# Pharmaceuticals, Personal Care Products and Anthropogenic Waste Indicators Detected in Streambed Sediments of the Lower Columbia River and Selected Tributaries

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## Abstract

One byproduct of advances in modern chemistry is the accumulation of synthetic chemicals in the natural environment. These compounds include "pharmaceuticals and personal care products" (PPCPs) and "anthropogenic waste indicators" (AWIs), some of which are endocrine disrupting compounds (EDCs) that can have detrimental reproductive effects in wildlife and in humans. Methods have been developed to screen for large suites of PPCPs and AWIs in aqueous media, but the role of sediments in exposure of aquatic organisms to these chemicals is less well understood. The first methods capable of analyzing a large suite of these compounds in solid media were published in 2005. Here we present an application of these methods to a small-scale reconnaissance of PPCPs and AWIs in natural bed sediments of the lower Columbia River Basin. Surficial bed sediment samples were collected from the Columbia River, the Willamette River, the Tualatin River, and several small urban creeks in Oregon. Forty-nine compounds were detected at concentrations ranging from <1 to >1000 ng [g sediment]$^{-1}$ dry weight basis (<1 to >10 µg [g OC]$^{-1}$). Concentrations and frequency of detection were higher in tributaries and small urban creeks than in the Columbia River mainstem, pointing to a higher risk of toxicity to juvenile salmonids and other aquatic life in lower order streams. Thirteen known or suspected EDCs were detected during the study. At least one EDC was detected at 22 of 23 sites sampled; several EDCs were relatively widespread among the sites. This study is the first to document the occurrence of a large suite of PPCPs and AWIs in the sediments of the Columbia River Basin. A better understanding of the fate and effects of these classes of emerging contaminants is needed, especially because their use and discharge into the environment is likely to increase in the future.

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**Introduction**

Modern chemistry has produced numerous compounds that facilitate everyday life and save lives through human and veterinary medicine. One byproduct of these advances is the accumulation of synthetic chemicals in the natural environment. The compounds include pharmaceuticals, synthetic fragrances, detergents, disinfectants, plasticizers, preservatives, and others present in wastewater and agricultural and urban runoff, and are commonly referred to as "pharmaceuticals and personal care products" (PPCPs) and/or anthropogenic waste indicator (AWI) compounds. A subset of PPCPs and AWIs includes endocrine disrupting compounds (EDCs) that have detrimental reproductive effects in fish (e.g., Brian et al. 2007) and in humans (Guillette 1995). EDCs mimic or block natural hormones in the body and disrupt normal function and development. These compounds enter the aquatic environment from a myriad of sources, including, for example, treated industrial and municipal wastewater or private septic systems, untreated sewage overflows resulting from storm surges, biosolids applied to land as fertilizer, landfill leachate, unintended cross-connections of storm and sewer systems, illegal dumping, and aquaculture.

Although little is known about the environmental transport and fate of most compounds considered here, many sorb to sediments, and several hydrophilic compounds that are weak sorbates have been shown to migrate through river bed sediments (Labadie et al. 2007). Once in the aquatic environment, in waters and/or sediments, PPCPs may present an exposure risk to aquatic organisms, although toxicity levels are largely unknown. PPCP concentrations in surface water are rapidly diluted but have been measured in rivers and streams nationwide (Kolpin et al. 2002), and their continual release into water by these various routes can create a chronic exposure or "pseudo-persistence". Sediment sorption is one of the mechanisms by which it is thought that PPCPs may persist in the aquatic environment and degrade habitat for fish and other organisms, and the role of sediments in potential exposure of aquatic organisms to PPCPs needs to be investigated.

Various methods have been developed to screen for large suites of compounds having diverse chemical and physical properties in aqueous media (e.g., Trenholm et al. 2006). Reconnaissance efforts have been made in recent years to assess the presence of some of these compounds in natural waters (e.g., Kolpin et al. 2002, Cahill et al. 2004). The first methods capable of analyzing a large suite of these compounds in solid media were recently published (Burkhardt et al. 2005, 2006, Kinney et al. 2006a,b). To date, published studies using these methods have primarily focused on biosolids, irrigated soils, and test materials. Here we apply these methods to a natural system and present a small-scale reconnaissance of PPCPs in bed sediments of the lower Columbia River and several tributaries and urban creeks in northwest Oregon.

**Study Site and Sample Collection**

The Columbia River is the fourth largest by volume in the Nation and drains a 295,000 square-mile basin that comprises land in seven states and one Canadian territory. The Columbia River Basin provides important hydroelectric power generation, anadromous

fisheries, large recreational areas and scenic beauty, and valuable habitat for wildlife and fish.

Surface sediment (top 1-3 cm) samples were collected from the lower Columbia River, the Willamette River, the Tualatin River, and several small urban creeks (Figure 1; Table 1). NOAA and the Oregon Department of Environmental Quality (ODEQ) collected Columbia River sediment samples from Pt. Adams to Warrendale. Each sample represents a homogenized composite of three petite Ponar or Van Geen bottom grabs. At the NOAA sites (river mile [RM] 4, 54, 82, 101, 141), three samples were taken at regular intervals from a shallow, near-shore area of approximately 10 m diameter. The ODEQ sample sites (RM 66, 68, 102, 110) were randomly selected and not targeted to a specific environment. These were also composites of three grab samples collected while on station (the acceptable tolerance for being on station was 0.02 nautical miles [+/-37 m]). The U.S. Geological Survey collected sediment samples at the tributary sites by single-point grab sampling. Shallow water depositional areas were selected and site locations were targeted upstream and downstream of several area wastewater treatment facility (WWTF) effluents. However, many of the small tributary sites were close enough to the receiving river so that flooding could confuse the source of the sampled sediments. All samples were collected using solvent-rinsed materials and were stored frozen in certified organics-free glass jars until analyzed at the U.S. Geological Survey National Water Quality Laboratory in Denver, CO.



Figure 1. Sampling site locations in the lower Columbia River and selected tributaries. Refer to Table 1 for explanation of site name abbreviations.

Table 1. Site names, percent organic carbon, and locations

| Site Name | % OC | River Mile | Location |
|---|---|---|---|
| *Tributary Sites* | | | |
| CS2 | 11.5 | n/a | Columbia Slough at Smith and Bybee Park |
| CS1 | 5.0 | n/a | Columbia Slough at Portland Rd |
| W5 | 3.4 | n/a | Willamette R at Morrison Street Bridge |
| Jo | 3.6 | n/a | Johnson Cr at Willamette R confluence |
| W4 | 2.8 | n/a | Willamette R d/s Kellogg Cr and WWTF |
| Ke | 2.4 | n/a | Kellogg Cr at Willamette R confluence |
| W3 | 4.7 | n/a | Willamette R u/s Kellogg Cr and WWTF |
| W2 | 3.9 | n/a | Willamette R d/s Tryon Cr and WWTF |
| Tr | 4.4 | n/a | Tryon Cr at Willamette R confluence |
| W1 | 1.4 | n/a | Willamette R u/s Tryon Cr and WWTF |
| T2 | 4.5 | n/a | Tualatin R d/s Durham City WWTF |
| T1 | 5.3 | n/a | Tualatin R at Fanno Cr |
| Fa2 | 5.2 | n/a | Fanno Cr near Tualatin R confluence |
| Fa1 | 3.9 | n/a | Fanno Cr at Durham City Park |
| *Columbia River Sites* | | | |
| C1 | 2.2 | 4 | at Point Adams |
| C2 | 0.8 | 54 | at Beaver Army Terminal |
| C3 | 0.7 | 66 | d/s Cowlitz R |
| C4 | 14 | 68 | at Cowlitz R and Longview WWTF |
| C5 | 0.7 | 82 | at Columbia City |
| C6 | 1.4 | 101 | at Willamette R |
| C7 | 11.7 | 102 | u/s Willamette R d/s Columbia Blvd WWTF |
| C8 | 0.7 | 110 | u/s Willamette R and Columbia Blvd WWTF |
| C9 | 3.5 | 141 | at Warrendale |

OC = organic carbon, R = river, d/s = downstream, u/s = upstream, Cr = creek,
   WWTF = waste water treatment facility

## Analytical Methods

Samples underwent accelerated solvent extraction (Dionex ASE 200) and were prepared for instrumental analysis using methods previously described (Kinney et al. 2006a,b). Extracts were analyzed for 20 pharmaceutical compounds by liquid chromatography-mass spectrometry (LC-MS) in positive electrospray ionization (ESI) mode. All values below the level of the lowest standard (0.005 ng $\mu l^{-1}$) were discarded. These same extracts were also analyzed separately by LC-MS/MS to confirm compound identities. They were then re-analyzed by LC-MS/MS to screen for 13 antidepressant compounds, which required use of a "tighter" gradient profile (less net change between aqueous and organic eluents over the course of the LC separation) because the antidepressants tend to share more structural similarities than exist between the more diverse compounds in the other pharmaceutical analysis. A separate extraction was performed on all samples for analysis of 61 anthropogenic waste indicator compounds; these extracts were prepared and analyzed by positive ESI gas chromatograph-mass spectrometry (GC-MS) using methods previously described (Burkhardt et al. 2005, 2006). The organic carbon (OC) content of each sediment sample was determined by loss on ignition after drying (Fishman and Friedman 1989). Compound concentrations were normalized to the OC content on a dry weight percent basis.

## Results and Discussion

*Pharmaceutical Compounds*

Pharmaceutical uses and some common trade names are listed in Table 2. Cotinine, codeine, caffeine, trimethoprim, thiabendazole, diphenhydramine, diltiazem, dehydronifedipine, miconazole, azithromycin, and four anti-depressant compounds, venlafaxine, fluoxitine, citalopram and carbamazapine, were detected in samples at concentrations ranging from 4 to >1500 ng (g OC)$^{-1}$. Cimetidine was detected at or below the level of the lowest standard. 1,7-dimethylxanthine, acetaminophen, albuterol, erythromycin, ranitidine, sulfamethoxazole, and warfarin were not detected. Non-detects may owe to small source load or to the chemical characteristics of individual compounds. For instance, sulfamethoxazole does not readily partition to sediments, whereas erythromycin is more likely to partition to sediments if it is present at detectible levels in the environment. The largest number of pharmaceutical compounds was found in sediments from the Tualatin River and Fanno Creek (Figure 2).

Table 2. Pharmaceuticals detected: compounds, uses and trade names

| Compound (generic) | Use | Trade Name |
|---|---|---|
| azithromycin | antibiotic | Zithromax[®a] |
| caffeine | stimulant | - |
| carbamazapine | antiepileptic, antidepressant | Tegretol[®] |
| cimetidine | antacid | Tagamet[®] |
| codeine | analgesic | - |
| cotinine | caffeine metabolite | - |
| dehydronifedipine | antianginal metabolite | - |
| diltiazem | antihypertensive | Cardizem[®] |
| diphenhydramine | antihistamine | Benadryl[®] |
| fluoxetine | antidepressant | Prozac[®] |
| miconazole | antifungal | - |
| thiabendazole | veterinary anthelmintic/pesticide | Mertect[®] |
| trimethoprim | antibiotic | Bactrim[®] |
| venlafaxine | antidepressant | Effexor[®] |
| citalopram | antidepressant | Cipramil[®] |
| sertraline | antidepressant | Zoloft[®] |

[a]Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government
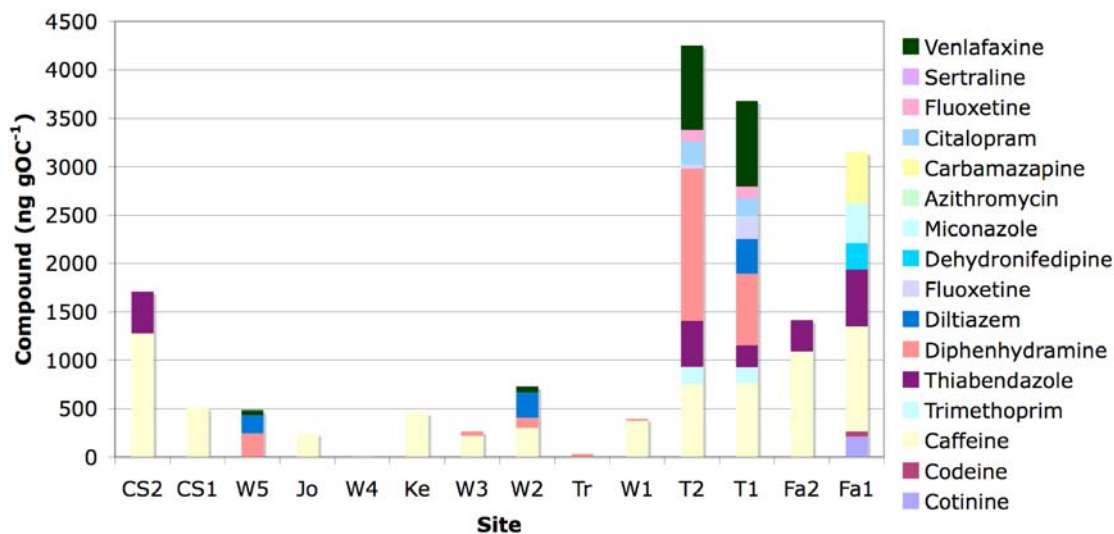
Figure 2. Pharmaceutical compounds (ng gOC$^{-1}$) detected in sediments of the tributary sites displayed from downstream (left) to upstream (right). Number of compounds detected at each site: CS2(2), CS1(1), W5(3), Jo(1), W4(1), Ke(1), W3(2), W2(4), Tr(1), W1(2), T2(8), T1(10), Fa2(2), and Fa1(7).
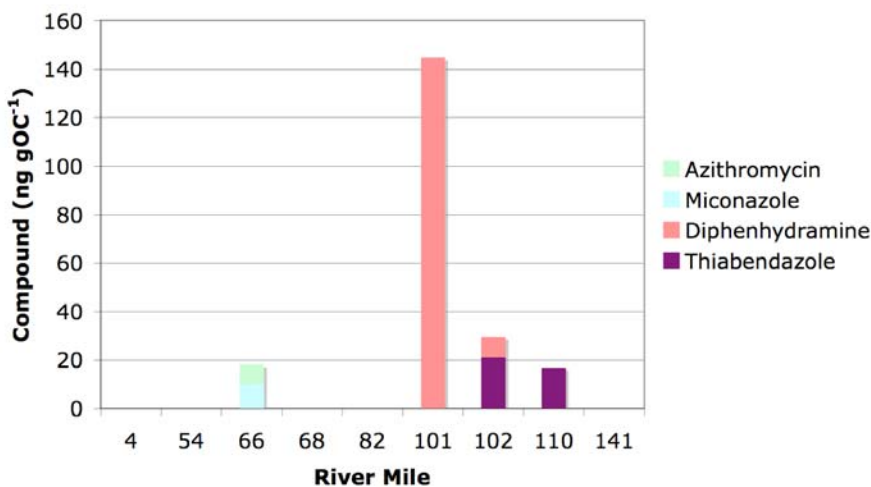


Figure 3. Pharmaceutical compounds (ng gOC$^{-1}$) measured in sediments of the Columbia River sites displayed moving upstream from RM 4 at Point Adams to RM 141 at Warrendale. Note that the concentration scale is different from that of Figure 2. Number of compounds detected at each site: 66(2), 101(1), 102(2), 110(1).

The sites on the Tualatin River and Fanno Creek are the most urbanized sites and probably have the least sediment dilution. The Willamette River downstream of Tryon Creek (W2) and at the Morrison Street Bridge (W5) had more detections than the other Willamette River sites. Site W2 is downstream of the Tryon Creek WWTF, and site W5 periodically receives stormwater overflow. Columbia Slough at Smith and Bybee Park (CS2) had only two compounds present, but their concentrations were relatively high. Historically, Columbia

Slough received stormwater overflow from the Columbia Boulevard WWTF. Sedimentary concentrations of concern have not been determined for these compounds. In water, 50 percent of a test species of green algae showed growth inhibition at a trimethoprim concentration of 16 mg $L^{-1}$ (Lindberg et al. 2007), but effects levels for sedimentary concentrations have not been determined.

Compared to tributary sites, far fewer compounds were detected in Columbia River sediments, and those detected occurred at lower concentrations (Figure 3). None of the anti-depressants were detected in the Columbia River sediments. These samples typically had coarser grain size and higher sand content than sediments from the tributaries and creeks. Columbia River sediments also generally had lower OC content, although two of the sites had very high OC content (Table 1).

*Anthropogenic Waste Indicator Compounds*

At least 2 anthropogenic waste indicator (AWI) compounds (out of 61 total) were detected at every site; many sites had greater than 10 compounds detected. Similar to the pharmaceuticals, many AWI compounds were detected and present at relatively high concentrations in sediments from the Tualatin River and Fanno Creek (Figure 4). The Columbia Slough at Smith and Bybee Park (CS2) and the Willamette River at the Morrison Street Bridge (W5) had a relatively high number of detections and/or relatively large concentrations of AWI compounds present. The Willamette River sites upstream and downstream of Tryon Creek and downstream of Kellogg Creek were relatively "clean" with respect to the waste indicator compounds analyzed.

Although several of these compounds were detected at every Columbia River site, there were fewer compounds detected overall and on average lower concentrations at these sites (Figure 5) as compared to the tributary sites, similar to the patterns observed for the pharmaceuticals. The site at the confluence with the Cowlitz River (RM 68) and the site downstream of the Columbia Blvd WWTF (RM 102) had the most compounds detected (11 and 10, respectively), followed by the site at Pt. Adams (RM 4) (8).
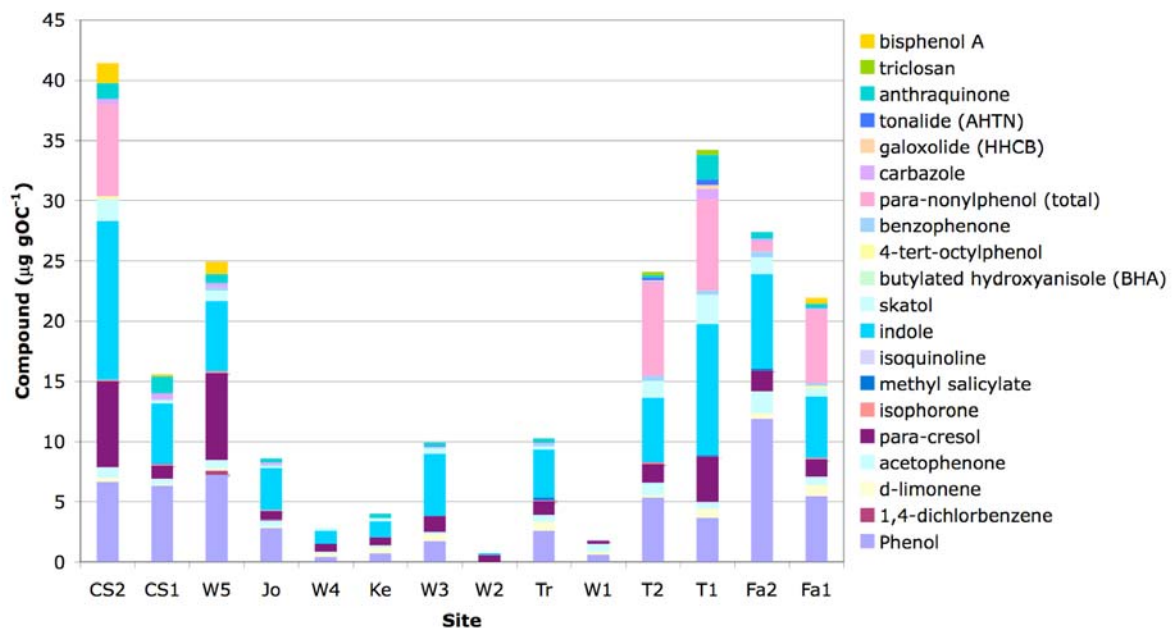
Figure 4. Anthropogenic waste indicator compounds ($\mu$g gOC$^{-1}$) detected in sediments of the tributary sites from downstream (left) to upstream (right). Number of compounds detected at each site: CS2(12), CS1(12), W5(12), Jo(10), W4(6), Ke(7), W3(9), W2(2), Tr(11), W1(5), T2(15), T1(14), Fa2(11), and Fa1(14).
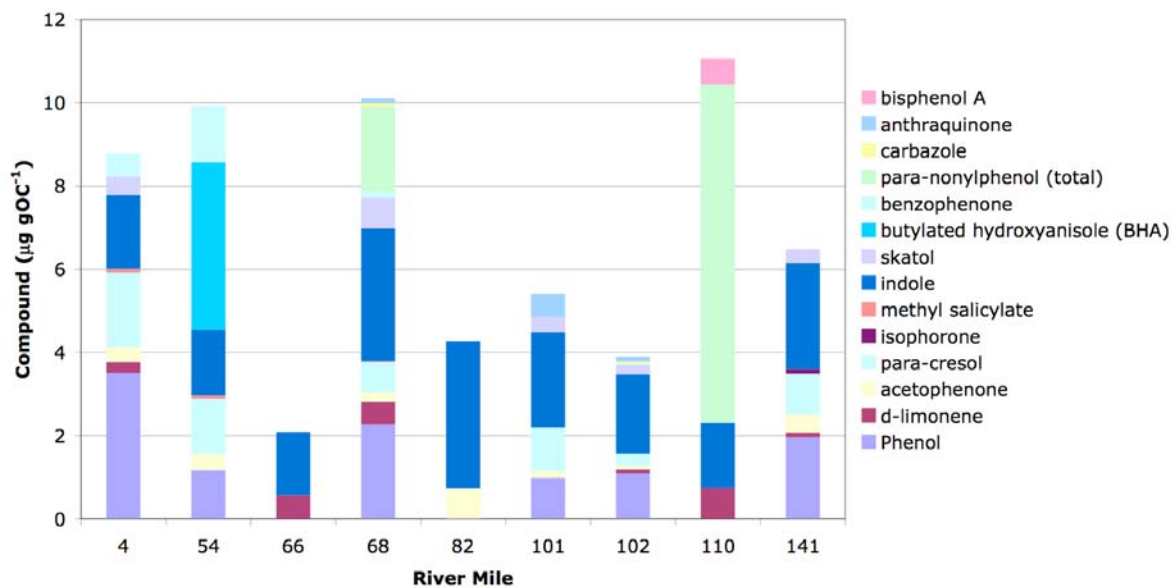


Figure 5. Anthropogenic waste indicator compounds ($\mu$g gOC$^{-1}$) detected in sediments of the Columbia River sites displayed moving upstream from RM 4 at Point Adams to RM 141 at Warrendale. Note that the concentration scale is different from that of Figure 4. Number of compounds detected at each site: 4(8), 54(7), 66(2), 68(11), 82(2), 101(6), 102(10), 110(4), and 141(7).

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There is no clear pattern apparent in the concentrations of AWI compounds at the Columbia River sites. However, when the concentrations are displayed in ng g$^{-1}$ (without normalizing to OC), patterns emerge that reflect those in the number of compounds detected (Figure 6). The sites with the highest number of detections and relatively higher concentrations (in ng g$^{-1}$) are RM 141; RM 102, which is just upstream of the Willamette confluence but just downstream of the Columbia Boulevard WWTF discharge site; RM 68, near the Cowlitz River confluence and Longview WWTF site; and at Point Adams at RM 4, in proximity to another WWTF. The fact that these patterns are most evident when the data are not normalized to %OC reflects the fact that the OC content is widely variable at different sites on the Columbia mainstem, ranging from less than 1% up to 14%. The tributary sites, in contrast, had roughly the same contaminant concentration trends when normalized and when not normalized to OC content (latter not shown). Sediment characteristics are variable between sites, but do not appear to be as great a factor in retention of compounds in sediments at the tributary sites as compared to the Columbia River mainstem sites. Perhaps organic carbon concentrations are sufficiently high in the tributary sediments that they do not limit sorption of contaminants, whereas the organic carbon content of some of the Columbia River samples could be low enough to limit contaminant sorption.
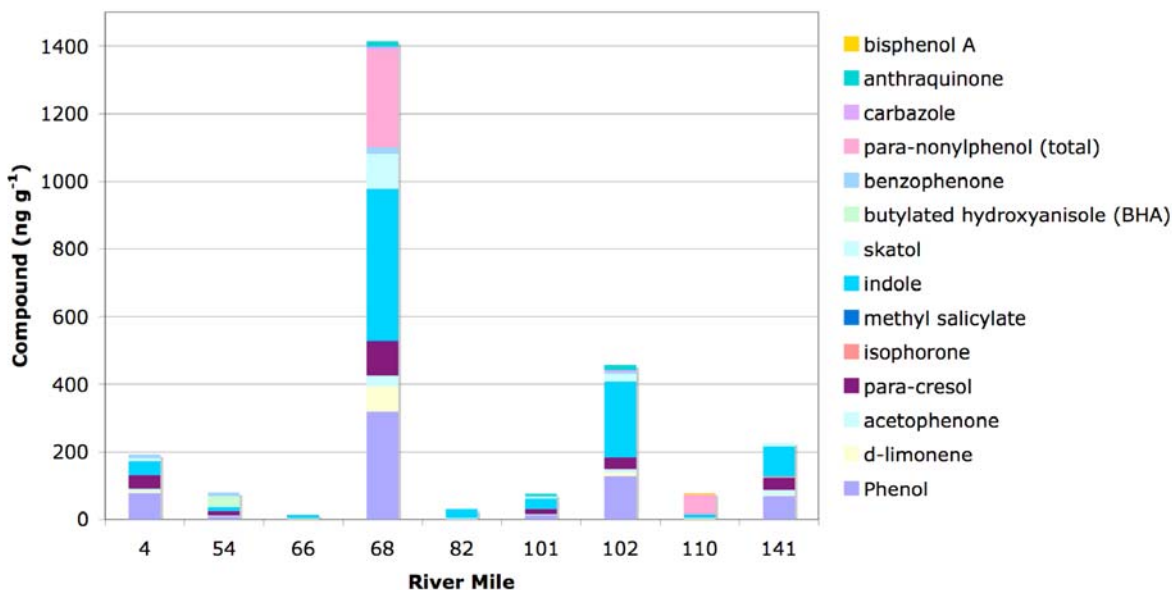


Figure 6. Anthropogenic waste indicator compounds (ng [g sediment]$^{-1}$) detected in sediments of the Columbia River sites. Number of compounds detected at each site as in Figure 5.

*Endocrine Disrupting Compounds (EDCs)*

Most notable was the presence in sediments of several strictly anthropogenic known or suspected EDCs (a subset of the AWI compounds). All AWI compounds, including EDCs, that were detected during this reconnaissance effort are listed in Table 3. Among the tributary sites, the Tualatin River, Fanno Creek, Columbia Slough and the Willamette River at the

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Morrison Street Bridge had relatively more compounds and/or higher concentrations of EDCs detected (Figure 7). Similar to the other classes of compounds, concentrations and detections were lower at the Columbia River sites than at the tributary sites. However, at least one of these compounds was detected at every site sampled except the Columbia River ~2 miles downstream of the Cowlitz River at RM 66 (Figure 8). With a few exceptions, similar patterns were observed in concentrations of polycyclic aromatic hydrocarbons (PAHs) (in addition to benzo[a]pyrene shown in Figures 7 and 8) and steroid compounds (data not shown), which are considered anthropogenic waste indicator compounds but also have natural sources.

Ranking the EDCs by total mass of compound measured at all sites and by frequency of detection shows that para-nonylphenol and para-cresol were present at the highest concentrations, and para-cresol and benzophenone were the most widespread (Figure 9). Although these data do not identify inputs, para-cresole may be sourced predominantly from creosote-coated pilings that are relatively widespread in the system, whereas benzophenone, a fixative for perfumes and soaps, is more likely to enter the system with wastewater. An effects level criteria maximum concentration of 27.75 µg L$^{-1}$ was determined for nonylphenol in water (Brooke and Thursby, 2005), but sedimentary concentrations of concern have not been determined for these compounds. Spatial patterns in concentrations of contaminants in sediments are probably influenced by a combination of factors including contaminant loading, dilution, sedimentary sorption capacity, and compound-dependent characteristics such as partition coefficient and half-life in the environment.
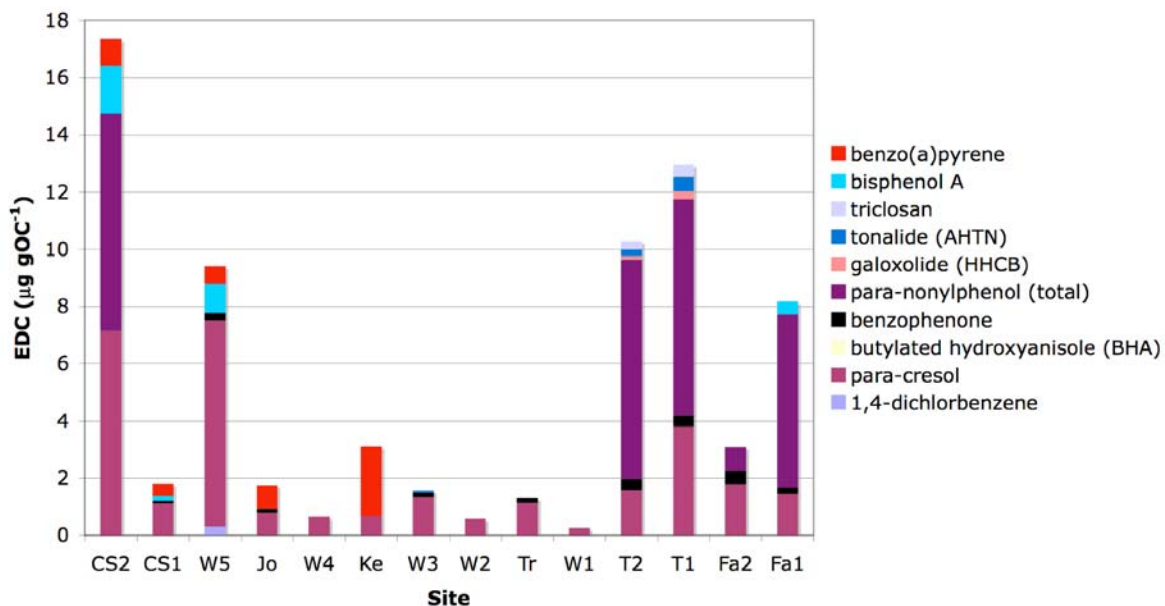


Figure 7. Endocrine disrupting compounds (µg gOC$^{-1}$) detected in sediments of the tributary sites from downstream (left) to upstream (right). Number of compounds detected at each site: CS2(4), CS1(4), W5(5), Jo(3), W4(1), Ke(2), W3(3), W2(1), Tr(2), W1(1), T2(6), T1(6), Fa2(3), and Fa1(4).

Table 3. Wastewater indicator compounds detected during lower Columbia River reconnaissance, known (K) or suspected (S) endocrine disrupting potential, and possible compound uses or sources (after Burkhardt et al. 2006)

| Compound | Strictly anthropogenic | Endocrine disrupting potential | Possible uses or sources |
|---|---|---|---|
| phenol | | | disinfectant, manufacturing |
| 1,4-dichlorobenzene | | S | moth repellant, fumigant, deodorant |
| d-limonene | | | fungicide, antimicrobial, antiviral, fragrance |
| acetophone | | | fragrance in detergent and tobacco, flavor in |
| para-cresol | | S | wood preservative |
| isophorone | | | solvent, resin |
| methyl salicylate | | | liniment, food, beverage, UV-absorbing lotion |
| isoquinoline | | | flavors and fragrances |
| indole | | | fragrance in coffee |
| skatol | | | fragrance, present in feces and coal tar |
| butylated hydroxyanisole (BHA) | | K | food additive, preservative, antioxidant |
| 4-tert-octylphenol | y | K | nonionic detergent metabolite |
| benzophenone | | S | fixative for perfumes and soaps |
| para-nonylphenol | y | K | nonionic detergent metabolite |
| carbazole | y | | insecticide, manufacturing, explosives, lubricants |
| tonalide (AHTN) | y | S | synthetic fragrance |
| galaxolide (HHCB) | y | S | synthetic fragrance |
| anthraquinone | | | manufacturing, seed treatment, bird repellent |
| triclosan | y | S | disinfectant, antimicrobial |
| bisphenol A | | K | manufacturing resins, antioxidant, flame retardant |
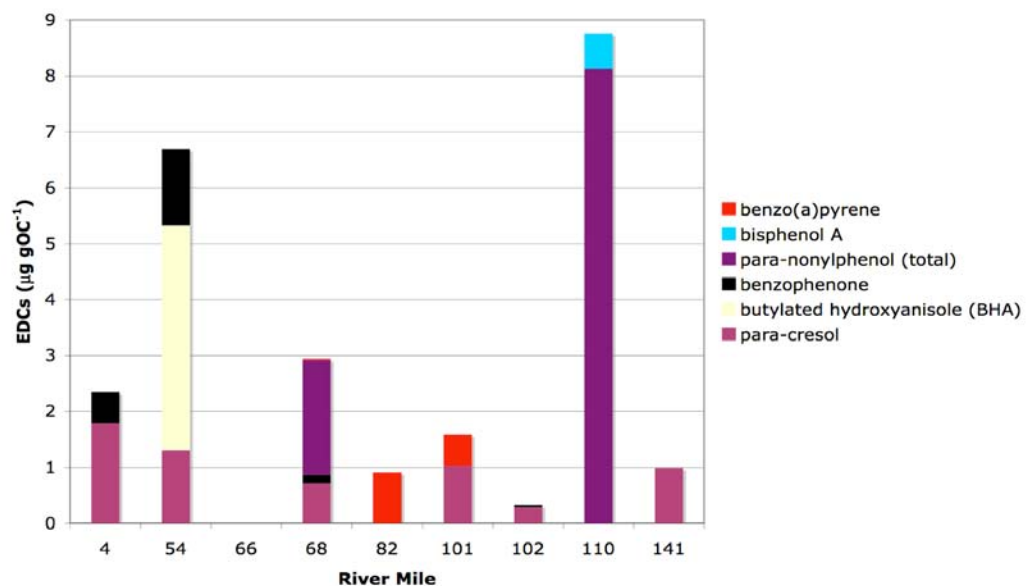


Figure 8. Endocrine disrupting compounds ($\mu$g gOC$^{-1}$) detected in sediments of the Columbia River sites displayed moving upstream from RM 4 at Point Adams to RM 141 at Warrendale. Number of compounds detected at each site: 4(2), 54(3), 66(0), 68(4), 82(1), 101(2), 102(2), 110(2), and 141(1).
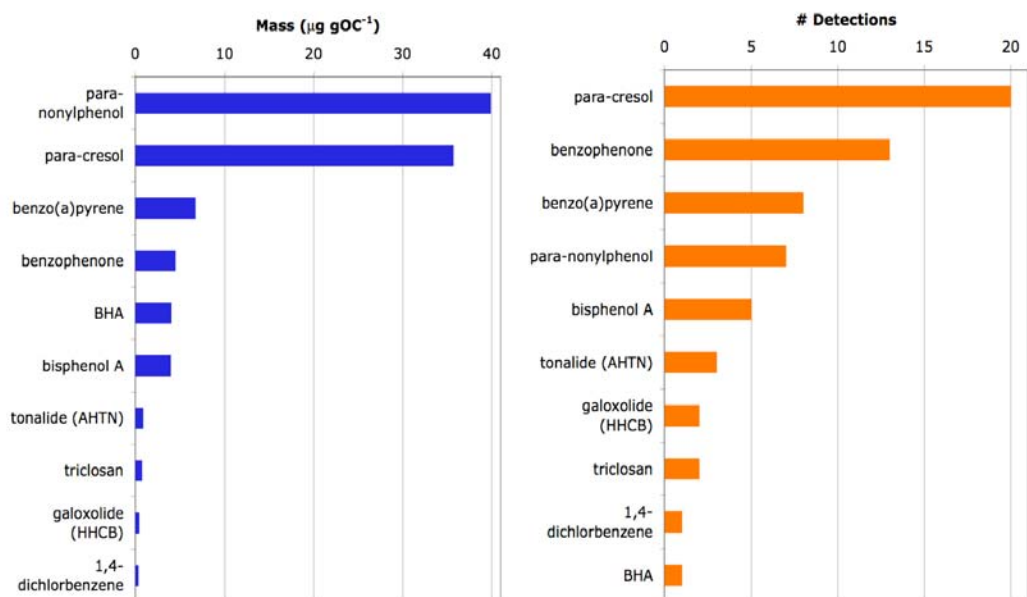
Figure 9. Known and suspected endocrine disrupting compounds ranked by total mass of compound measured at all sites (left) and by frequency of detection at sites (right).

**Implications and Future Work**

This work is the first documented case of the occurrence of a large suite of PPCPs and AWIs in the sediments of the Columbia River Basin. It is now known that these compounds are present in the system, and that they accumulate in the sediments. Several of the compounds detected are known to have detrimental impacts on aquatic life, although little is yet known about their sedimentary concentrations of concern. The effects of many compounds are not understood and require further study. Their presence in this ecosystem raises the possibility of biomagnification through the food web. A monitoring strategy is needed for these classes of emerging contaminants in this and other ecosystems, especially because their use and subsequent discharge into the environment is likely to increase into the future. The results of this reconnaissance work suggest that it would be valuable to monitor the mouths of tributaries and sites downstream of WWTFs. Lower order streams appear to pose greater exposure risks to juvenile salmonids and other wildlife, although, even on the mainstem Columbia, most sites had at least one EDC present in sediments. Future work is needed to determine effects levels for these compounds and relate sedimentary concentrations to water column concentrations and/or loads. Future efforts are also needed to understand routes of exposure and bioaccumulation pathways. Management implications include the need to work toward removing more of these compounds during wastewater treatment processes, possibly by increasing solids retention times or implementing reverse osmosis (e.g., Christen 2005).

**Acknowledgements**

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