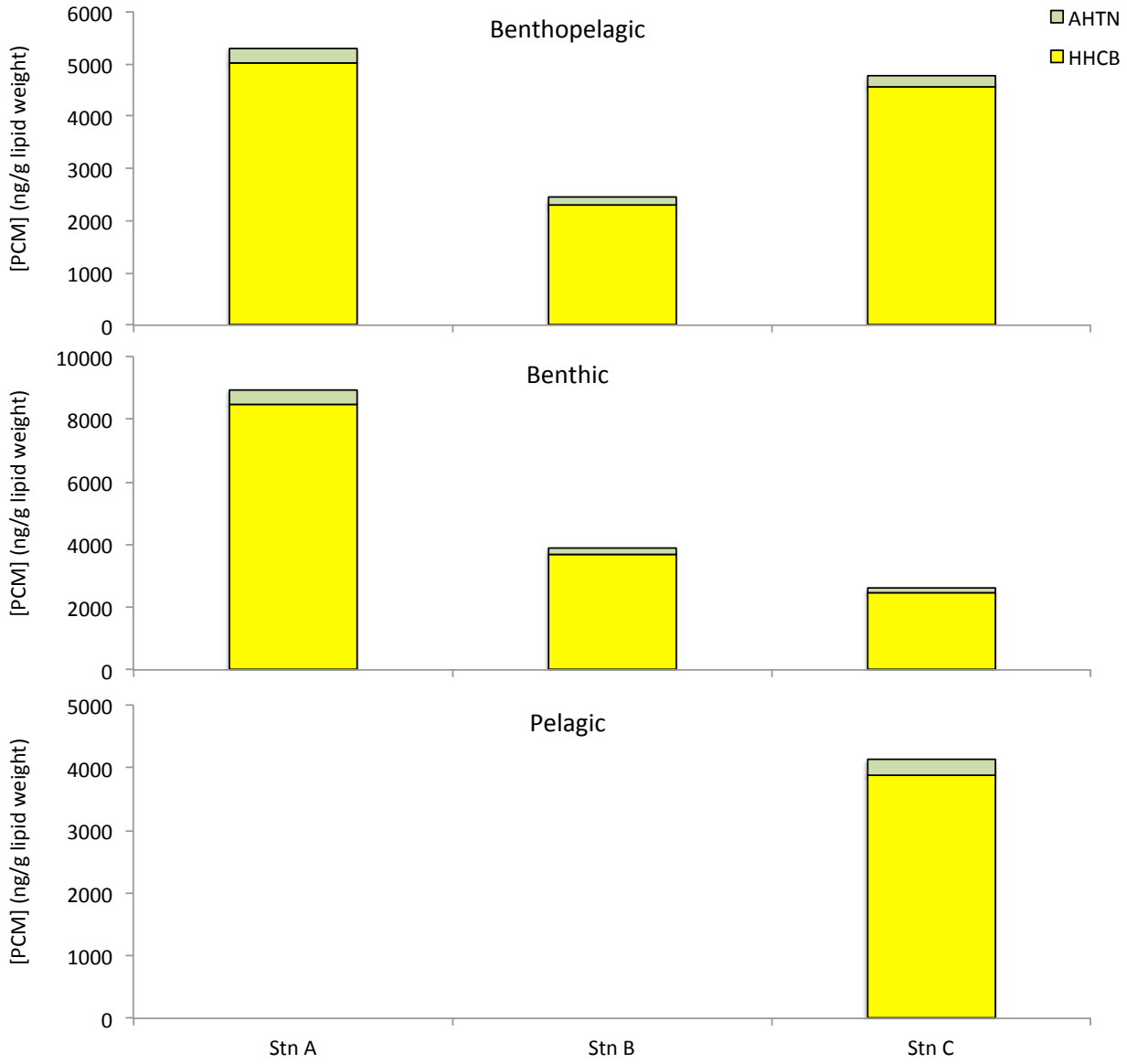

Figure 2. Mean concentration of AHTN and HHCB, the most abundant PCMs (ng/g lipid) in carnivore, omnivore, and planktivore species at each location. No pelagic biota samples were available for Stn A and Stn B.

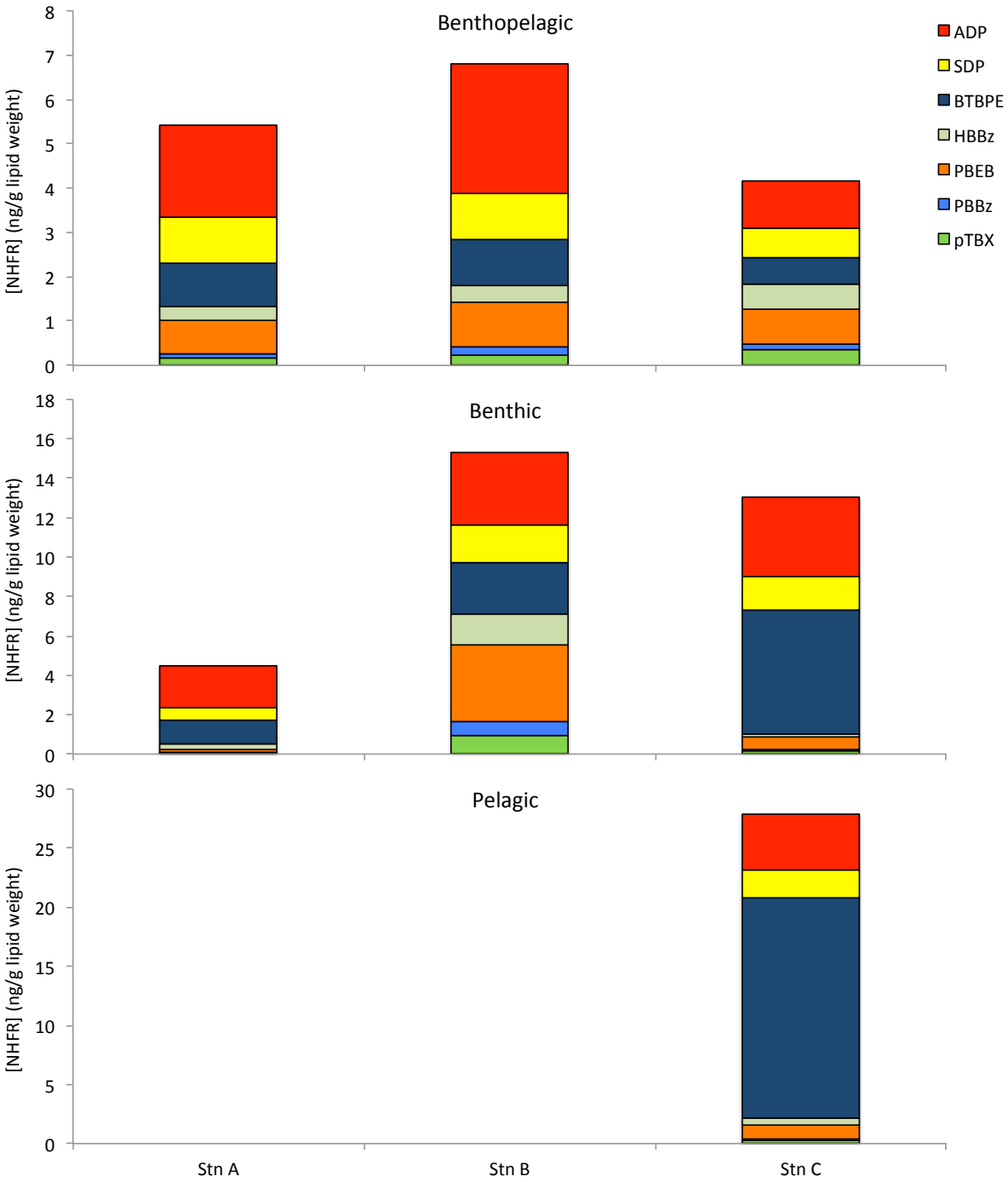

Figure 3. Mean concentration of NHFRs (ng/g lipid) in carnivore, omnivore, and planktivore species at each location. No pelagic biota samples were available for Stn A and Stn B.

Species	pTBX		PBBz		PBEB		HBBz		BTBPE		SDP		ADP	
	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww	pg/g lipid	pg/g ww
Station A (Above Dundee Dam)														
Banded killifish	138	2.76	76.8	1.54	439	8.78	776	15.5	557	11.1	1423	28.5	3745	74.9
Bluegill	205	6.75	46.5	1.54	330	10.9	150	4.94	630	20.8	627	20.7	1114	36.8
Eastern silvery minnow; benthic	43.8	2.37	30.1	1.63	172	9.30	289	15.7	1202	65.0	649	35.1	2062	112
Pumpkinseed fish	133	3.20	91.5	2.20	523	12.6	188	4.51	1683	40.4	1553	37.3	2757	66.2
Redfin pickerel	179	1.79	123	1.23	1793	17.9	158	1.58	1010	10.1	529	5.29	719	7.19
Station B (16.1 km)														
American eel (10 cm)	671	6.71	461	4.61	2635	26.4	591	5.91	1497	15.0	7063	70.6	14237	142
American eel (110 cm)	33.9	2.03	23.0	1.38	132	7.91	1302	78.1	42.6	2.56	552	33.1	717	43.0
Banded killifish	112	2.24	76.8	1.54	1271	25.4	167	3.35	330	6.59	97.7	1.95	214	4.28
Blue crab	3803	87.5	2613	60.1	14945	344	3354	77.1	9796	225	605	13.9	1155	26.6
Eastern silvery minnow	105	3.66	71.8	2.51	411	14.4	1305	45.7	1103	38.6	808	28.3	1447	50.6
Pumpkinseed fish	355	8.52	200	4.81	824	19.8	645	15.5	1247	29.9	1377	33.1	1753	42.1
Striped bass	251	4.02	227	3.64	988	15.8	306	4.90	1517	24.3	1640	26.2	6822	109
White perch	183	7.30	130	5.22	1420	56.8	1317	52.7	492	19.7	633	25.3	1022	40.9
Station C (7.4 km)														
Atlantic silverside (7.8-9.4 cm)	210	5.03	144	3.46	824	19.8	318	7.63	3140	75.3	1330	31.9	2713	65.1
Eastern silvery minnow	57.5	2.01	39.5	1.38	226	7.91	50.7	1.77	16513	578	2387	83.6	6035	211
Gizzard shad	289	3.18	132	1.46	1650	18.1	687	7.55	34287	377	3189	35.1	6858	75.4
Mummichog	198	2.18	136	1.50	1352	14.9	174	1.92	1495	16.4	2238	24.6	5462	60.1
Striped bass (10 cm)	680	6.80	173	1.73	988	9.88	1299	13.0	386	3.86	1297	13.0	1905	19.1
Striped bass (20 cm)	229	3.7	157	2.51	898	14.4	217	3.47	1387	22.2	478	7.64	1066	17.1
Striped bass (30 cm)	113	1.92	77.5	1.32	443	7.53	220	3.74	55.9	0.951	164	2.79	216	3.67
White perch	265	10.6	34.6	1.38	198	7.91	377	15.1	797	31.9	409	16.3	590	23.6

Table 2. Summary of NHFR concentrations in biota from the lower Passaic River in pg/g lipid weight and pg/g wet weight. .

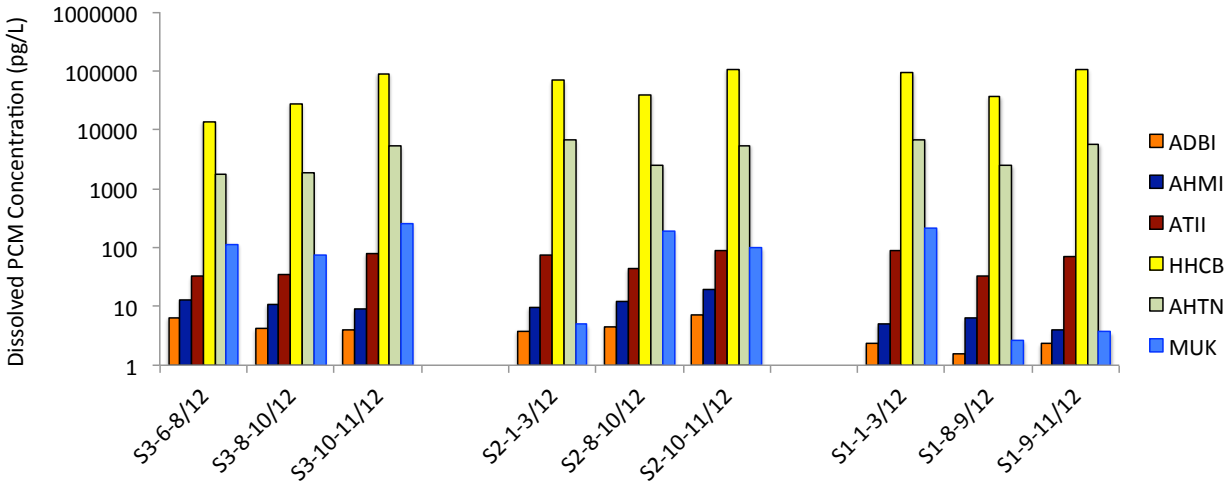

Figure 4. Composition of PCMs in surface water of the lower Passaic.

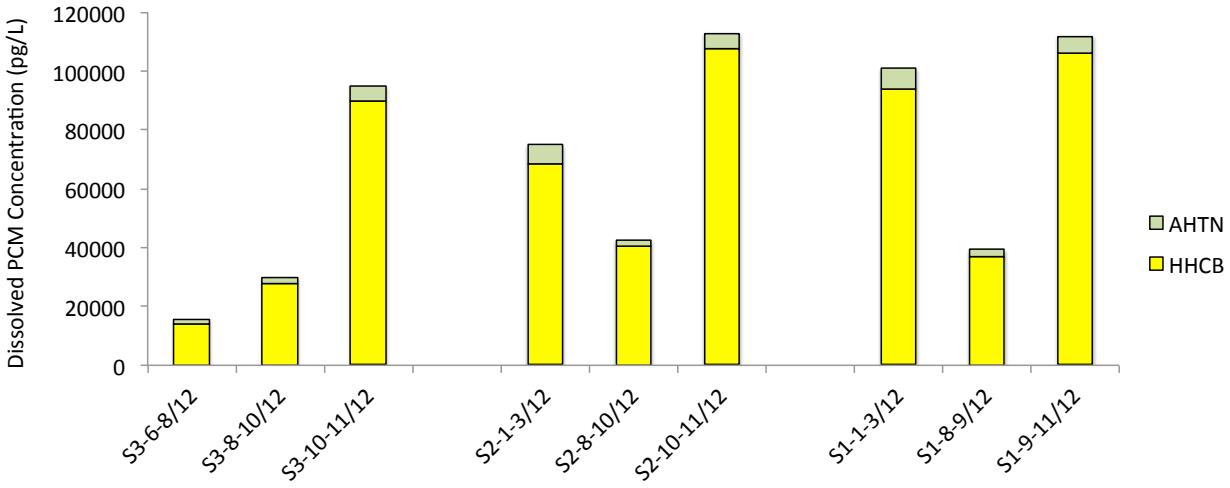

Figure 5. Concentration of HHCB and AHTN dissolved in surface water of the lower Passaic.

Site	S1	S1	S1	**S1**	S2-1-3/12	S2-8-10/12	S2-10-11/12	**S2**	S3-6-8/12	S3-8-10/12	S3-10-11/12	**S3**
Dates Deployed	1-3/2012	8-9/2012	9-11/2012	**MEAN**	1-3/2012	8-10/2012	10-11/2012	**MEAN**	6-8/2012	8-10/2012	10-11/2012	**MEAN**
River Km	KM 28	KM 28	KM 28	**KM 28**	KM 14	KM 14	KM 14	**KM 14**	KM 9.6	KM 9.6	KM 9.6	**KM 9.6**
Polycyclic Musks (PCMs)												
ADBI	2.36	1.54	2.32	**2.08**	3.86	4.46	7.10	**5.14**	6.38	4.16	3.93	**4.82**
AHMI	4.95	6.21	3.97	**5.05**	9.47	12.2	19.1	**13.6**	12.9	10.7	8.85	**10.8**
ATII	88.8	33.7	69.8	**64.1**	76.1	43.7	88.4	**69.4**	31.9	34.6	78.4	**48.3**
HHCB	93713	36807	106104	**78875**	68244	40201	107274	**71906**	13877	27912	89619	**43803**
AHTN	6934	2452	5574	**4987**	6567	2479	5426	**4824**	1767	1853	5188	**2936**
MUX	1.40	0.744	1.02	**1.06**	1.36	0.767	2.64	**1.59**	1.98	0.718	3.86	**2.19**
MUK	210	2.69	3.68	**72.2**	4.91	186	98.6	**96.4**	115	74.8	261	**150**
Novel Halogenated Flame Retardants (NHFRs)												
pTBX	0.074	0.040	0.054	**0.056**	0.583	0.041	0.470	**0.365**	0.343	0.241	0.054	**0.213**
PBB	4.98	0.832	1.14	**2.32**	38.7	27.2	24.6	**30.2**	21.5	27.1	50.9	**33.2**
PBT	0.061	0.034	0.046	**0.047**	0.246	0.035	0.047	**0.110**	0.141	0.033	0.044	**0.073**
PBEB	0.034	0.121	1.22	**0.458**	0.205	0.228	0.135	**0.189**	0.295	0.110	0.274	**0.226**
HBB	0.352	0.192	0.259	**0.267**	0.923	0.197	0.265	**0.462**	0.415	0.185	0.257	**0.286**
BTBPE	0.036	0.183	0.082	**0.100**	0.037	0.245	0.105	**0.129**	0.326	0.068	0.160	**0.185**
SDP	0.006	0.010	0.019	**0.012**	0.013	0.030	0.026	**0.023**	0.049	0.025	0.025	**0.033**
ADP	0.012	0.018	0.035	**0.022**	0.022	0.048	0.035	**0.035**	0.083	0.030	0.035	**0.049**

Table 3. Concentrations of PCMs and NHFRs in surface waters from the lower Passaic derived from PE samplers.

	ADBI		AHMI		ATII		HHCB		AHTN		MUX		MUK	
	lipid	ww	lipid	ww	lipid	ww	lipid	ww	lipid	ww	lipid	ww	lipid	ww
Station A (Above Dundee Dam)														
Banded killifish	3317157	66343	2643585	52872	2201960	44039	41690	834	71910	1438	3135841	62717	1080	22
Bluegill	5555920	183345	1847838	60979	346500	11435	80014	2640	89817	2964	315755	10420	65116	2149
Eastern silvery minnow; benthic	15303242	535613	5595853	195855	295310	10336	107533	3764	89870	3145	300602	10521	539	19
Pumpkinseed fish	1681709	40361	464291	11143	81086	1946	16680	400	27201	653	451529	10837	29962	719
Redfin pickerel	11024606	110246	1327536	13275	224209	2242	116124	1161	43553	436	1024263	10243	1838	18
Station B (16.1 km)														
American eel (10 cm)	3205862	32059	1736144	17361	274617	2746	36119	361	58956	590	14015356	140154	3235	32
American eel (110 cm)	439647	26379	2475	148	193819	11629	321	19	4455	267	119866	7192	243	15
Banded killifish	1384170	27683	590312	11806	140760	2815	36289	726	23762	475	2170795	43416	151723	3034
Blue crab	2871733	66050	12912	297	178810	4113	10070	232	13587	312	2751503	63285	213916	4920
Eastern silvery minnow	8956582	313480	1563844	54735	283525	9923	138680	4854	88165	3086	293550	10274	594	21
Pumpkinseed fish	2295821	55100	326372	7833	154178	3700	24466	587	33477	803	8671238	208110	2022	49
Striped bass	1514926	24239	855957	13695	163522	2616	35057	561	42073	673	1498329	23973	3033	49
White perch	6467874	258715	1107655	44306	284908	11396	69908	2796	45863	1835	163454	6538	187181	7487
Station C (7.4 km)														
Atlantic silverside (7.8-9.4 cm)	2067369	49617	2761044	66265	570181	13684	48909	1174	83029	1993	969271	23262	354	8
Eastern silvery minnow	8279796	289793	3903299	136615	356590	12481	61170	2141	72890	2551	951100	33289	381	13
Gizzard shad	49865	548.51	6720444	73925	1148578	12634	128460	1413	82415	907	678599	7465	1213	13
Mummichog	2964532	32610	1786102	19647	125856	1384	48476	533	45017	495	475019	5225	134086	1475
Striped bass (10 cm)	8288084	82881	3508657	35087	1448791	14488	207813	2078	136074	1361	3644405	36444	1334	13
Striped bass (20 cm)	2327440	37239	405933	6495	216728	3468	43881	702	30400	486	2975358	47606	1168	19
Striped bass (30 cm)	2393392	40688	1639188	27866	166097	2824	61680	1049	31710	539	307365	5225	549	9
White perch	2521825	100873	792602	31704	312378	12495	59003	2360	41011	1640	562550	22502	28081	1123

Table 4. BAFs for PCMs in biota from the lower Passaic based on both lipid and wet weight concentrations compared to truly dissolved surface water concentrations.

	pTBX		PBBz		PBEB		HBBz		BTBPE		SDP	
	lipid	ww	lipid	ww	lipid	ww	lipid	ww	lipid	ww	lipid	ww
Station A (Above Dundee Dam)												
Banded killifish	2467029	49341	33158	663	959826	19197	2902544	58051	5560407	111208	119554940	2391099
Bluegill	3662523	120863	20096	663	721031	23794	560099	18483	6287235	207479	52638345	1737065
Eastern silvery minnow; benthic	783353	42372	12981	702	375770	20326	1082467	58552	12007948	649521	54517003	2948874
Pumpkinseed fish	2383440	57203	39497	948	1143323	27440	703525	16885	16805953	403343	130454096	3130898
Redfin pickerel	3201460	32015	53053	531	3918220	39182	589891	5899	10082873	100829	44412903	444129
Station B (16.1 km)												
American eel (10 cm)	1839615	18396	15269	153	13918048	139180	1280269	12803	11609573	116096	306719498	3067195
American eel (110 cm)	92937	5576	763	46	695902	41754	2819758	169185	330455	19827	23973756	1438425
Banded killifish	306602	6132	2545	51	6710851	134217	362470	7249	2556672	51133	4241979	84840
Blue crab	10432596	239950	86590	1992	78930327	1815398	7260503	166992	75994021	1747862	26291562	604706
Eastern silvery minnow	286693	10034	2380	83	2169046	75917	2825645	98898	8560568	299620	35081941	1227868
Pumpkinseed fish	974099	23378	6635	159	4349390	104385	1396979	33527	9670820	232100	59801135	1435227
Striped bass	689855	11038	7538	121	5219268	83508	662674	10603	11769604	188314	71205248	1139284
White perch	500812	20032	4322	173	7499487	299979	2851384	114055	3820589	152824	27485914	1099437
Station C (7.4 km)												
Atlantic silverside (7.8-9.4 cm)	985819	23660	4341	104	3639686	87352	1113245	26718	16980811	407539	40417523	970021
Eastern silvery minnow	270396	9464	1191	42	998314	34941	177507	6213	89312584	3125940	72550938	2539283
Gizzard shad	1361266	14974	3987	44	7290571	80196	2404832	26453	185450283	2039953	96926289	1066189
Mummichog	930789	10239	4098	45	5973439	65708	611037	6721	8084808	88933	68009242	748102
Striped bass (10 cm)	3199269	31993	5209	52	4367623	43676	4550651	45507	2089430	20894	39432838	394328
Striped bass (20 cm)	1075438	17207	4735	76	3970567	63529	759630	12154	7502754	120044	14514061	232225
Striped bass (30 cm)	530188	9013	2334	40	1957478	33277	770035	13091	302517	5143	4980004	84660
White perch	1245682	49827	1042	42	873525	34941	1321020	52841	4308872	172355	12418620	496745

Table 5. BAFs for NHFRs in biota from the lower Passaic based on both lipid and wet weight concentrations compared to truly dissolved surface water concentrations.

Supporting Information

Table S1: PE Deployment Details

	Date Deployed	Date Recovered	Days Deployed	Average Temperature (°C)	PE Mass (g)	Sampling Rate (L/day)
River km 28: 40.895 N, 74.129 W						
PE-19-1	12/19/11	3/24/12	96	8.5	1.81	100
PE-19-2	8/12/12	9/29/12	48	25.2	1.88	97
PE-19-3	9/29/12	11/29/12	61	13	2.11	97
River km 14: 40.805 N, 74.1391 W						
PE-9.6-1	1/5/12	3/24/12	79	7.4	1.95	117
PE-9.6-2	8/12/12	10/8/12	57	20.8	2.12	82
PE-9.6-3	10/8/12	11/29/12	52	13.2	2.09	61
River km 9.6: 40.744 N, 74.166 W						
PE-6.4-1	6/8/12	8/12/12	65	24.5	1.88	66
PE-6.4-2	8/12/12	10/8/12	57	23.1	2.09	86
PE-6.4-3	10/8/12	11/29/12	52	13.6	2.05	153

Table S2: Summary of Collected Biota

Species	Common Name	Habitat	Trophic Level	Number of Pooled Specimens	Specimen Length (cm)	Date Collected	Total Wet Weight (g) (PCM Analysis)	Total Wet Weight (g) (NHFR Analysis)	Percent Lipid Content	$\delta^{13}C$	$\delta^{15}N$
Above Dundee Dam (Station A)											
Esox americanus	Redfin pickerel	Bentho-pelagic	Carnivore	10	11.5-13	10/16/11	10.6	23	1	-25	12
Fundulus diaphanus	Banded killifish	Bentho-pelagic	Carnivore	20	4.5-7.5	10/16/11	9.0	18/12*	2	-25	13
Hybognathus regius	Eastern silvery minnow	Benthic	Omnivore	55	4.0-7.5	10/16/11	10.3	17	3.5	-25	12
Lepomis gibbosus	Pumpkinseed fish	Bentho-pelagic	Carnivore	18	9.0-12.5	10/16/11	10.0	17/10*	2.4	-24	14
Lepomis macrochirus	Bluegill	Bentho-pelagic	Carnivore	34	3.0-5.0	10/16/11	10.4	18	3.3	-27	13
Downriver km 16.1 (Stn B)											
Anguilla rostrata	American eel (110 cm)	Benthic	Carnivore	1	110	10/19/12	10	20	6	-25	14
Anguilla rostrata	American eel (11-12 cm)	Benthic	Carnivore	5	11-12	8/25/11	4.5	6	1	-26	11
Callinectes sapidus	Blue crab	Benthic	Omnivore	4	9.0-11	9/22/11	5	20	2.3	-25	11
Fundulus diaphanus	Banded killifish	Bentho-pelagic	Carnivore	28	4.5-7.5	8/25/11	9.6	15/18*	2	-25	13
Hybognathus regius	Eastern silvery minnow	Benthic	Omnivore	10	4.5-5.8	8/25/11	7	11	3.5	-25	12
Lepomis gibbosus	Pumpkinseed fish	Bentho-pelagic	Carnivore	15	10-13	8/26/11	3.0	8	2.4	-24	14
Morone americana	White perch	Benthic	Carnivore	15	8.0-16	8/25/11	11	18	4	-26	14
Morone saxatilis	Striped bass	Bentho-pelagic	Carnivore	2	18-20	10/19/12	5.0	10	1.6	-23	14
Downriver km 7.4 (Stn C)											
Dorosoma cepedianum	American gizzard shad	Pelagic	Planktivore	15	5.0-9.0	9/22/11	7	19	1.1	-28	10
Fundulus heteroclitus	Mummichog	Benthic	Omnivore	25	4.4-7.9	8/19/11	10	18/19*	1.1	-25	9
Hybognathus regius	Eastern silvery minnow	Benthic	Omnivore	25	4.5-5.8	9/22/11	7	20	3.5	-25	12
Menidia menidia	Atlantic silverside (large)	Pelagic	Planktivore	15	7.8-9.4	10/20/11	11	8	2.4	-24	14
Morone americana	White perch	Benthic	Carnivore	12	7.0-13	10/20/11	11.2	20	4	-24	13
Morone saxatilis	Striped bass (10 cm)	Bentho-pelagic	Carnivore	5	9.6-10.4	9/22/11	7	16	1	-23	15
Morone saxatilis	Striped bass (20 cm)	Bentho-pelagic	Carnivore	4	20-23	9/22/11	5	11	1.6	-23	14
Morone saxatilis	Striped bass (30 cm)	Bentho-pelagic	Carnivore	3	28.2-32.5	10/19/12	10	21	1.7	-19	16

*Samples with two weights listed were prepared in duplicate.

Table S3. Target Compounds and Derived PE-Water Partitioning Coefficients

Compound	Full Name	CAS Number	log K_{PEW} (L/L)[a]
Polycyclic Musks (PCMs)			
ADBI	Celestolide	13171-00-1	6.7
AHMI	Phantolide	15823-35-0	6.4
ATII	Traesolide	68140-48-7	5.9
HHCB	Galaxolide	1222-05-5	4.5
AHTN	Tonalide	1506-02-1	4.6
MUX	Musk xylene	81-15-2	5.0
MUK	Musk ketone	81-14-1	4.4
Novel Halogenated Flame Retardants (NHFRs)			
pTBX	tetrabromo-*p*-xylene	23488-38-2	5.7
PBBz	pentabromobenzene	608-90-2	4.0
PBT	pentabromotoluene	87-83-2	5.9
PBEB	pentabromoethylbenzene	85-22-3	6.5
HBBz	hexabromobenzene	87-82-1	5.8
BTBPE	1,2-bis[2,4,6-tribromophenoxy)ethane	37853-59-1	8.1
SDP	*syn*-Dechlorane Plus	13560-89-9	11.5
ADP	*anti*-Dechlorane Plus	13560-89-9	11.5

[a] K_{PEW} values for PCMs were calculated from aqueous solubility as in Lohmann (2012), and K_{PEW}s for NHFRs were calculated from K_{OW} as in Lohmann & Muir (2010).

Table S4. Ions Monitored in Chemical Analysis Methods

	RT (min)	Primary Ion	Secondary Ion	Tertiary Ion
Polycyclic Musks (PCMs)				
ADBI	16.1	229	244	
AHMI	16.6	229	244	
ATII	17.9	215	258	
HHCB	17.9	243	258	
AHTN	18.0	243	258	
Musk Xylene	17.9	282	297	
Musk Ketone	19.4	279	294	
Novel Halogenated Flame Retardants (NHFRs)				
DiBB	14.9	79	81	
pTBX	17.3	79	81	422
PBBz	17.6	79	81	472
PBT	19.7	486	79	81
PBEB	20.4	79	81	500*
TetraBB	20.6	79	81	
HBBz	22.5	551	79	81
PentaBB	23.5	548	79	81
OCN	28.1	404	368	
BTBPE	35.3	79	81	
SDP	36.7	652	654	584
ADP	37.4	652	654	584

* PBEB was quantified using ion 500 as the primary ion for PE extract analysis due to interference from brominated performance reference compound TetraBB on the 79/81 ions.

Table S5. Trophic Magnification Factors for All Biota Samples. Calculated from the antilog of the slope of regression between lipid-normalized natural log of tissue concentration versus trophic position. P-value of the regression is shown and compounds for which the trophic position-concentration relationship was significant are in bold.

Compound	TMF	p
pTBX	0.63	0.38
PBBz	0.55	0.27
PBEB	0.40	0.10
HBBz	1.0	1.00
BTBPE	**0.11**	**0.003**
SDP	**0.36**	**0.03**
ADP	**0.28**	**0.02**
ADBI	1.4	0.58
AHMI	0.61	0.61
ATII	1.0	0.97
HHCB	0.72	0.63
AHTN	0.81	0.60
MUX	0.82	0.78
MUK	0.26	0.26