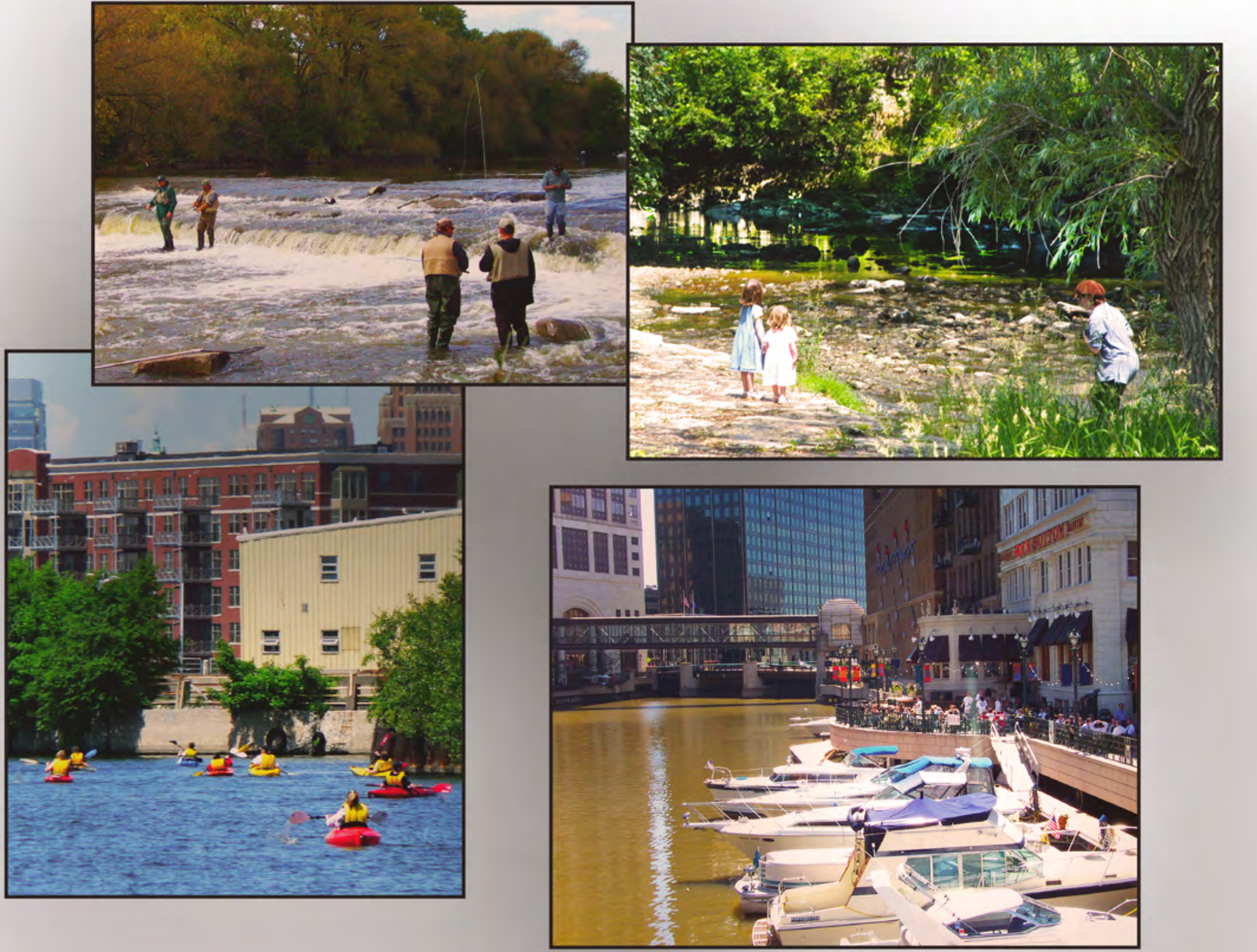
Prepared in cooperation with the
Milwaukee Metropolitan Sewerage District

Organic Waste Compounds in Streams: Occurrence and Aquatic Toxicity in Different Stream Compartments, Flow Regimes, and Land Uses in Southeast Wisconsin, 2006–9



Scientific Investigations Report 2013–5104

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

Cover: All photos provided by Milwaukee Metropolitan Sewerage District.

Upper left: Fishing the falls on the Milwaukee River at Estabrook Park, Milwaukee, Wis.

Lower left: Kayakers explore the Milwaukee Estuary, Milwaukee, Wis.

Upper right: Children play on the banks of the Menomonee River at Lime Kiln Park, Menomonee Falls, Wis.

Lower right: Diners enjoy the sunshine at an outdoor café along the Riverwalk, downtown Milwaukee, Wis.

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Contents

Figures

Tables

Conversion Factors

Inch/Pound to SI

Multiply	By	To obtain
	Length	
inch (in.)	2.54	centimeter (cm)
inch (in.)	25.4	millimeter (mm)
foot (ft)	0.305	meter (m)
	Area	
square mile (mi^2)	259	hectare (ha)
square mile (mi^2)	2.59	square kilometer (km^2)
	Flow rate	
cubic foot per second (ft^3/s)	0.028	cubic meter per second (m^3/s)

SI to Inch/Pound

Multiply	By	To obtain
	Length	
centimeter (cm)	0.394	inch (in.)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
liter (L)	2.113	pint (pt)
liter (L)	1.057	quart (qt)
liter (L)	0.264	gallon (gal)
liter (L)	61.02	cubic inch (in^3)

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

°F=(1.8×°C)+32

Horizontal coordinate information is referenced to the Wisconsin Transverse Mercator (WTM) Projection and the North American Datum of 1983 (NAD 83), with 1991 adjustment.

Concentrations of chemical constituents in water are given in micrograms per liter (µg/L).

Concentrations of chemical constituents in sediment are given in micrograms per kilogram (µg/kg).

Acronyms and Abbreviations

AqT	aquatic toxicity
CAS	Chemical Abstracts Service
C. dubia	*Ceriodaphnia dubia*
C. tentans	*Chironomus tentans*
DEET	N,N-diethyl-meta-toluamide
DEHP	Bis(2-ethylhexyl) phthalate
EDCs	endocrine-disrupting compounds/chemicals
EPA	U.S. Environmental Protection Agency
EWI	equal-width increment
GC/MS	gas chromatography/mass spectrometry
H. azteca	*Hyallela azteca*
HCW	Honey Creek at Wauwatosa
KRM	Kinnickinnic River at S. 11th Street, at Milwaukee
LCM	Lincoln Creek 47th Street at Milwaukee
LMM	Little Menomonee River at Milwaukee
MDL	minimum detection limit
MMF	Menomonee River at Menomonee Falls
MMSD	Milwaukee Metropolitan Sewerage District
MRC	Milwaukee River near Cedarburg
MRJ	Milwaukee River at the mouth at Jones Island
MRM	Milwaukee River at Milwaukee
MRW	Menomonee River at Wauwatosa
NP	4-Nonylphenol (sum of all isomers)
NWQL	National Water Quality Laboratory
OH-03	Middle Harbor Milwaukee Outer Harbor Site
OH-14	Middle Outside Harbor Breakwater Lake Site
OSM	Oak Creek at South Milwaukee
OWC	organic waste compound
PAH	polycyclic aromatic hydrocarbon
PCD	Parnell Creek near Dundee
QA/QC	quality assurance/quality control
RL	reporting limit
RRF	Root River near Franklin
RRG	Root River at Grange Avenue, at Greenfield
SEWRPC	Southeastern Wisconsin Regional Planning Commission
tPAH	total concentration of polycyclic aromatic hydrocarbons
TQ	toxicity quotients
UCW	Underwood Creek at Wauwatosa
USGS	U.S. Geological Survey
UV	ultraviolet
WCG	Willow Creek at Maple Road, near Germantown
WDNR	Wisconsin Department of Natural Resources
YFC	yeast/fish food/cereal leaves

Organic Waste Compounds in Streams: Occurrence and Aquatic Toxicity in Different Stream Compartments, Flow Regimes, and Land Uses in Southeast Wisconsin, 2006–9

By Austin K. Baldwin,[1] Steven R. Corsi,[1] Kevin D. Richards,[2] Steven W. Geis,[3] and Christopher Magruder[4]

Abstract

An assessment of organic chemicals and aquatic toxicity in streams located near Milwaukee, Wisconsin, indicated high potential for adverse impacts on aquatic organisms that could be related to organic waste compounds (OWCs). OWCs used in agriculture, industry, and households make their way into surface waters through runoff, leaking septic-conveyance systems, regulated and unregulated discharges, and combined sewage overflows, among other sources. Many of these compounds are toxic at elevated concentrations and (or) known to have endocrine-disrupting potential, and often they occur as complex mixtures. There is still much to be learned about the chronic exposure effects of these compounds on aquatic populations.

During 2006–9, the U.S. Geological Survey, in cooperation with the Milwaukee Metropolitan Sewerage District (MMSD), conducted a study to determine the occurrence and potential toxicity of OWCs in different stream compartments and flow regimes for streams in the Milwaukee area. Samples were collected at 17 sites and analyzed for a suite of 69 OWCs. Three types of stream compartments were represented: water column, streambed pore water, and streambed sediment. Water-column samples were subdivided by flow regime into stormflow and base-flow samples.

One or more compounds were detected in all 196 samples collected, and 64 of the 69 compounds were detected at least once. Base-flow samples had the lowest detection rates, with a median of 12 compounds detected per sample. Median detection rates for stormflow, pore-water, and sediment samples were more than double that of base-flow samples. Compounds with the highest detection rates include polycyclic aromatic hydrocarbons (PAHs), insecticides, herbicides, and dyes/pigments.

Elevated occurrence and concentrations of some compounds were detected in samples from urban sites, as compared with more rural sites, especially during stormflow conditions. These include the PAHs and the domestic wastewater-indicator compounds, among others. Urban runoff and storm-related leaks of sanitary sewers and (or) septic systems may be important sources of these and other compounds to the streams. The Kinnickinnic River, a highly urbanized site, had the highest detection rates and concentrations of compounds of all the sampled sites. The Milwaukee River near Cedarburg—one of the least urban sites—and the Outer Milwaukee Harbor site had the lowest detection rates and concentrations.

Aquatic-toxicity benchmarks were exceeded for 12 of the 25 compounds with known benchmarks. The compounds with the greatest benchmark exceedances were the PAHs, both in terms of exceedance frequency (up to 93 percent for some compounds in sediment samples) and magnitude (concentrations up to 1,024 times greater than the benchmark value). Other compounds with toxicity-benchmark exceedances include Bis(2-ethylhexyl) phthalate (a plasticizer), 2-Methylnapthalene (a component of fuel and oil), phenol (an antimicrobial disinfectant with diverse uses), and 4-Nonylphenol (sum of all isomers; a detergent metabolite, among other uses). Analyzed as a mixture, the suite of PAH compounds were found to be potentially toxic for most non-base-flow samples.

Bioassay tests were conducted on samples from 14 streams: *Ceriodaphnia dubia* in base-flow samples, *Ceriodaphnia dubia* and *Hyallela azteca* in pore-water samples, and *Hyallela azteca* and *Chironomus tentans* in sediment samples. The greatest adverse effect was observed in tests with *Chironomus tentans* from sediment samples. The weight of *Chironomus tentans* after exposure to sediments decreased with increased OWC concentrations. This was most evident in the relation between PAH results and *Chironomus tentans* bioassay results for the majority of samples; however, solvents and flame retardants appeared to be important for one site each. These results for PAHs were consistent with assessment of PAH potency factors for sediment, indicating that PAHs were likely to have adverse effects on aquatic organisms in many of the streams studied.

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Introduction

The occurrence and effects of organic waste compounds (OWCs) in streams has garnered increasing attention in recent years, both within the scientific community and in the popular press (Kolpin and others, 2002; Richardson and Ternes, 2005; Blue, 2012). OWCs are natural or synthetic chemicals derived from domestic, agricultural, and industrial wastewater sources. OWCs include fire retardants, fuel by-products, polycyclic aromatic hydrocarbons (PAHs), herbicides, antimicrobial disinfectants, and flavors and fragrances, among others. These compounds enter the environment through a number of pathways, including washing into streams from impervious surfaces and agricultural fields, industrial discharges into the air or water, leaching into the groundwater from unlined landfills, discharges from wastewater-treatment plants, combined sewer overflows, leaking septic systems, and leaking municipal sanitary and storm sewer systems (Kolpin and others, 2002; Gilliom and others, 2006; Phillips and others, 2012).

Numerous recent studies have focused on the health effects of OWCs on aquatic communities (Thorpe and others, 2001; U.S. Environmental Protection Agency, 2003; Barber and others, 2007; Vandenberg and others, 2012; Kemble and others, 2013). Although environmental concentrations tend to be low (generally, in the range of micrograms per liter or nanograms per liter), exposure is often chronic, spanning entire life cycles. Many of the OWCs sampled in this study are known or suspected endocrine-disrupting chemicals (EDCs), meaning they possess hormone-like activity that can disrupt normal endocrine function. EDCs have been shown to disrupt sexual development, brain development, and the immune system, among others (Vandenberg and others, 2012). In aquatic environments, EDCs and other OWCs usually occur as complex mixtures, which may have additive or synergistic effects (Sumpter and Jobling, 1995; Marinovich and others, 1996; Thorpe and others, 2001; U.S. Environmental Protection Agency, 2003; Vandenberg and others, 2012). Chronic, multigenerational exposure of low-concentration mixtures of OWCs may result in incremental and irreversible impacts to aquatic communities (Daughton and Ternes, 1999).

In 2006, the U.S. Geological Survey (USGS) and the Milwaukee Metropolitan Sewerage District (MMSD) began a cooperative study of OWCs in Milwaukee area streams and in the Milwaukee Harbor. The goal of the study was to provide an assessment of OWCs and toxicity in different stream compartment and flow regimes, including surface water during base flow and stormflow, streambed pore water, and streambed sediment. During 2006–9, 196 samples were collected from 17 sites: 14 stream sites, 2 harbor sites, and 1 stream/harbor transition site (table 1; fig. 1). Water samples were analyzed for 69 OWCs; sediment samples were analyzed for 57 OWCs. Additionally, water column, pore-water, and sediment samples from the 14 stream sites were analyzed for aquatic toxicity (AqT) using bioassays.

This study can assist watershed managers in prioritizing management and remediation efforts by describing OWC occurrences at different sites and in different stream compartments and flow regimes, and by identifying frequently occurring compounds and their potential toxicity to aquatic communities.

Purpose and Scope

This report presents results of an assessment of OWCs and AqT in different stream compartments (stream water, sediment pore water, and bed sediment) and flow regimes (stormflow and base flow) in aquatic ecosystems within the MMSD service area. Occurrence and concentrations of OWCs are analyzed by sampling site, stream compartment, and flow regime. AqT is assessed by comparing measured concentrations to benchmark values, and by bioassays. This study was part of the Watercourse Corridor Study—Phase III, a cooperative effort among the MMSD, WDNR, SEWRPC, USGS, and local universities. Data from this study builds on data collected during 2004–5 as part of the Watercourse Corridor Study—Phase II (Thomas and others, 2007).

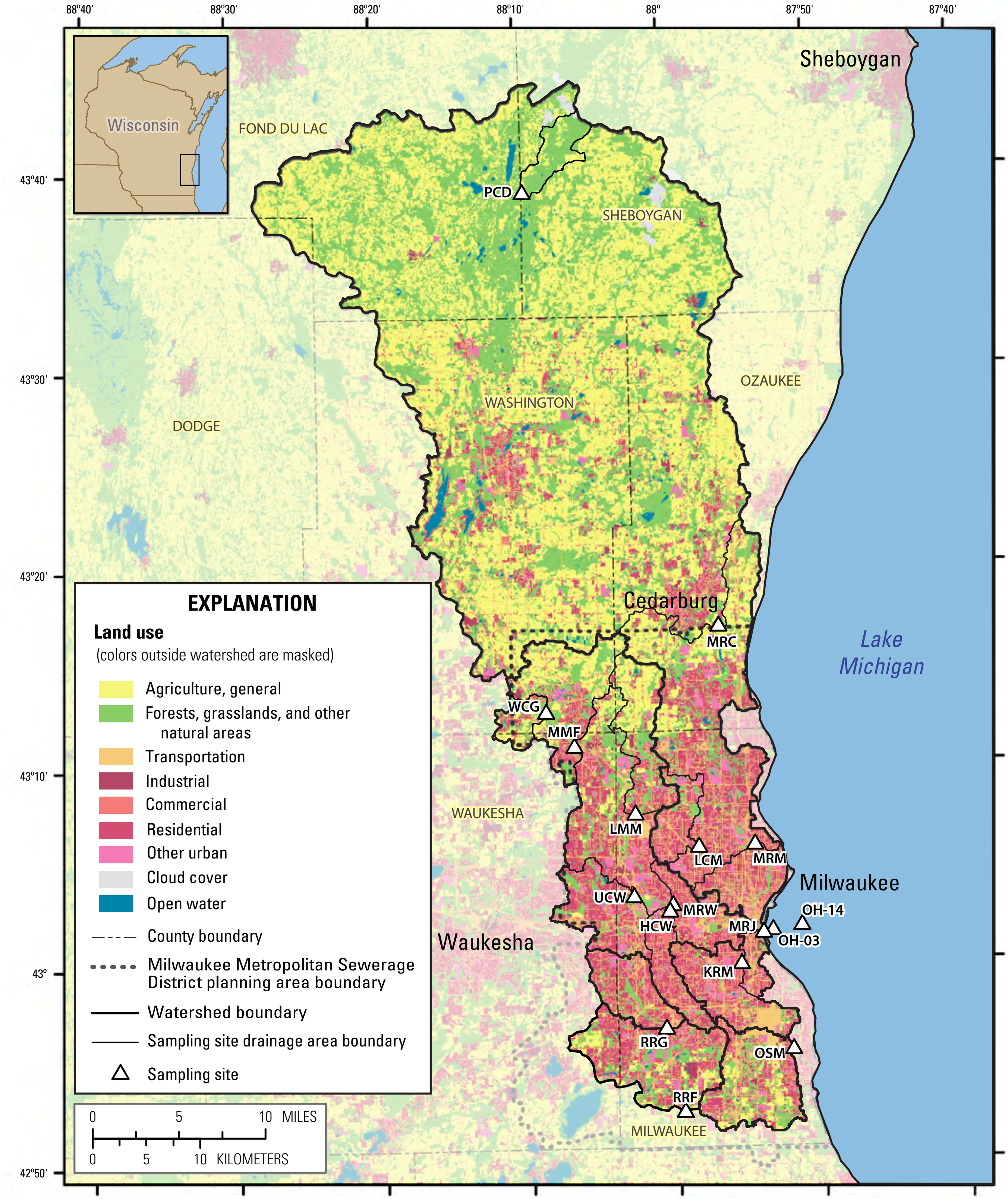
Description of Study Area

Most sampling sites were within the MMSD planning area. In total, 17 sites were sampled: 14 stream sites, 2 sites in the Milwaukee Harbor (Middle Harbor Milwaukee Outer Harbor Site (OH-03) and Middle Outside Harbor Breakwater Lake Site (OH-14)), and 1 stream/harbor transition site (Milwaukee River at the mouth at Jones Island (MRJ)). The stream/harbor transition site, MRJ, receives water from the Milwaukee, Kinnickinnic, and Menomonee River watersheds, as well as Lake Michigan. Basin-drainage areas of the stream sites (including MRJ) range from 9.56 to 872 square miles (mi^2), with annual mean discharges from 13.2 to 735 cubic feet per second (ft^3/s) (Scudder Eikenberry and others, 2010; U.S. Geological Survey, 2011). The sampled basins span a broad range of land uses and urban influence, with 10.4 to 96.0 percent urban land use. Of the harbor sites, OH-14 is located outside the break wall and OH-03 is located inside the break wall, approximately midway between the break wall and the mouth of the Milwaukee River.

Table 1. Site characteristics, land use, and types of samples collected, southeast Wisconsin, 2006–9.

[USGS, U.S. Geological Survey; mi^2, square mile; ft^3/s, cubic foot per second; natural areas include forests, grasslands, wetlands, and shrublands, among others; OWC, organic waste compound; AqT, aquatic toxicity, samples were collected one time, where indicated; --, not sampled; na, not available; land use percentages calculated from National Land Cover Database, 2001, and Southeastern Wisconsin Regional Planning Commission, 2000]

USGS site name	Site abbre-viation	USGS station number	Site characteristics and land use					Type of samples collected	Number of samples collected for OWCs			
			Drainage area (mi^2)	Mean annual discharge (ft^3/s)	Per-cent urban	Percent agricul-tural	Percent natural areas		Base flow	Storm-flow	Pore water	Sedi-ment
Streams sampled at low intensity												
Milwaukee River at Milwaukee	MRM	04087000	696	442	19	50	31	OWC, AqT	1	--	1	1
Root River near Franklin	RRF	04087220	49.2	44.8	68	11	21	OWC, AqT	1	--	1	1
Menomonee River at Menomonee Falls	MMF	04087030	34.7	31.5	35	38	27	OWC, AqT	1	--	1	1
Willow Creek at Maple Road, near Germantown	WCG	040870195	6.33	na	34	37	28	OWC, AqT	1	--	1	1
Parnell Creek near Dundee	PCD	04086175	9.35	na	3	26	71	OWC, AqT	1	--	1	1
Streams sampled at middle intensity												
Lincoln Creek 47th Street at Milwaukee	LCM	040869415	9.56	13.2	89	0	11	OWC, AqT	9	--	1	2
Little Menomonee River at Milwaukee	LMM	04087070	19.7	18	41	34	26	OWC, AqT	10	--	2	4
Underwood Creek at Wauwatosa	UCW	04087088	18.2	15.2	84	1	15	OWC, AqT	9	--	1	3
Honey Creek at Wauwatosa	HCW	04087119	10.3	15.2	96	0	4	OWC, AqT	9	--	1	3
Root River at Grange Avenue, at Greenfield	RRG	04087214	14.7	17.3	86	1	14	OWC, AqT	9	--	1	3
Streams sampled at high intensity												
Menomonee River at Wauwatosa	MRW	04087120	123	107	61	19	20	OWC, AqT	9	12	1	3
Kinnickinnic River at S. 11th Street, at Milwaukee	KRM	04087159	18.8	25.3	93	1	7	OWC, AqT	9	11	1	3
Oak Creek at South Milwaukee	OSM	04087204	25	23.9	58	18	24	OWC, AqT	9	10	1	3
Milwaukee River near Cedarburg	MRC	04086600	607	453	13	55	32	OWC, AqT	9	6	1	3
Stream/harbor transition and harbor sites												
Milwaukee River at the mouth at Jones Island	MRJ	04087170	872	727	30	43	28	OWC	9	--	--	--
Middle Harbor Milwaukee Outer Harbor Site	OH-03	430134087532900	na	na	na	na	na	OWC	8	--	--	--
Middle Outside Harbor Breakwater Lake Site	OH-14	430138087514800	na	na	na	na	na	OWC	8	--	--	--



Base composited from Southeastern Wisconsin Regional Planning Commission digital land use inventory, 1:4,800, 2000; Wisconsin Department of Natural Resources WISCLAND land cover, 1:40,000, 1998; National Hydrologic Dataset Waterbody, 1:24,000, 2010; Wisconsin Transverse Mercator Projection, referenced to North American Datum of 1983, 1991 adjustment.

Figure 1. Location of sampling sites, drainage basins, and land-use areas used in the study. (Sampling site abbreviations are defined in table 1.)

Water and Sediment Sampling: Design and Collection

Whole-water and sediment samples were collected for analysis of OWCs as well as for AqT bioassays. Sampling for OWCs was conducted during 2007–9, and was a collaborative effort between USGS and MMSD field personnel. Sampling for AqT was conducted during May 2006 and July–August 2007. Table 1 outlines the types and numbers of samples collected at each of the 17 sites.

Sampling Design

The 17 sites were sampled at different intensities, and at some sites only certain sample types were collected (table 1). Of the 14 stream sites, 5 were sampled at low intensity: 1 base-flow water column, 1 streambed pore-water, and 1 streambed-sediment sample per site, all analyzed for both OWCs and AqT. These sites were Milwaukee River at Milwaukee (MRM), Root River near Franklin (RRF), Menomonee River at Menomonee Falls (MMF), Willow Creek at Maple Road, near Germantown (WCG), and Parnell Creek near Dundee (PCD). Another five of the stream sites were sampled at middle intensity, with the same sampling schedule as the low-intensity sites but with additional OWC samples of streambed sediment and base-flow water column. The middle-intensity stream sites were Lincoln Creek 47th Street at Milwaukee (LCM); Little Menomonee River at Milwaukee (LMM); Underwood Creek at Wauwatosa (UCW); Honey Creek at Wauwatosa (HCW); and Root River at Grange Avenue, at Greenfield (RRG). The 4 high-intensity stream sites—Menomonee River at Wauwatosa (MRW); Kinnickinnic River at South 11th Street, at Milwaukee (KRM); Oak Creek at South Milwaukee (OSM); and Milwaukee River near Cedarburg (MRC)—followed the same sampling schedule as the middle-intensity sites, but with the addition of 6–12 stormflow samples collected for OWC analysis. At the two harbor sites (OH03 and OH14) and the stream/harbor transition site (MRJ), samples were collected from the water column only, and analysis was limited to OWCs.

All streambed-sediment and streambed pore-water samples were collected during spring and summer months, as were the 1 to 2 base-flow water-column samples analyzed for both OWCs and AqT. At the middle- and high-intensity stream sites, base-flow water-column samples for OWCs were collected quarterly, for a total of 9 to 10 samples over a 2-year period. At the high-intensity stream sites, stormflow water-column OWC samples were collected based on availability of runoff events rather than on a set schedule. Runoff events were less frequent at the more rural site (MRC) allowing collection of fewer stormflow samples than at the more urban sites. A total of 6–12 stormflow samples were collected per high-intensity sampling site.

Sample-Collection Methods

In accordance with USGS protocols (U.S. Geological Survey, variously dated), samples were collected and processed in a manner consistent with minimal contamination of organic compounds. Glass, Teflon, or stainless-steel equipment was used during sample collection and processing, whenever possible. Water samples were neither filtered nor acidified. Samples were sent to the USGS National Water Quality Laboratory (NWQL) for analysis of OWCs and to the Wisconsin State Laboratory of Hygiene for AqT bioassays.

Base-flow water-column samples from stream sites were collected using the equal-width-increment (EWI) method (Edwards and Glysson, 1999). Approximately 10 subsamples were collected at equal widths across the stream cross section using a DH-81 with a Teflon nozzle and a 1liter (L) Teflon bottle. Subsamples were composited in a 14-L Teflon churn and then churned into a 1L baked amber-glass bottle for analysis. At the three harbor and stream/harbor transition sites, samples were collected using a single dip of the baked amber-glass bottle.

Stormflow samples were collected using refrigerated automated samplers with Teflon-lined sample lines and up to four 10-L glass bottles. The automated samplers were controlled by dataloggers programmed to collect flow-weighted composites throughout the storm-runoff period. The 10-L bottles were composited in a Teflon churn and then the sample was churned into a 1-L baked amber-glass bottle for analysis.

Streambed pore water was collected by inserting a vented Teflon probe 10–15 centimeters (cm) into the sediment and using a peristaltic pump to pull the surrounding pore water out through the vents, as described in Lutz and others (2008). An acrylic disc was attached to the sampler, and the sampler was inserted to the point where the disc made contact with the top of the bed sediment. This disc helped to minimize intrusion into the pore water from the water column during sampling. At each site, subsamples of approximately 2 L of pore water were collected at three to five locations, composited in a 14L Teflon churn, and then churned into a 1-L baked amber-glass bottle.

Streambed sediment was collected by inserting a 2-inch (in.) diameter Teflon core approximately 2 in. into the sediment, inserting a stiff Teflon sheet under the bottom of the core, and gently lifting the sheet and core out of the water. Sediment was collected at three to five locations per site, composited in a stainless-steel bucket, and mixed with a stainless-steel spoon. Fine sediments (silts) were targeted at all sites, when available. Sediment samples for OWC analysis were put through a stainless-steel sieve using a stainless-steel spoon to remove any gravel or large organic debris. The AqT samples were not sieved. After being composited, the sediment samples were put in baked amber-glass bottles for analysis.

Analytical Methods

Following collection, whole-water and sediment samples were shipped on ice overnight to the USGS NWQL for analysis of OWCs (analysis schedule 4433 for water and 5433 for sediment samples), and delivered to the Wisconsin State Laboratory of Hygiene for bioassay toxicity tests.

Organic Waste Compounds

Whole-water and sediment samples were analyzed at the NWQL for 69 and 57 compounds, respectively (table 2, in back of report). The compounds were chosen because they are good indicators of domestic or industrial wastewater, and (or) because they are associated with human or environmental health risks (Kolpin and others, 2002; Zaugg and others, 2007). Approximately one-third of the compounds are known or suspected endocrine disruptors (The Endocrine Disruption Exchange, Inc., 2011).

Compounds in water samples were extracted using continuous liquid-liquid extraction and methylene chloride solvent, then determined by capillary-column gas chromatography/mass spectrometry (GC/MS; Zaugg and others, 2007). Compounds in sediment samples were extracted using a pressurized solvent-extraction system, then determined by capillary-column GC/MS (Burkhardt and others, 2006).

The NWQL spikes of these compounds in reagent and surface water, and in sand, river sediment, and topsoil, show variable recovery performance. Percent recovery and variability data for each compound are listed in Zaugg and others (2007) and Burkhardt and others (2006).

For most compounds, the NWQL sets the reporting limit (RL) at 2–10 times higher than the minimum detection limit (MDL; table 2; Oblinger Childress and others, 1999). The RLs may be elevated because of interferences, especially in sediment samples. Reasons for interferences are discussed in Zaugg and others (2007) and Burkhardt and others (2006). Measured concentrations below the RL are still reported by the lab but are assigned an “estimated” (“E”) qualifier. The “E” qualifier also may be assigned if the concentration is below the lowest calibration standard [(usually 0.2 micrograms per liter (µg/L)], or if there are matrix interferences. Further, some compounds have been permanently assigned an “E” qualifier because historically quality-assurance criteria have not been met. In terms of qualitative identification of a compound (detection as opposed to nondetection), results with the “E” qualifier generally can be considered reliable, with reliability decreasing as the measured concentration nears or falls below the MDL (Oblinger Childress and others, 1999; Zaugg and others, 2007). In this report, concentrations reported with the “E” qualifier are considered detections.

Bioassays for Toxicity Testing in Sediment

Sediments were tested for toxicity along with formulated laboratory sediment as the laboratory control. Solid-phase sediment-toxicity tests were performed using the amphipod, *Hyalella azteca*, and the larval stage of the midge, *Chironomus tentans* (*C. tentans*). These two organisms, which burrow and come into direct contact with the sediments, are recommended by the U.S. Environmental Protection Agency (EPA) for use in sediment-toxicity testing (U.S. Environmental Protection Agency, 1994).

Sediments were stored at 4 degrees Celsius (°C) in the dark. Prior to testing, the sediment was thoroughly homogenized (by mixing) using a large stainless-steel spoon. A synthetic laboratory-control sediment was prepared containing 77 percent sand, 17 percent clay, 5 percent organic matter (composted cow manure), and 1 percent buffer (calcium carbonate). Homogenized sediment was placed in test beakers with dechlorinated tap water, with a sediment to overlying water ratio of 1:1.75. After allowing the sediments to settle for 24 hours, organisms were randomly added to the test beakers. Test beakers were randomly placed in a walk-in environmental chamber at 23 ± 1°C with a 16:8-hour light:dark cycle. Test conditions are summarized in appendix 1.

Juvenile *Hyallela azteca* (*H. azteca*) and larval *C. tentans* were ordered from Aquatic Research Organisms (Hampton, New Hampshire) to be the appropriate age at test initiation (7–14 days old for *H. azteca* and 9 days old for *C. tentans*). Ten individuals were randomly placed in each 400 milliliter (mL) polypropylene test beaker with eight replicates per sediment site. Overlying water was replaced twice daily, and organisms were fed daily (1.5 mL yeast/fish food/cereal leaves (YFC) leaves for *H. azteca* and 4 milligrams Tetramin® flake fish food for *C. tentans*). Dissolved oxygen, pH, and temperature of the overlying water were recorded daily. On day 10, the organisms were recovered from the sediment to determine the number of survivors in each replicate. The organisms then were subjected to ultraviolet (UV) light for a period of 2 hours following the procedure for PAH photoactivation described by Ankley and others (1994). This procedure at times can provide information on additional adverse impacts owing to PAH photoactivation in the organisms. Surviving individuals were transferred to 100 mL beakers containing 30 mL of dechlorinated tap water. The beakers were set below the UV bulbs (UVA-340, Q-Panel Lab Products, Cleveland, Ohio, USA) so that the bottom of each beaker was approximately 8.5 cm from the bottom of the UV bulb. After 2 hours of UV exposure, the beakers were removed to record post-UV survival. One replicate from each treatment was kept away from the UV light during the 2-hour exposure to serve as controls. Both surviving and dead organisms then were removed from the beakers and placed in tared aluminum weigh pans to determine dry weight. Pans were placed in an oven for a minimum of 2 hours at 110°C prior to weighing. *C. tentans* then were ashed at 550°C overnight to determine ash-free dry weight. The weight of inorganic material (ash weight) was subtracted from the total dry weight to determine weight of organic matter. Survival and ash-free dry weight were used as final endpoints for *C. tentans* test. Ash-free dry weight is referred to as “weight” throughout this report. Survival and dry weight were used as final endpoints for *H. azteca* tests.

Bioassays for Toxicity Testing in Water

Surface- and pore-water samples were stored at 4°C upon delivery from the field. Aliquots were removed daily to prepare test solutions. Samples were warmed in a water bath to the appropriate test temperature. Toxicity tests were performed without dilution using the *C. dubia* chronic test (U.S. Environmental Protection Agency, 2002). Test-chamber conditions were 25°C with a 16:8-hour light:dark cycle. The organisms were fed with each daily renewal a combination of YFC and the green algae *Selenastrum capricornutum*. Production of young was recorded daily, and the tests were terminated after 80 percent of controls released their third brood (6 to 7 days). Survival and production of young (reproduction) were used as final endpoints for this test.

Additionally, pore-water samples were assayed using a 48-hour *H. azteca* acute test. Each treatment consisted of 10 replicates, each containing 2 *H. azteca* organisms in 30 mL of water and 1 square centimeter (cm^2) of Nitex® mesh for substrate. Organisms were fed YFC daily. Survival was recorded at the end of the 48-hour exposure and was used as the final endpoint for this assay.

Quality Assurance/Quality Control

Quality-assurance/quality-control (QA/QC) samples were collected during both laboratory and field activities to assess data quality and identify possible contamination. The NWQL QA/QC samples included blanks, reagent spikes, and surrogates. A blank consists of analyte-free water that is analyzed alongside environmental samples to ensure there is no contamination from the lab. A reagent spike is water with a known concentration of the analytes being measured. A reagent spike also is analyzed alongside environmental samples to measure the performance of the analytical methods. At least one laboratory blank and reagent spike accompany each set of 18 or fewer environmental samples being analyzed at the NWQL (Zaugg and others, 2007).

Surrogates are compounds that are physically and (or) chemically similar to the other compounds being measured. The NWQL adds surrogate compounds to each OWC sample prior to analysis. Surrogate recovery rates, reported as percentages, are used to measure the accuracy of the analytical method and identify sample-processing problems and matrix effects. The four surrogates added to water samples were bisphenol A-d3, caffeine-13C, decafluorobiphenyl, and fluoranthene-d10, with median recovery rates of 84, 75, 60, and 74 percent, respectively. Overall, the recovery rates in water samples ranged from 13 to 162 percent, with a median of 71 and standard deviation of 21. The same surrogates were added to OWC sediment samples, minus caffeine-13C. Median recovery rates in sediment samples were 83 for bisphenol A-d3, 23 for decafluorobiphenyl, and 82 for fluoranthene-d10. Overall, the recovery rates in sediment samples ranged from 0 to 266 percent, with a median of 60 and standard deviation of 44.

Field QA/QC activities consisted of one replicate water-column sample collected for OWCs during base-flow conditions at MRC. A replicate sample is collected concurrently with an environmental sample to determine the variability in sample-collection and processing procedures. Of the 69 comparisons (compounds) between the 2 samples, 10 compounds were detected in both samples; for 9 of those compounds, the detections were below 0.2 μg/L. The tenth compound, cholesterol, had estimated detections of 0.45 and 0.89 μg/L, for a relative percent difference of 66. For 22 compounds, there were estimated detections in 1 sample but not the other, but in only 5 of those were the detections greater than 0.2 μg/L, and those were still below the reporting level as set by the other sample.

No OWC field blanks were collected for this study. The lack of field blanks decreases confidence in the concentrations detected in the environmental samples, especially detected concentrations at or below the RL. Other studies sampling OWCs have reported low concentration detections of a number of compounds in field blanks, including naphthalene, phenanthrene, benzo[a]pyrene, benzophenone, bisphenol A, methyl salicylate, N,N-diethyl-meta-toluamide (DEET), isophorone, phenol, 4-Nonylphenol (sum of all isomers; hereafter NP), triphenyl phosphate, and Tris(2-chloroethyl) phosphate (Phillips and others, 2005; Phillips and Chalmers, 2009; Wilkison and others, 2002; Kingsbury and others, 2008). Three of these compounds stand out as having fairly high (greater than 25 percent) detection frequencies in base-flow samples in this study: phenanthrene (51 percent), DEET (58 percent), and Tris (2-chloroethyl) phosphate (42 percent). Therefore, low-concentration detections of these compounds, as well as others, should be cautiously interpreted.

Data Analysis

Organic Waste Compounds

Individual OWC compounds were aggregated into 15 classes: antioxidants, dyes and pigments, fire retardants, PAHs, plasticizers, fuels, solvents, herbicides, insecticides, antimicrobial disinfectants, detergent metabolites, flavors and fragrances, human drugs (nonprescription), sterols, and miscellaneous (table 2). The classes are consistent with those used in the Watercourse Corridor Study—Phase II (Thomas and others, 2007), and were originally based on aggregations by Sullivan and others (2005).

A number of the analyses in this report are based on whether or not compounds were detected in the environmental samples. Results reported as "estimated" were considered detections, regardless of their concentration. Detection frequencies were analyzed for individual compounds as well as for classes of compounds. For individual compounds, detection frequencies were computed as the percentage of samples with detections of those compounds. For classes, detection frequencies were determined by whether any one of the compounds in a class was detected in a given sample. For example, if any one of the six compounds in the PAH class were detected in a sample, that would be considered a detection of the PAH class.

Analyses in this study use estimated concentrations at their reported value, whether above or below the RL for that compound. Median, mean, and maximum detected concentrations were computed for individual compounds using detected concentrations only. Class concentrations were computed for each sample by summing the detected concentrations within each class, using zeros for compounds without detections. Total sample concentrations were computed by summing all detected concentrations, using zeros for compounds without detections. Inadequate sample numbers prevented the use of nondetect-estimation methods such as those described by Helsel (2012).

To assess how the total concentration of each compound class varied by sample type, total class concentrations were computed for each sample using detected concentrations only, with zeros for nondetections. The total class concentrations then were averaged by site to give equal weight to each site, as different sites had different numbers of samples.

A subset of 20 compounds considered to be domestic-wastewater indicators was analyzed to assess contributions from that source. These are compounds commonly associated with sanitary-sewer and septic-system leaks and overflows, though other sources are possible. These compounds are indicated in table 2 and include all of the fire retardants and detergent metabolites, most of the flavors/fragrances, and the antimicrobial disinfectant triclosan. Total concentrations of these 20 domestic-wastewater indicator compounds were computed by summing the detected concentrations in each sample, using zeros for nondetections.

The AqT criteria and guidelines (hereafter cumulatively referred to as benchmarks) for OWCs were compiled and compared to concentrations in environmental samples. For the majority of the compounds in this study, AqT benchmarks have not been established. Benchmarks from the EPA and other sources, such as the Canadian Council of Ministers of the Environment were found for 25 of the compounds sampled.

Toxicity quotients (TQ) were computed for individual OWC compounds with AqT benchmarks. The TQ provides a unitless measure of the scale of the benchmark exceedance, and is computed by dividing the measured concentration of a compound by the lowest of the compound's benchmarks. Mean toxicity quotients (TQ_{mean}) then were computed for each compound, using samples with detected concentrations only.

The potential combined toxicity of the six PAH compounds was assessed using an equilibrium-partitioning method developed by the EPA (U.S. Environmental Protection Agency, 2003; 2010). Using the measured concentrations and compound-specific multipliers and divisors, acute- and chronic-potency ratios are computed for each PAH compound in a sample. The potency ratios then are summed for an acute- and chronic-potency factor, which if greater than 1.0 indicates potential toxicity to aquatic organisms. Because only 6 PAH compounds were analyzed in this study, rather than the 27 compounds used in the EPA method, and because the potency ratios for each compound are additive, the resulting potency factors likely are conservative. Based on a previous study, which analyzed 17 PAHs in sediment at these same streams (Corsi and others, 2011), it is estimated that the potency factors presented in this study likely would be 1.4 to 1.6 times greater had the same 17 PAHs been sampled.

The OWC-focused analyses in this report omit samples from the five low-intensity sampling sites (MRM, RRF, MMF, WCG, and PCD) because of the low sample numbers at those sites (table 1). This includes all analyses discussed in the section titled "Occurrence of OWCs." These sites are included in analyses focused on the AqT bioassays, in the section titled "Adverse Impacts on Aquatic Life."

Toxicity in Stream Compartments

Resulting toxicity endpoints were normalized by the laboratory-control results and presented as a percent of laboratory control. Standard errors of replicates are presented on all graphs as a representation of uncertainty in bioassay sample results. A specific threshold of adverse effect is difficult to define owing to many factors that influence aquatic organisms. An adverse effect is likely if the result is below 50 percent; if the value falls between 100 and 50 percent, the likelihood of an adverse effect increases with a decreasing value. Examination of the range of the error bars on the graphs also can provide information on the likelihood of differences in sample results from the laboratory control (overlapping with 100 percent) and from other samples (overlapping with corresponding-error bars). For sediment-toxicity results, likely adverse effects were observed, so further analysis was done to explore association with the corresponding OWC-sample results. These sediment OWC-sample results were aggregated by chemical class as previously discussed. Concentrations could not be compared directly among categories for association to the AqT data because consistent toxicity-benchmark data were not available from comparable laboratory testing for the various OWC compounds, and the actual concentration levels that could possibly affect AqT in different categories likely are very different. As an attempt to address this inconsistency, the sites were given a ranking from lowest to highest concentration for each OWC category that was considered a potential contributor to toxicity. This included the following categories: detergent metabolites, fire retardants, fuel, insecticides, PAHs, and solvents. Rank summations were computed for all one, two, and three class combinations of these rankings. Scatter plots and Pearson correlation coefficients of the rank summations as compared to *C. tentans* weight were generated for final analysis. For example, when considering three of the categories to explain bioassay results, the rankings for the three categories were summed for each site giving a potential rank-sum from 3 to 42. Lacking consistent toxicity-benchmark data among different categories precluded more sophisticated and relevant analysis such as a summation of toxicity units (ratio of sample concentrations to toxicity-benchmark concentrations).

Occurrence of Organic Waste Compounds

At least 1 compound was detected in all 196 samples collected with as many as 41 of the 69 compounds detected in 1 of the stormflow samples (table 3). As a group, the base-flow samples had the lowest detection rate, with a median of 12 compounds detected per sample. The stormflow and pore-water samples had medians of 26 and 28 detections per sample, respectively. The sediment samples had a median of 22 detections per sample but were tested for only 57 compounds instead of 69. Figure 2 shows the percent of compounds detected for samples collected at the middle- and high-intensity sampling sites.

Detection Frequencies of Classes of OWCs

Of the 69 compounds analyzed, 64 were detected in at least 1 sample (all sample types combined), and all 15 of the classes were detected [(tables 4a, 4b, 5a, and 5b (in back of report)]. The five compounds not detected in any sample are all from different classes.

The base-flow samples had class-detection frequencies from 25 to 75 percent for all but two classes (fig. 3). The two classes with base-flow detection frequencies above 75 percent were PAHs (80 percent) and herbicides (88 percent). None of the classes had base-flow detection frequencies below 25 percent.

At the four high-intensity sampling sites—MRC, MRW, OSM, and KRM—class-detection frequencies in stormflow samples averaged 1.3 to 2.0 times higher than those in base-flow samples. Eleven of the 15 classes had stormflow-detection frequencies above 75 percent, and 5 classes (dyes/pigments, PAHs, solvents, herbicides, and insecticides) had stormflow-detection frequencies above 90 percent. The only class with a stormflow-detection frequency below 25 percent was the miscellaneous class (fig. 3). These results indicate that urban storm water is an important source of OWCs to Milwaukee area streams.

The pore-water samples, which were collected during base-flow conditions, had higher detection frequencies than base-flow samples for all classes except human drugs. The three compounds in the human-drug class were not detected in pore-water samples. Ten of the 15 classes had pore-water detection frequencies above 75 percent, and 7 classes (antioxidants, dyes/pigments, PAHs, fuels, herbicides, insecticides, and flavors/fragrances) had pore-water detection frequencies above 90 percent. Human drugs was the only class with a pore-water detection frequency below 25 percent.

None of the classes had sediment-detection frequencies in the range of 25 to 75 percent, a notably different occurrence distribution from the base-flow samples. Ten of the 15 classes had sediment-detection frequencies of 75 percent or above, and 7 classes (dyes/pigments, PAHs, fuels, insecticides, antimicrobial disinfectants, flavors/fragrances, and sterols) had detection frequencies above 90 percent. Five of the classes had sediment-detection frequencies below 25 percent, and two of those (herbicides and nonprescription human drugs) were not detected in sediment samples.

Table 3. Detection statistics for organic waste compounds, by sample type, all sites combined, southeast Wisconsin, 2006–9.

Sample type	Number of samples	Number of compounds analyzed	Minimum number of compounds detected, per sample	Median number of compounds detected, per sample	Maximum number of compounds detected, per sample
Base flow	107	69	1	12	37
Stormflow	39	69	11	26	41
Pore water	10	69	18	28	37
Sediment	28	57	7	22	28

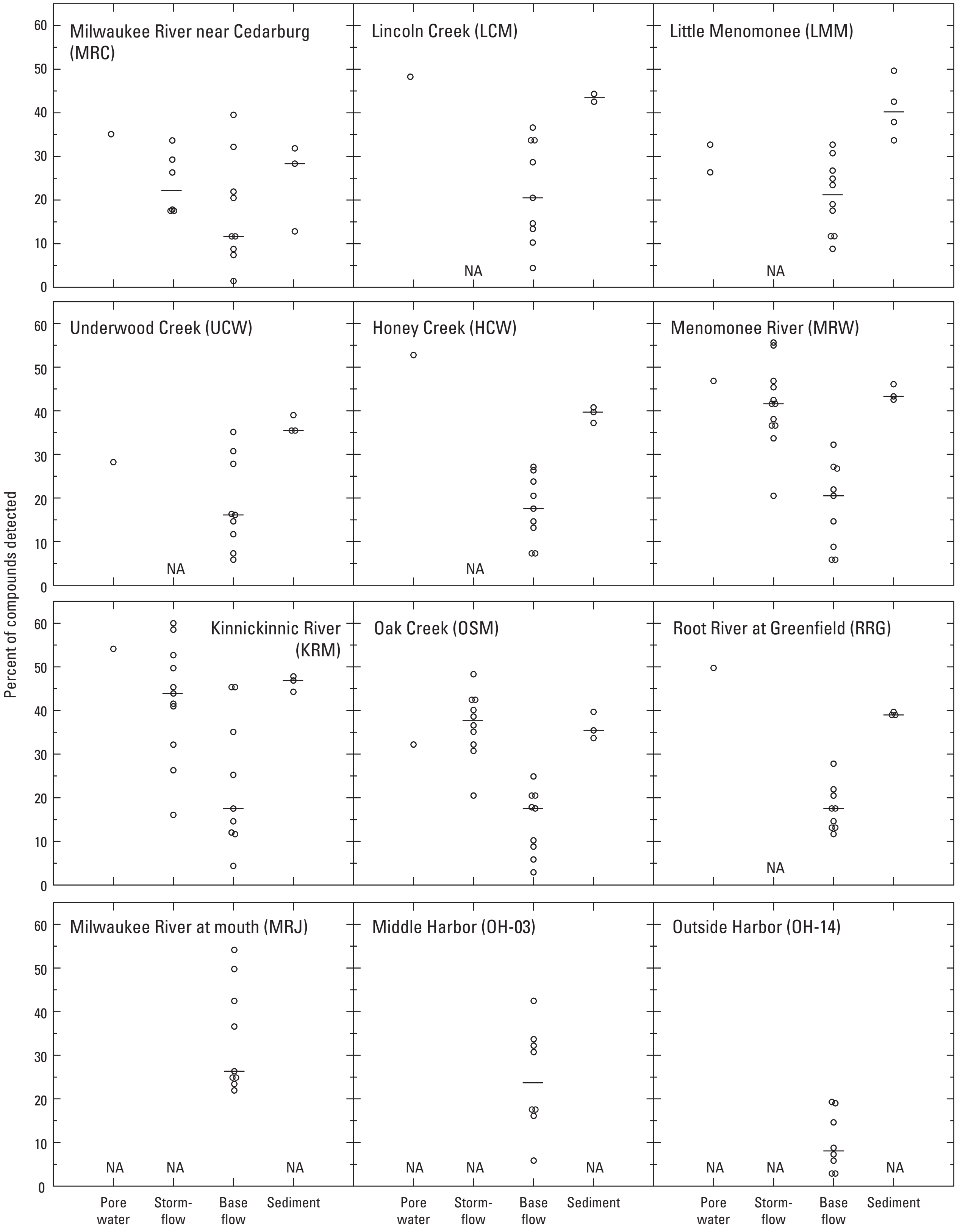


Figure 2. Percent of compounds detected per sample, organized by site and sample type. (Site abbreviations are defined in table 1; horizontal lines mark medians; NA, no samples collected)

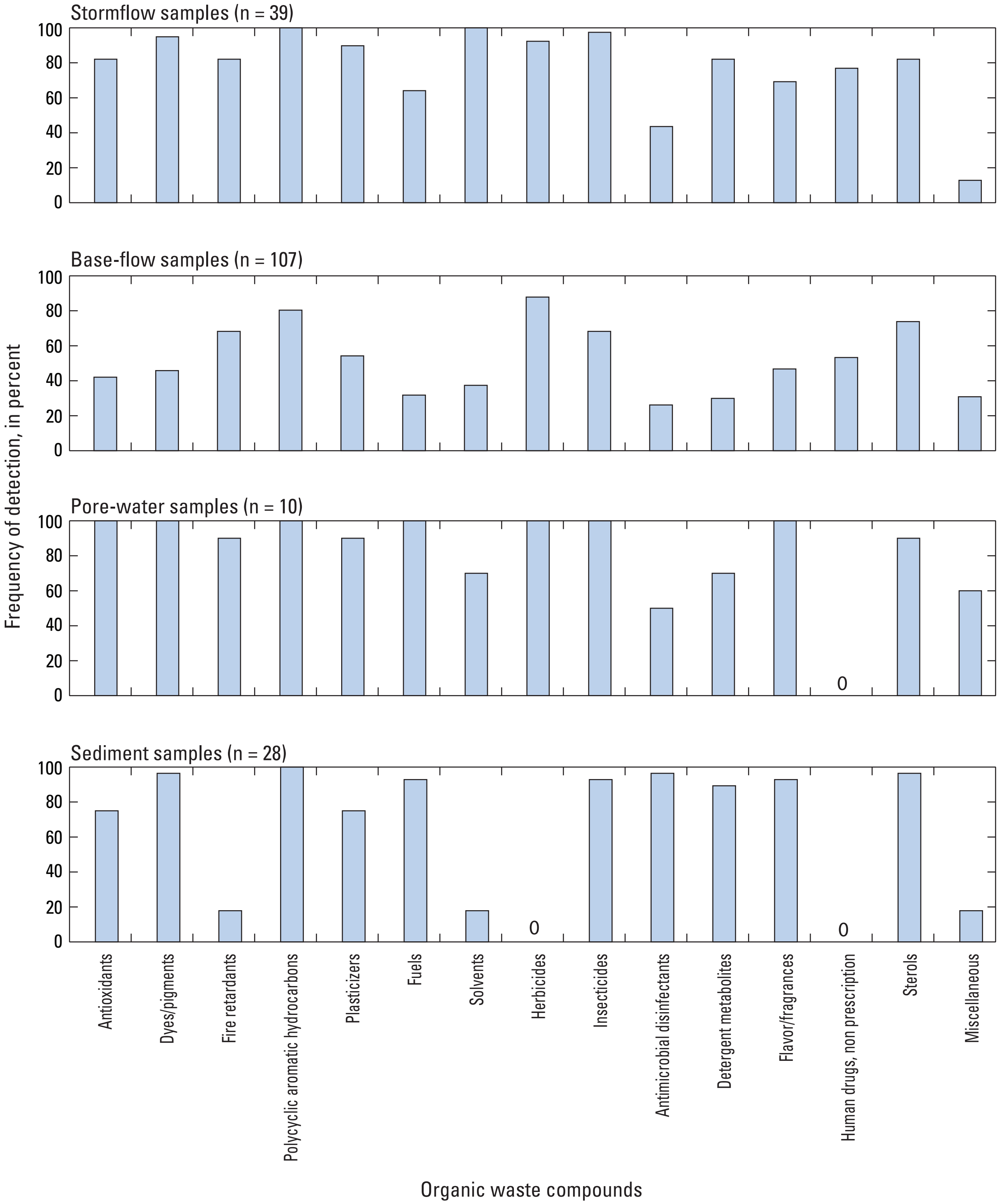


Figure 3. Frequency of detection of organic waste compounds, by class, for each sample type. (n, number of samples; 0, no detections)

Several of the classes with low sediment-detection frequencies had high-detection frequencies in the water samples, especially herbicides and fire retardants. The one class in which the sediment samples had a markedly higher-detection frequency than the water samples was the antimicrobial disinfectants, with detection frequencies of 96 percent in sediment samples and 50 percent or below in water samples. The high-detection frequency of antimicrobial disinfectants in sediment samples can be attributed to the wood preservative p-Cresol. The source of p-Cresol may be creosote-treated railroad ties and guardrail posts (Thomas, 2009).

The PAHs were the most frequently detected class overall, occurring in 100 percent of the stormflow, pore-water, and sediment samples, and in 80 percent of the base-flow samples. In the stormflow, pore-water, and sediment samples, all six of the PAH compounds had detection frequencies above 60 percent, and four had detection frequencies ranging from 90 to 100 percent. Overall, the miscellaneous class was the least-frequently detected.

Detection Frequencies of Individual OWCs

Detection frequencies for some classes were dominated by only one or two of the compounds within that class. Two of the three compounds in the antioxidants class had low-detection frequencies (0 to 40 percent), but the third compound, Bisphenol A, was detected in more than 75 percent of all stormflow, pore-water, and sediment samples (tables 4a, 4b, 5a, and 5b). Detections in the plasticizers class (five compounds) were dominantly DEHP (sediment samples) and Tris(2-chloroethyl) phosphate (all water-sample types). Four of the six compounds in the insecticides class had detection frequencies below 20 percent; the high-detection frequency of insecticides can be attributed to Carbazole and, to a lesser extent, DEET. The detergent metabolite-class detections were mostly NP (4-Nonylphenol, also used in pesticide and asphalt formulations) and, to a lesser extent, 4-Cumylphenol. In the nonprescription human-drug class, base-flow and stormflow-detection frequencies were mostly caffeine.

For some of the classes, most or all of the compounds were detected at high frequencies. All six of the PAH compounds (anthracene, benzo[a]pyrene, fluoranthene, naphthalene, phenanthrene, and pyrene) had very high-detection frequencies in stormflow, pore-water, and sediment samples, and three of the six had high-detection frequencies in base-flow samples (tables 4a, 4b, 5a, and 5b). Three of the four compounds in the fuels class (1-Methylnapthalene, 2-Methylnapthalene, and 2,6Dimethylnapthalene) were detected in the majority of stormflow, pore-water, and sediment samples. Detection frequencies for all compounds and sample types are listed in tables 4a, 4b, 5a, and 5b.

Concentrations of OWCs

The median detected concentrations of most compounds were higher in stormflow and pore-water samples than in base-flow samples. Median concentrations in stormflow samples were greater than those in base-flow samples by a factor of 2 or more for 23 compounds and by a factor of 10 or more for 5 compounds (fluoranthene, phenanthrene, pyrene, isophorone, and acetophenone; tables 4a, and 4b), indicating that urban runoff is likely a source of many of the compounds to the stream. Conversely, median concentrations in base-flow samples were greater than those in stormflow samples by a factor of two or more for three compounds: dichlorvos, HHCB, and 1,4-Dichlorobenzene. Median pore-water concentrations were comparable to stormflow concentrations for most compounds.

Five compounds had median detected concentrations greater than 0.5 µg/L in base-flow samples: the herbicide pentachlorophenol, the detergent metabolite 4-Nonylphenol diethoxylate, and the sterols beta-sitosterol, beta-stigmastanol, and cholesterol (tables 4a, and 4b). The highest detected concentration of the base-flow samples was 115 µg/L (estimated), for the non-crop herbicide, dichlorophenyl isocyanate, at LMM. The source of the herbicide may have been weed control along the railroad, which crosses the stream at the sampling location.

In stormflow samples, 13 compounds had median detected concentrations greater than 0.5 µg/L, and 5 compounds had median detected concentrations greater than 1.0 µg/L: the antioxidant 5-methyl-1H-benzotriazole, the plasticizer DEHP, the solvent isophorone, the detergent metabolite 4Nonylphenol diethoxylate, and the sterol beta-sitosterol. The three compounds with the highest stormflow concentrations were all PAHs: fluoranthene (25.6 µg/L, estimated), phenanthrene (26.3 µg/L, estimated), and pyrene (25.6 µg/L, estimated), all from the same KRM sample.

PAH concentrations in stormflow samples were directly related to the percentage of the basin in urban land use (fig. 4). The median total PAH (tPAH) concentration from stormflow samples at the most urban site (KRM) was 83 times greater than at the least urban site (MRC). Likewise, the magnitude of the difference between tPAH concentrations in base-flow as opposed to stormflow samples appears to be related to the percentage of the basin in urban use. The difference between the median base-flow and median stormflow concentration at MRC was less than 1.0 µg/L (dependent upon value used for nondetections), compared to a difference of approximately 14 µg/L at KRM.

In pore-water samples, 11 compounds had median detected concentrations greater than 0.5 µg/L, and 5 compounds had median detected concentrations greater than 1.0 µg/L: the PAH fluoranthene, the herbicide dichlorophenyl isocyanate, and the sterols beta-coprostanol, beta-sitosterol, and cholesterol (tables 4a, and 4b). The compound with the highest concentration in pore-water samples was p-Cresol, measured at 119 µg/L (estimated) in a sample from HCW.

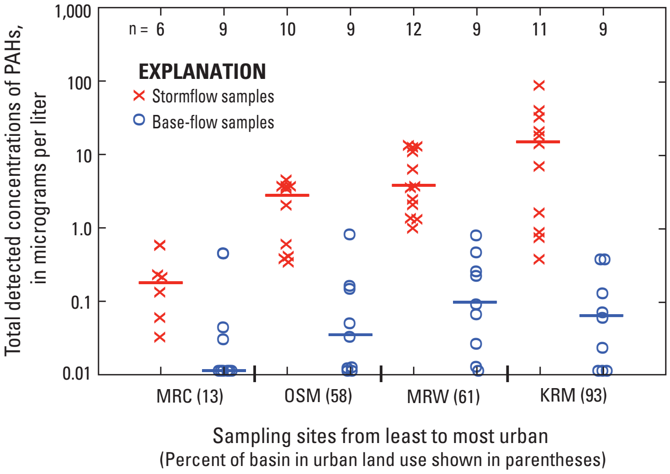


Figure 4. Total detected concentrations of polycyclic aromatic hydrocarbons (PAHs) in stormflow and base-flow samples. (Nondetections plotted at 0.01 micrograms per liter; horizontal lines mark medians; site abbreviations are defined in table 1; n, number of samples)

In sediment samples, the median detected concentrations of most compounds (36) were less than 100 micrograms per kilogram (µg/kg) (tables 5a, and 5b). Eleven compounds had median detected concentrations greater than 500 µg/kg, and six had median detected concentrations greater than 1,000 µg/kg: four PAHs (benzo[a]pyrene, fluoranthene, phenanthrene, and pyrene) and two sterols (beta-sitosterol and cholesterol). Fluoranthene had the highest detected concentration of all compounds in sediment samples, measured at 14,900 µg/kg in a sample from MRW.

By summing the detected concentrations within each OWC class and for each sample (hereafter referred to as class concentrations), some general patterns become apparent. Figure 5*A* and *B* show maximum class concentrations for each sampling location and sample type. Maximum class concentrations above 1 µg/L are much more common in the stormflow and pore-water samples than in the base-flow samples (fig, 5*A*). Herbicide maximum concentrations are uniformly high across the three water-sample types, but completely undetected in the sediment samples (fig, 5*B*). Most of the high herbicide concentrations can be attributed to one compound, dichlorophenyl isocyanate, a noncrop herbicide commonly used in urban areas. PAH maximum concentrations are high in the stormflow, pore-water, and sediment samples, but less than 1 µg/L in the base-flow samples. Solvents have high maximum concentrations in the stormflow samples but not in any other sample type. Sterols have high, uniform maximum concentrations across all sample types and sites. Sterols have been shown to be effective tracers of wastewater (Maldonado and others, 1999; Wilkison and others, 2002), but they also can occur naturally from plants or carnivores. Among the water samples, the fuels, human drugs (nonprescription), and miscellaneous classes were the only ones with no concentrations greater than 1 µg/L.

Class concentrations in each sample were averaged, by site, to give equal weight to sites with different numbers of samples. The resulting site-averaged class concentrations, plotted in figure 6, show that for most classes base-flow concentrations tend to be lower than stormflow and pore-water concentrations. This is especially true of the PAHs, dyes/pigments, and solvents. The miscellaneous class is the only one for which base-flow class concentrations are greater than stormflow class concentrations. This indicates that nonpoint-source runoff and (or) wastewater leakage associated with storm events may be important sources of OWCs to the streams.

Site-averaged stormflow and pore-water class concentrations are comparable for most classes, with some exceptions including plasticizers and solvents, for which stormflow concentrations are higher, and flavors/fragrances and the miscellaneous class, for which pore-water concentrations are higher. In sediment samples, site-averaged class concentrations are highest for the PAHs and sterols and lowest for fire retardants, solvents, herbicides (not detected), human drugs (not detected), and the miscellaneous class.

Domestic Wastewater Indicators

Most of the compounds sampled in this study are associated with either nonpoint-source runoff (such as herbicides, insecticides, PAHs, and fuels) or industrial or domestic wastewater. To identify OWC contributions primarily from domestic human wastewater sources (for example, from leaking sanitary sewers and septic systems), a subset of 20 compounds considered to be likely indicators of those sources were analyzed. These compounds are indicated in table 2 and include all of the fire retardants and detergent metabolites, most of the flavors/fragrances, and the antimicrobial disinfectant triclosan.

All pore-water and stormflow samples had detections of one or more of the domestic wastewater indicator compounds, with a median of 5.5 compounds in pore-water samples and 4 in stormflow samples. Domestic wastewater-indicator compounds were detected in 79 percent of base-flow samples, with a median of 2 compounds per sample, and in 89 percent of sediment samples, with a median of 3 compounds per sample. The maximum number of domestic wastewater-indicator compounds detected in a sample was 11, in a sample from MRJ, possibly because of its proximity to the effluent discharge of the Jones Island Water Reclamation Facility (wastewater treatment plant), approximately 1,000 feet (ft) away.

The total sample concentrations of the 20 domestic wastewater-indicator compounds are plotted by site and sample type in figure 7. Samples from most sample types, and especially stormflow samples, show a general trend of increasing total concentrations with increasing urban area. Total concentrations generally were lower in base-flow samples than in stormflow or pore-water samples. For both base-flow and stormflow samples, the site with the highest median total

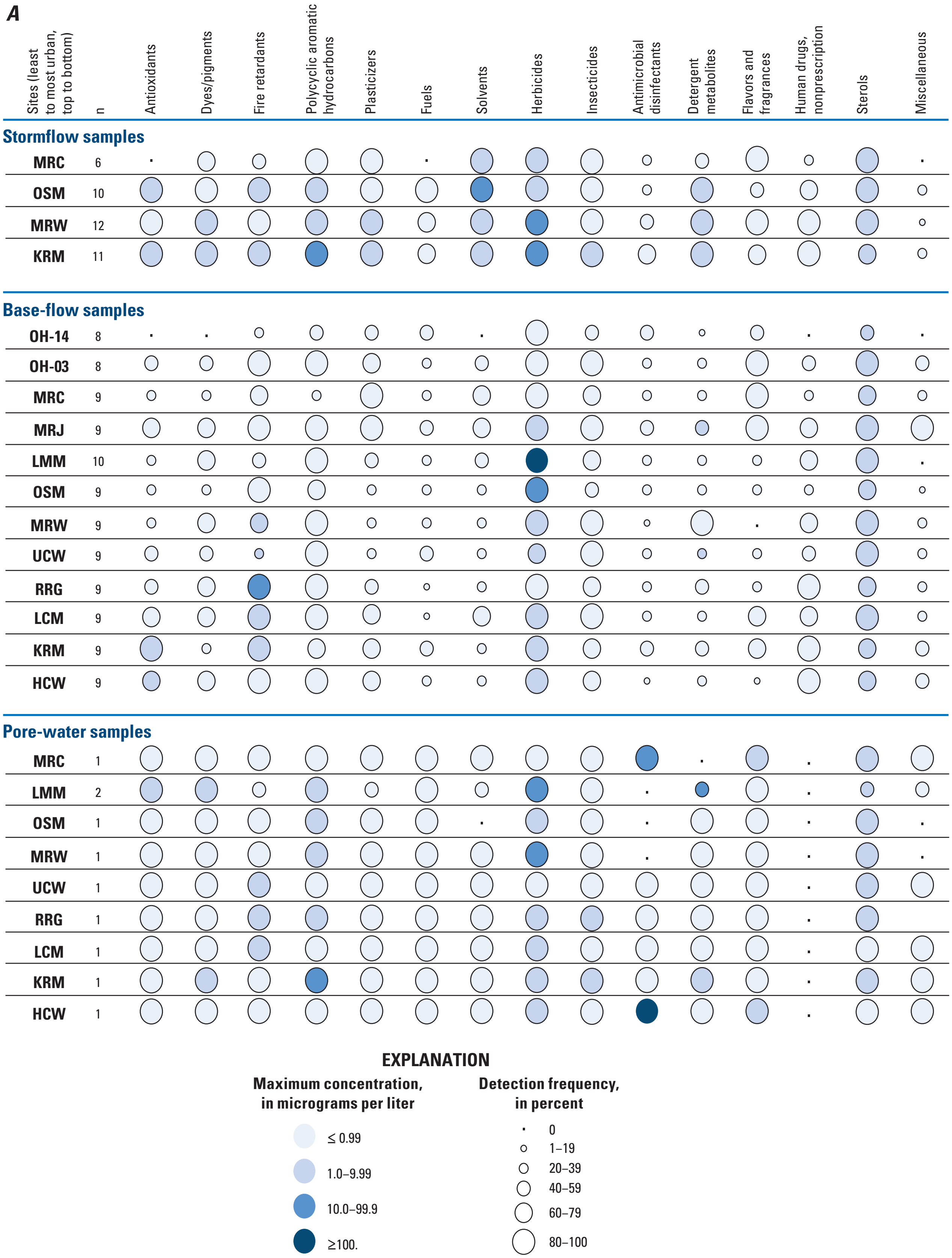


Figure 5. Class-detection frequencies and maximum class concentrations for each site. *A*, Stormflow, base-flow, and pore-water samples. *B*, Sediment samples. (Site abbreviations are defined in table 1; n, number of samples; ≤, less than or equal to; ≥, greater than or equal to; <, less than)

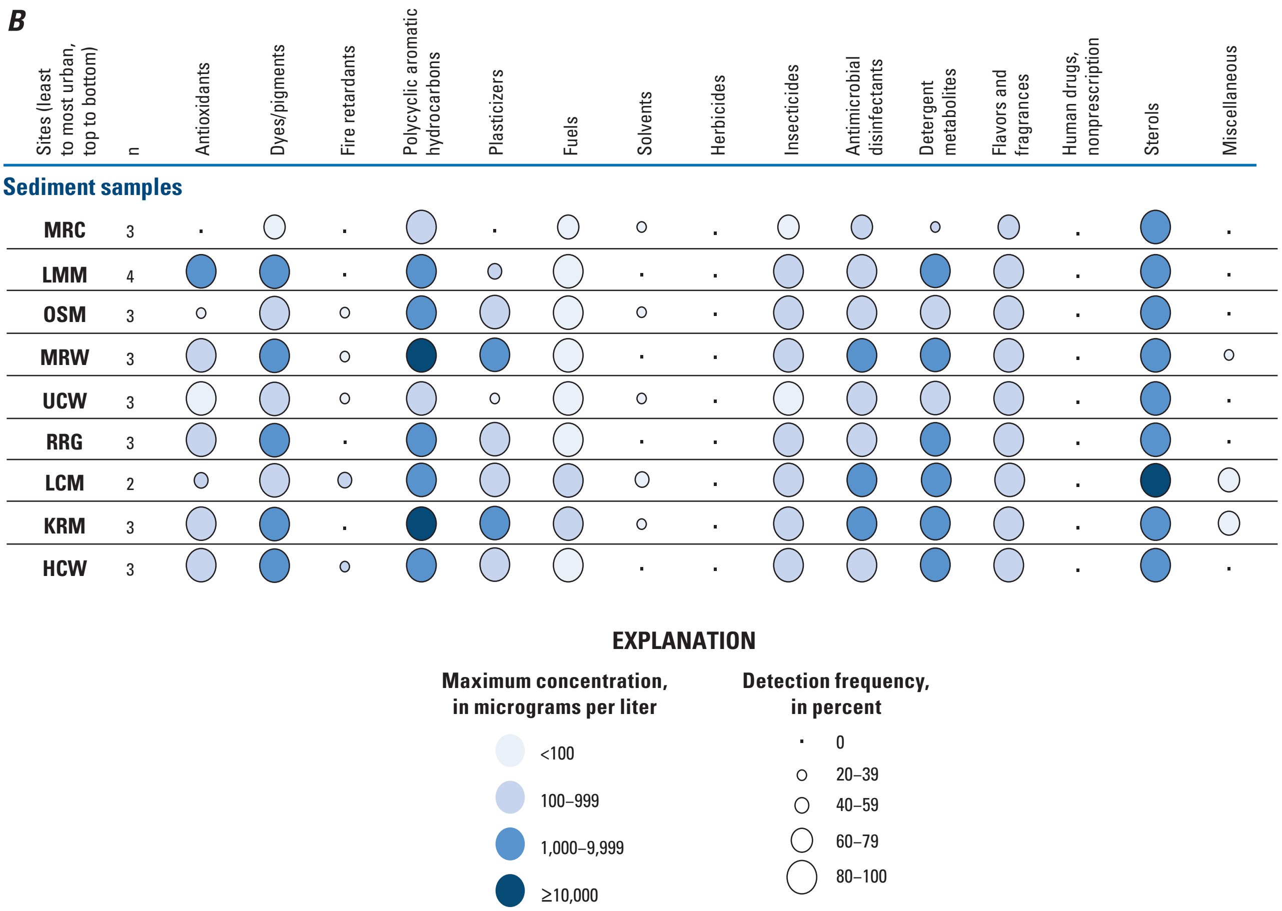


Figure 5. Class-detection frequencies and maximum class concentrations for each site. *A*, Stormflow, base-flow, and pore-water samples. *B*, Sediment samples. (Site abbreviations are defined in table 1; n, number of samples; ≤, less than or equal to; ≥, greater than or equal to; <, less than)—Continued

concentration was KRM. MRJ had the second highest median total concentration among the base-flow sampled sites.

Among the pore-water samples, the highest total concentration of domestic wastewater-indicator compounds was from one of the two samples from LMM. That sample had a total concentration of 17.2 µg/L, of which 17.15 µg/L came from only two compounds— 4-Cumylphenol and 4-Nonylphenol—both detergent metabolites. The other pore-water sample collected at that site, 1 year later, had a total concentration of 0.13 µg/L. Of the sediment samples, the highest median total concentrations were from sites MRW (2,974 µg/kg) and HCW (2,970 µg/kg).

OWCs and Sampling Sites

OWCs were detected at all sampled streams and harbor sites. The site where the fewest number of compounds were detected was the harbor site OH-14, with 22 of the 69 compounds detected at least once. The site where the most compounds were detected was KRM, with 59 of the 69 compounds detected at least once.

In order to compare detected concentrations among sites, the detected concentrations for each sample were summed, using zeros for nondetections. Figure 8 shows the median sum concentration for each site and sample type, as well as the percentage of parameters detected for each site and sample type. The median sum concentration for base-flow samples was less than 5 µg/L at all sites. At the four sites where stormflow samples were collected, the median sum concentrations ranged from 4.4 µg/L at MRC to 40.9 µg/L at KRM. For the pore-water samples, the median sum concentrations ranged from 5.6 µg/L at UCW to 136 µg/L at HCW. The high value at HCW can be attributed mostly to one compound, p-Cresol, which was measured at 119 µg/L (estimated). P-Cresol has numerous applications, including in antimicrobial disinfectants, antioxidants, dyes, plastics, and pesticides. Median sum concentrations for sediment samples ranged from 4,889 µg/kg at MRC to 46,526 µg/kg at KRM.

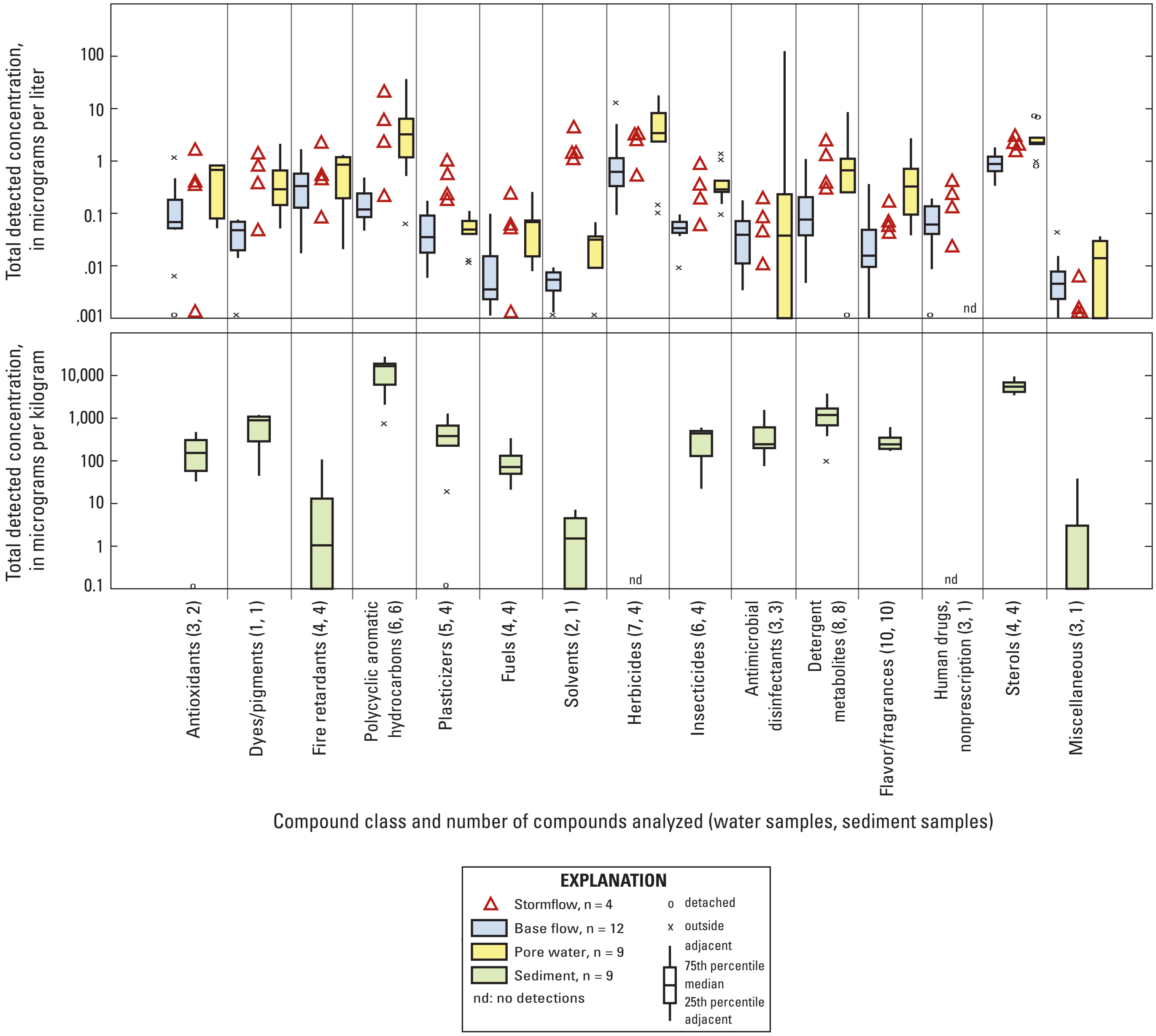


Figure 6. Total detected class concentrations for each sample type. (Total class concentrations from each sample are averaged by site to give all sites equal weight; n, number of samples)

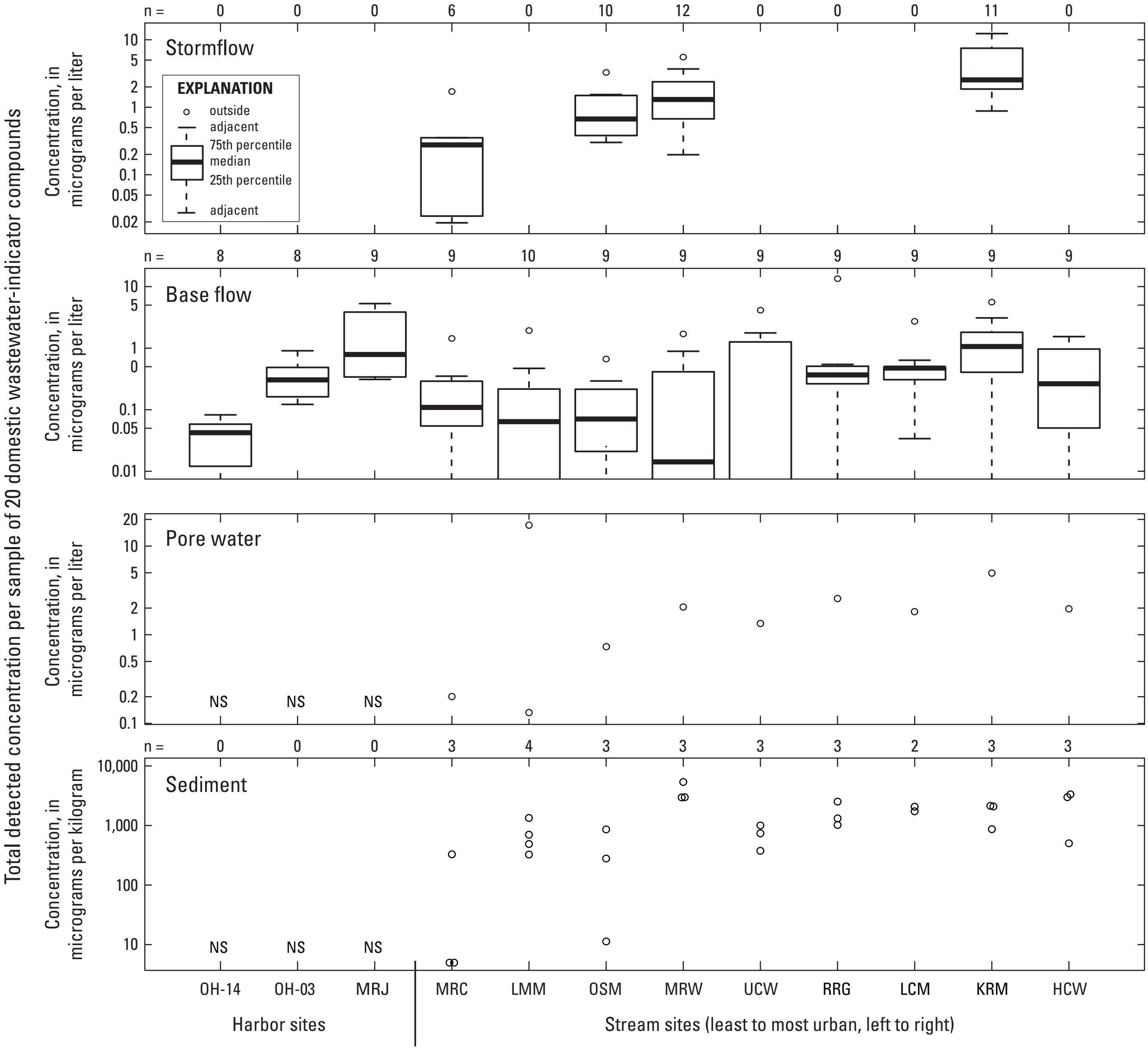
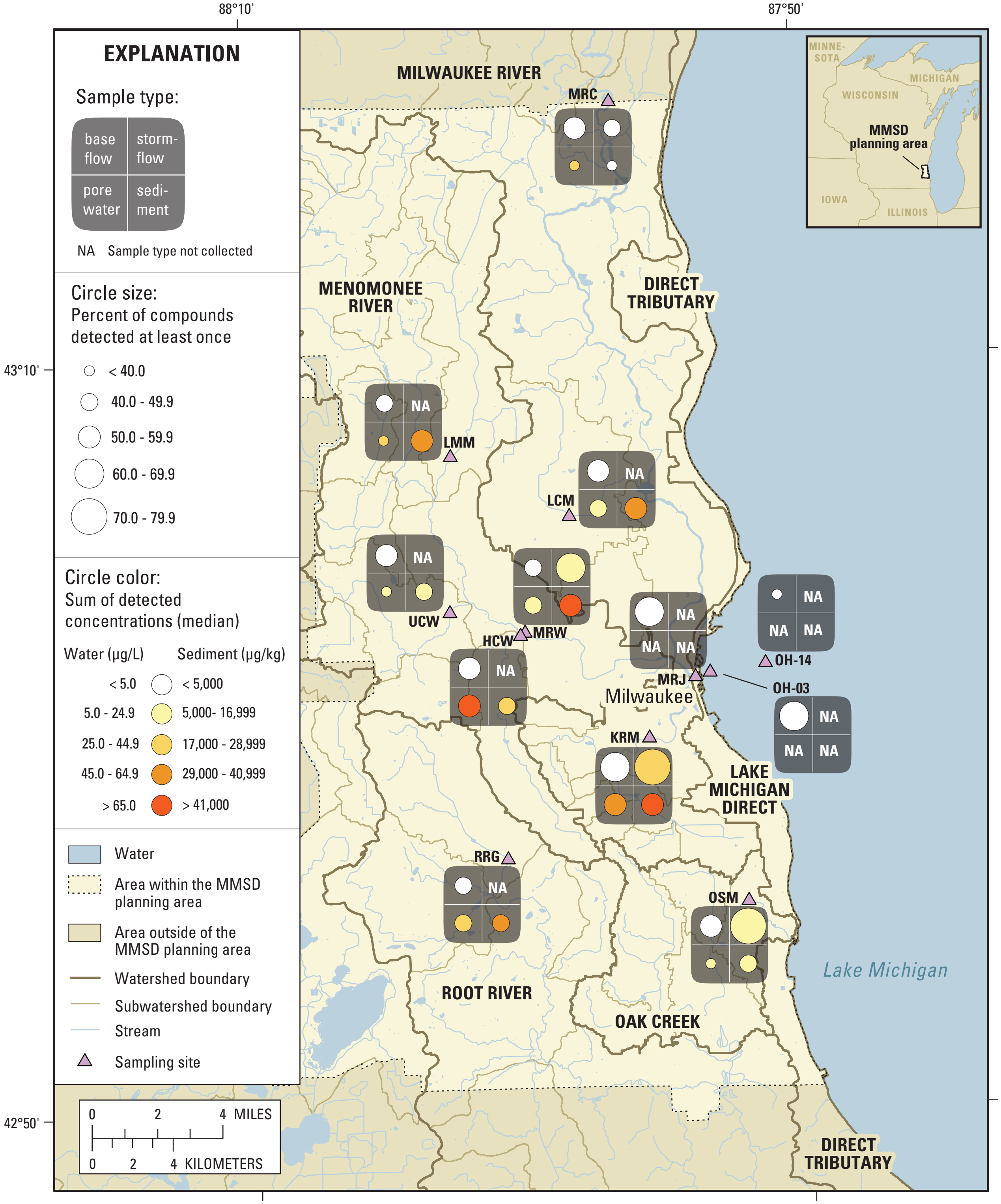


Figure 7. Total detected concentration per sample of 20 domestic wastewater-indicator compounds commonly associated with sanitary sewer and septic sources. (Scales differ between sample types; site abbreviations are defined in table 1; n, number of samples; NS, no samples collected)



Base composited from Southeastern Wisconsin Regional Planning Commission regional base map, 1:2,000, 1995; U.S. Geological Survey digital line graph hydrography, 1:100,000, 1989; Wisconsin Department of Natural Resources version 2 hydrography, 1:24,000, 2002. Wisconsin Transverse Mercator Projection, referenced to North American Datum of 1983, 1991 adjustment.

Figure 8. Summary of the distribution of organic waste compounds in the Milwaukee Metropolitan Sewerage District (MMSD) planning area, Wisconsin. (Sampling site abbreviations are listed in table 1; <, less than; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; >, greater than)

For most sites, the percentage of compounds detected at least once was lower for pore-water and sediment samples than for base-flow and stormflow samples. However, this is biased by the small number of pore-water and sediment samples as compared to the other sample types. For base-flow samples, the percentage of compounds detected at least once ranged from 32 percent at OH-14 to 69.6 percent at MRJ. The range for stormflow samples was from 44 percent at MRC to 79.7 percent at KRM. MRC was the only stormflow sampling site where a greater percentage of the compounds were detected in the base-flow samples than in the stormflow samples (57 as opposed to 44 percent). As MRC is the least urban of the stormflow sampling sites, this indicates that storm-water runoff is an important source of OWCs to urban streams but less so to rural ones. The difference also may be related to the greater number of base-flow as opposed to stormflow samples collected at MRC (nine base-flow samples compared to six stormflow samples), and (or) to dilution during storm events. Unlike most of the other sites, MRC has several wastewater-treatment plants discharging upstream. These plants likely input OWCs to the stream constantly, and those inputs would be most concentrated during base-flow conditions.

For pore water, the percentage of compounds detected at least once ranged from 28 percent at UCW to 54 percent at KRM. Sediment samples varied the least, ranging from 35 percent of the compounds detected at MRC to 54 percent at KRM. Overall, the site with the highest OWC concentrations and detection frequencies was KRM. For both detection rates and median sum concentrations, KRM had the highest or second-highest values for each of the four sample types. MRJ, the stream/harbor transition site, also had high concentrations and detection frequencies. Though only base-flow samples were collected there, those samples had a higher percentage of compounds detected at least once, and a higher median sum concentration than the base-flow samples at any other site, including KRM. This may be in part because of MRJ's location near the confluence of the Kinnickinnic, Menomonee, and Milwaukee watersheds, as well as its proximity to the Jones Island Water Reclamation Facility (wastewater-treatment plant) effluent discharge.

More maximum compound concentrations were measured in samples from KRM than from any other site (tables 4a, 4b, 5a, and 5b). In stormflow samples, the maximum measured concentrations for 43 of the 69 compounds were from KRM samples; the site with the second-highest number of maximum compound concentrations in stormflow samples, OSM, had only 8. Maximum compound concentrations in pore-water and sediment samples were dominantly from KRM samples as well, with 18 and 16 compounds, respectively. Maximum compound concentrations in base-flow samples were more evenly distributed among sites, with MRJ, UCW, and KRM having the most (9, 9, and 8, respectively) and RRG, MRW, and MRC having the least (2, 2, and 1, respectively).

The stream sites with the lowest OWC concentrations and detection frequencies were MRC (the least urban site) and UCW. For both stormflow and sediment samples, the median sum concentrations at MRC were approximately an order of magnitude less than at KRM. In the Milwaukee River Harbor, detections and concentrations decreased with increasing distance from the mouth of the river. Harbor site OH-14 had the lowest median sum concentration and the lowest percent of compounds detected, followed by harbor site OH-03, followed by the stream/harbor transition site MRJ.

Adverse Impacts on Aquatic Life

The potential impacts of OWCs on aquatic life are assessed by (1) discussion of EDCs, (2) comparisons of OWC concentrations to known toxicity benchmarks, and (3) analysis of toxicity bioassay results.

Endocrine-Disrupting Compounds

Of the 69 compounds sampled in this study, 27 are known or suspected endocrine disruptors (table 2; The Endocrine Disruption Exchange, Inc., 2011). Endocrine disruptors are compounds that possess hormone-like activity and have the potential to disrupt normal endocrine function by interfering with hormone signaling. Research has shown that exposure to EDCs can result in adverse reproductive, developmental, neurologic, immunologic, carcinogenic, and ecologic effects (National Research Council, 1999; Vajda and others, 2008; Vandenberg and others, 2012).

Known or suspected EDCs were found in all pore-water, stormflow, and sediment samples, and in more than 90 percent of base-flow samples. The median number of EDCs per sample was 8.5 for pore-water, 9 for stormflow, 4 for base-flow, and 6.5 for sediment samples. The maximum number of EDCs in a sample was 15, found in a stormflow sample from MRW.

In base-flow samples, the highest EDC-detection rates were found at two of the harbor sites, OH03 and MRJ. As discussed in previous sections, this may be related to the proximity of those sites to the Jones Island Water Reclamation Facility (wastewater-treatment plant) effluent discharge.

OWCs and Toxicity Benchmarks

Of the 69 OWCs sampled, 25 have known AqT benchmarks (tables 4a, 4b, 5a, and 5b). These benchmarks are based on individual compounds, rather than the complex mixtures of 20 to 40 different compounds observed in environmental samples in this study. Likewise, benchmarks often are based on exposure times of 10 days or less (U.S. Environmental Protection Agency, 2002; 2003), whereas environmental exposure to these compounds may be measured in weeks or months. For these reasons, comparisons to single-compound benchmarks may underestimate the potential for adverse effects on aquatic organisms (Gilliom and others, 2006).

Figure 9 shows compounds with toxicity-benchmark exceedances in one or more sample type. In water samples (stormflow, base flow, and pore water), the compounds with the most frequent benchmark exceedances were PAHs: anthracene, benzo[a]pyrene, fluoranthene, phenanthrene, and pyrene. Benchmarks for these five PAH compounds were exceeded in 50 to 95 percent of all stormflow and pore-water samples, and in 17 to 53 percent of all base-flow samples (phenanthrene excluded). In stormflow and pore-water samples, the highest mean toxicity quotients (TQ_{mean}, computed by dividing the mean detected concentration by the lowest of the benchmarks) were for pyrene, with values of 97 and 101, respectively. This means that of the stormflow and pore-water samples with pyrene detections, the mean concentration was approximately 100 times the AqT-benchmark value. The highest toxicity quotient observed also was for pyrene, in a stormflow sample from KRM, with a value of 1,024. The higher detection frequencies and higher mean toxicity quotients of PAHs in stormflow samples as compared to base-flow samples indicates urban runoff may be an important source of these compounds to the stream.

Aside from the PAHs, compounds with toxicity-benchmark exceedances in water samples include DEHP in stormflow and base-flow samples, NP in stormflow and pore-water samples, and bromacil and carbaryl in base-flow samples (fig. 9).

Most of the compounds with AqT exceedances in water samples also had exceedances in sediment samples (fig. 9). Benchmarks for the six PAH compounds were exceeded in 64 to 93 percent of all sediment samples, with TQ_{mean} values from 3.0 to 79 in samples with detected concentrations. Other compounds with benchmark exceedances in sediment samples were DEHP (54 percent of samples, TQ_{mean} of 3.8), 2-Methylnapthalene (61 percent of samples, TQ_{mean} of 2.8), p-Cresol (14 percent of samples, TQ_{mean} of 0.5), phenol (46 percent of samples, TQ_{mean} of 4.4), and NP (29 percent of samples, TQ_{mean} of 0.9).

While toxicity benchmarks for individual compounds are useful, aquatic organisms often are exposed to tens or hundreds of compounds at once; therefore, quantifying the toxicity of mixtures of compounds may provide a more realistic assessment. A number of authors have developed methods to compute the toxicity of mixtures of PAHs, including MacDonald and others (2000) and more recently the U.S. Environmental Protection Agency (2003; 2010).

The EPA method computes an acute and chronic toxicity-potency factor for each sample (each mixture of PAH compounds). Acute toxicity refers to toxicity to aquatic organisms over a short exposure time, usually a few days or less, whereas chronic toxicity is over a longer exposure time. Both the acute and chronic potency factors use a toxicity benchmark of 1, where potency factors greater than 1 are considered potentially toxic. None of the base-flow water-column samples showed potential toxicity using the EPA method (fig, 10*A*). In contrast, three of the four streams sampled during stormflow conditions showed potential PAH toxicity under chronic-exposure conditions, and two of the four streams showed potential toxicity under acute-exposure conditions (fig, 10*C*).

In sediment and pore-water samples, potential toxicity under chronic-exposure conditions was seen at seven of nine and four of nine sites sampled, respectively (figs. 10*B* and *D*). The EPA calculation for sediment requires knowledge of the organic content of the sample. Because that information was not available, a range of 1.5 to 7.0 percent organic content was assumed, based on the range of organic content seen in samples from the same and nearby streams in a previous study (Corsi and others, 2011). Most of the sediment samples were either above or below the chronic-toxicity benchmark regardless of the organic content assumed.

For comparison to the EPA method previously described, probable effect concentration (PEC) quotients were computed for sediment samples using the method described by MacDonald and others (2000). Generally, the MacDonald and the EPA methods agreed on which samples were above and below the toxicity benchmark.

Toxicity Bioassays

The greatest adverse effect in samples from different stream compartments was observed in *C. tentans* assays from sediment samples (figs. 11, 12, and 13). Results from water-column sample assays indicated that *C. dubia* survival was at least 80 percent of the laboratory-control survival, and the mean young produced (reproduction) was at least 59 percent of that from the laboratory control. The three samples that had the lowest weights for *C. dubia* in water-column assay results were from MRC, MRM, and WCG (64, 59, and 67 percent of the laboratory control, respectively; fig. 11). Results from the OWC analysis did not indicate an obvious reason that reproduction results from these three sites should be different than the other sample results. MRC and MRM are the only two sites with wastewater-effluent discharges upstream.

Results from pore-water sample assays indicated that survival of *C. dubia* and *H. azteca* was at least 80 percent of the laboratory-control survival in all samples (fig. 12). The mean *C. dubia* young produced for one sample was as low as 45 percent (at site RRF), with all other sites resulting in 70 percent or greater of the laboratory-control results. Examination of the OWC pore-water sample results for site RRF did not indicate that concentrations were greater than samples with no adverse-toxicity result. It does not appear that low *C. dubia* reproduction in the assay from the site RRF sample can be explained by concentrations of measured parameters for this study.

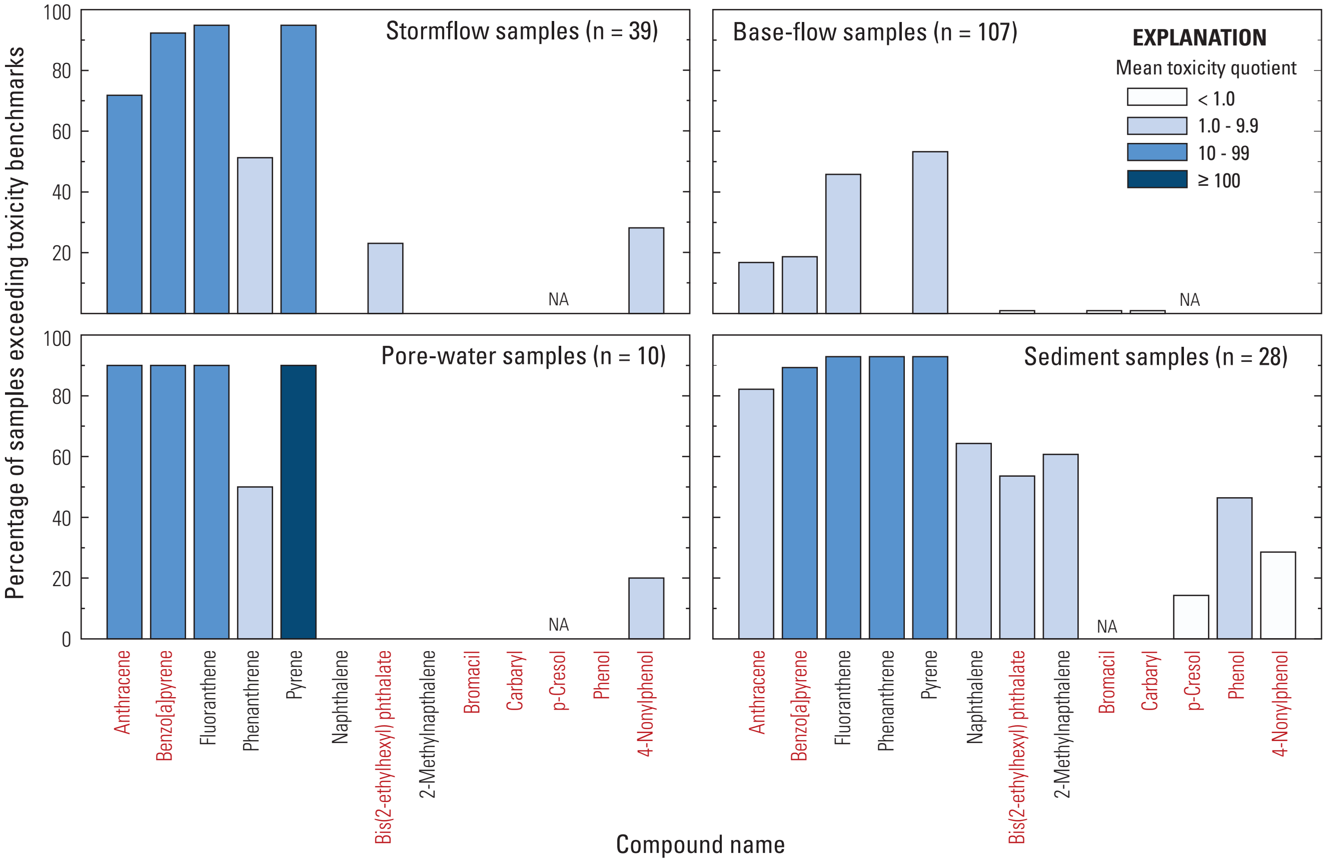


Figure 9. Compounds with exceedances of toxicity benchmarks for one or more sample type. Bar heights indicate the percentage of samples exceeding the lowest benchmark listed in tables 4 and 5. Bar color indicates the maximum toxicity quotient (concentration/benchmark) observed for each compound. Compounds in red are known or suspected endocrine disruptors according to The Endocrine Disruption Exchange, Inc. (2011). (n, number of samples; <, less than; ≥, greater than or equal to; 4-Nonylphenol is the sum of all isomers; NA, no known toxicity benchmark)

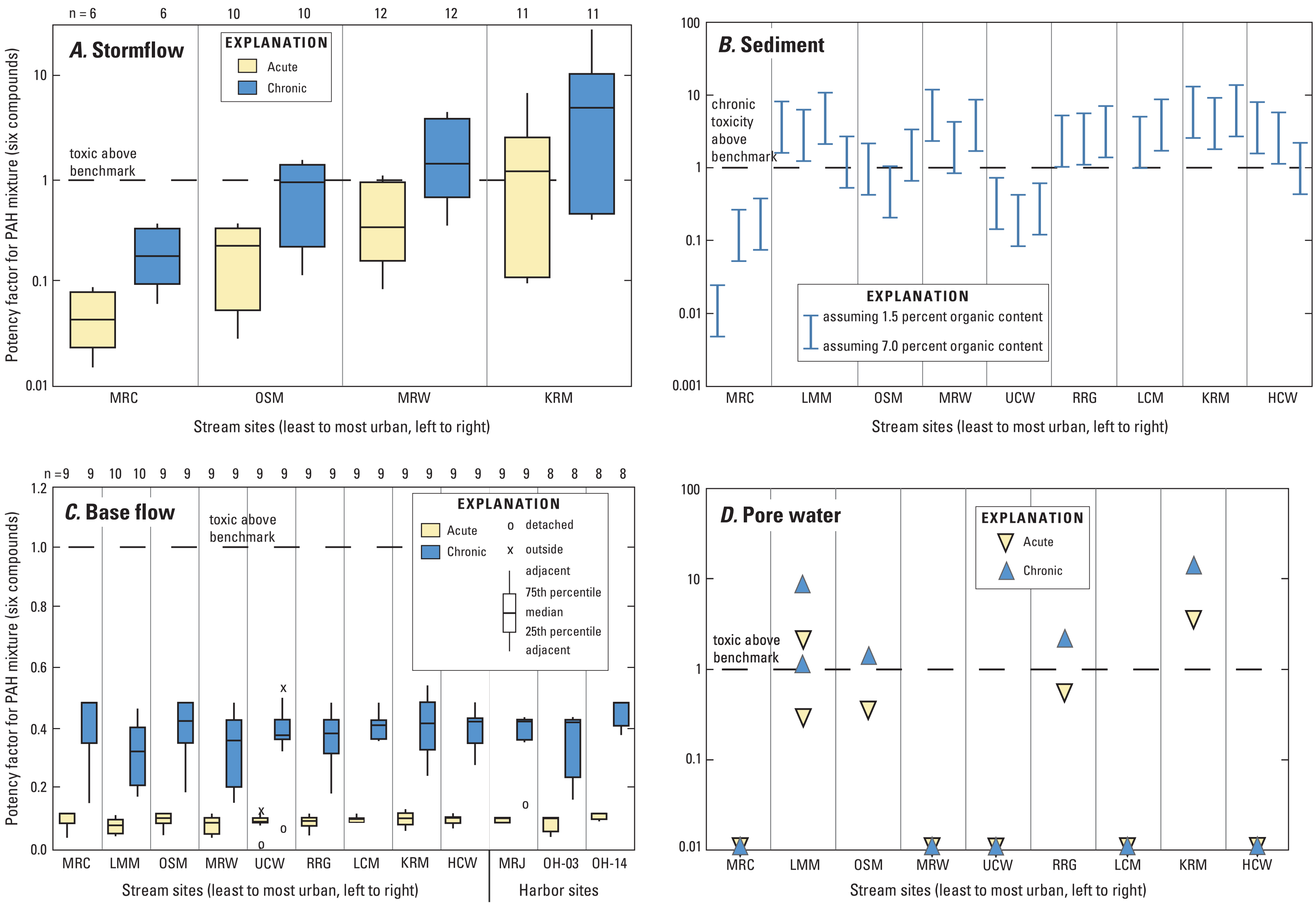


Figure 10. Polycyclic aromatic hydrocarbons toxicity. *A*, Stormflow samples. *B*, Sediment samples. *C*, Base-flow samples. *D*, Pore-water samples. A sample with a potency factor greater than 1 is considered toxic, based on calculations developed by the U.S. Environmental Protection Agency (2010). Acute and chronic toxicity were calculated for each sample, using different computations (only chronic toxicity is shown for sediment samples). For the sediment-sample calculations, an expected range of organic content was used because the actual organic content was not known. (Site abbreviations are defined in table 1; PAH, polycyclic aromatic hydrocarbon; n, number of samples)

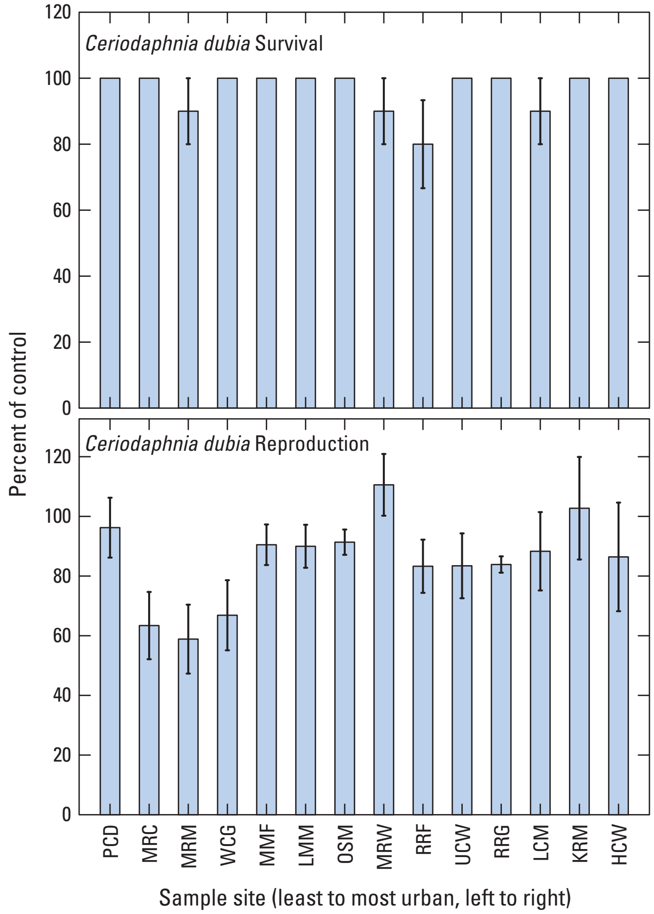


Figure 11. Water-column bioassay results for 14 sites in the Milwaukee area, 2006–7. Error bars indicate the standard error of replicates for each result. (Site abbreviations are defined in table 1.)

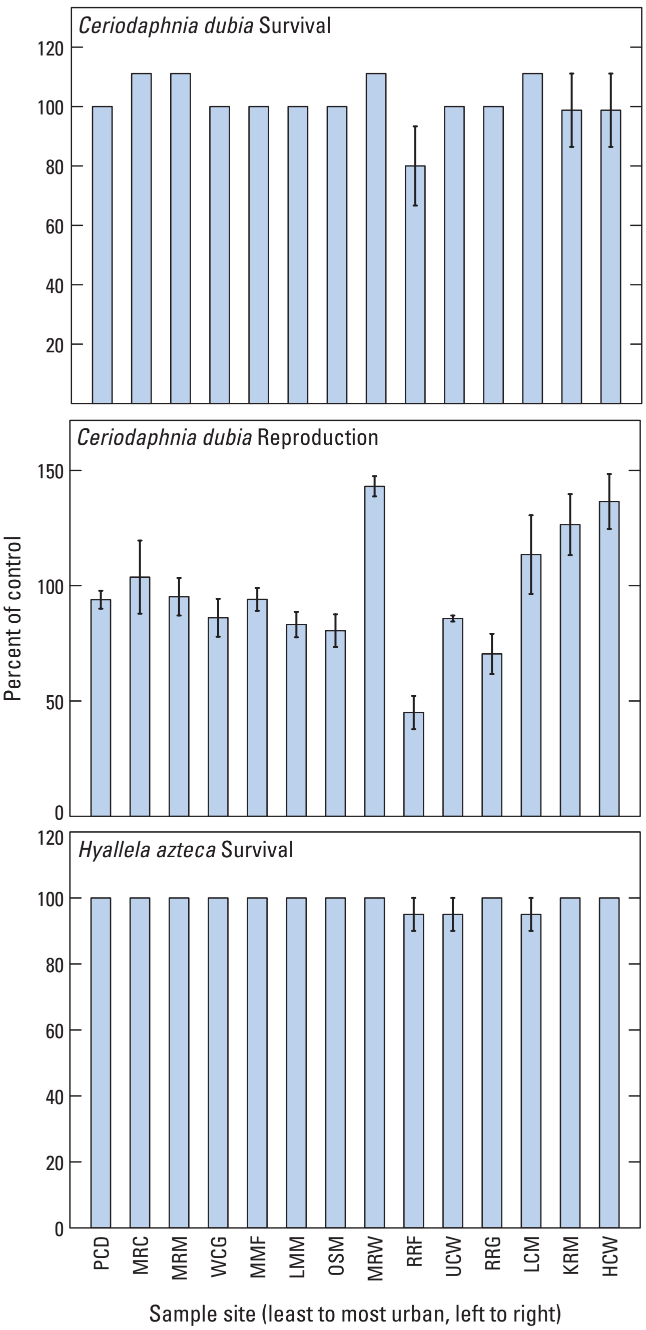


Figure 12. Pore-water assay results for 14 sites in the Milwaukee area, 2006–7. Error bars indicate the standard error of replicates for each result. (Site abbreviations are defined in table 1.)

Survival observed in sediment assay results for *C. tentans* was as low as 15 percent of that from the laboratory control at site LCM, but survival was 62 percent or greater of the laboratory control for all other samples (fig. 13). Weight of *C. tentans* was as low as 11 percent of the laboratory control for site LCM. Survival of *H. azteca* was at least 70 percent and weight was at least 97 percent of the laboratory control for all samples. Results for survival of both organisms were not substantially different after UV treatment (fig. 13).

C. tentans weights were analyzed with respect to OWC-analysis results as an attempt to explain adverse effect as defined in the section "Toxicity in Stream Compartments." Correlation coefficients of *C. tentans* weight to rank summations were all negative, indicating a clear increase of toxicity as OWC contamination increased in sediment samples (table 6). Scatter plots of the rank sums and *C. tentans* weight showed that three sites (HCW, LMM, and LCM) were persistent outliers in this relation. Less toxicity was observed in the samples from HCW and LMM than would have been predicted by any of the rank summations. More toxicity was observed in the LCM sample than would have been predicted by rank summations. This is not unexpected for the LCM sample since it was collected at a different time than the bioassay sample owing to laboratory complications. It is uncertain why the data from HCW and LMM appear different than other sites. Correlation coefficients computed without these outliers

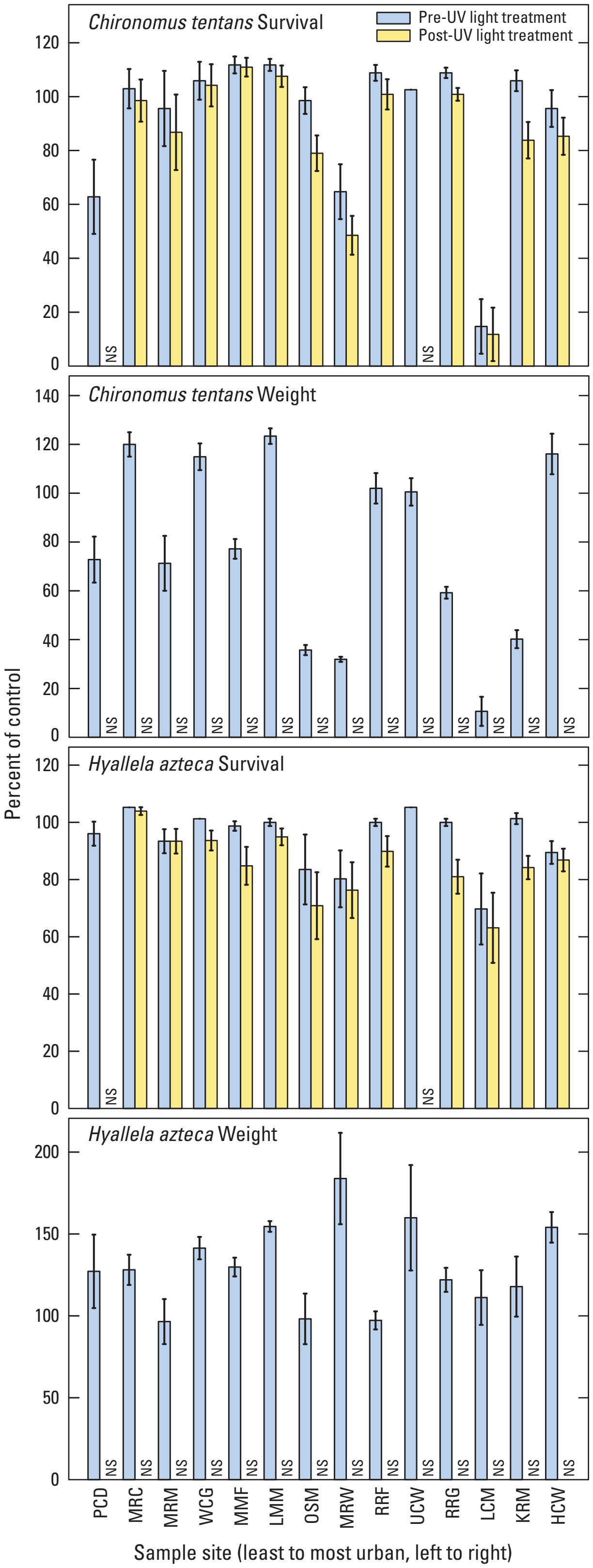


Figure 13. Sediment assay results for 14 sites in the Milwaukee area, 2006–7. Error bars indicate the standard error of replicates for each result. (Site abbreviations are defined in table 1; UV, ultraviolet; NS, not sampled)

improved for all except three of the class combinations. The classes that produced the best correlation (r = −0.97) of rank summations to *C. tentans* weight without outliers were PAHs, solvents, and fire retardants (fig. 14, table 6). The classes that produced the best correlation (r = −0.74) of rank summations to *C. tentans* weight using all sites including outliers were fuels, solvents, and fire retardants.

Most of the top 10 correlation coefficients using all sites or with the 2 outliers removed included PAHs, fuels, or both as categories. Other common variables included for the top 10 correlation coefficients were detergent metabolites, solvents, and fire retardants. Solvents and fire retardants were detected at only one site each (PCD and LMM, respectively), but inclusion of these two classes substantially improved explanation of *C. tentans* weight in sediment-assay variability (correlation coefficient closer to 1.0).

This comparison of OWC-category rankings with sediment bioassay results indicates a strong correlation between sediment toxicity and OWC presence. PAHs explain much of the variability in this relation and often were shown to exceed toxicity benchmarks in the potency-factor assessment for PAHs, so it is reasonable to assume that PAHs play a role in the observed toxicity. Still, there are strong cross-correlations between PAHs with fuels and insecticides (r = 0.79 and 0.95 for concentrations and r = 0.91 and 0.94 for ranks, respectively). This indicates a possibility that there could be influence from other OWCs or that the adverse effects observed could be owing to a mixture of these OWCs.

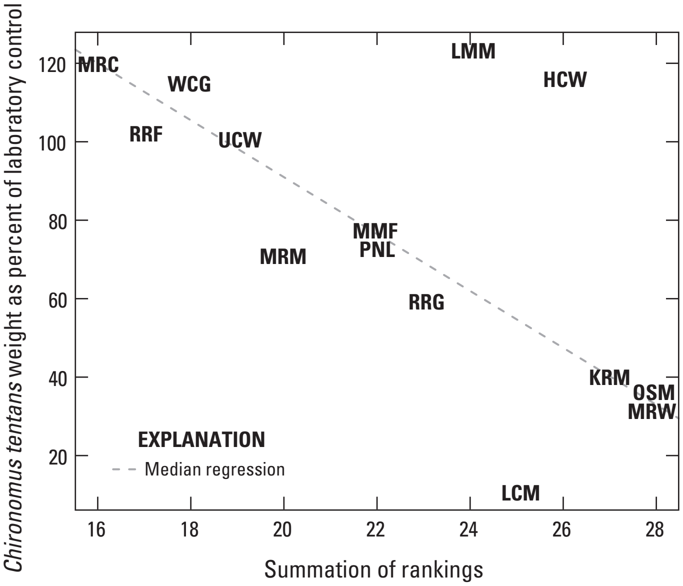


Figure 14. Relation between *Chironomus tentans* weight and rank summations of concentrations of polycyclic aromatic hydrocarbons, solvents, and fire retardants in sediment samples. (Site abbreviations are defined in table 1.)

Table 6. Pearson correlation coefficients of *Chironomus tentans* weight to rank summations of concentrations of classes of organic waste compounds in sediment samples, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; HCW, Honey Creek at Wauwatosa; LMM, Little Menomonee River at Milwaukee; LCM, Lincoln Creek 47th Street at Milwaukee; PAH, polycyclic aromatic hydrocarbon; top ten correlation coefficients are shown in **bold**]

OWC classes included			Pearson correlation coefficients of *Chironomus tentans*	
			All sites	Sites without persistent outliers (HCW, LMM, and LCM)
Detergent metabolites			−0.49	−0.81
	Fire retardant		−.55	−.86
	Fuel		**−.61**	−.82
	Insecticide		−.49	−.82
	PAH		−.53	−.82
	Solvent		−.5	−.82
	Fire retardant	Fuel	**−.66**	**−.89**
	Fire retardant	Insecticide	−.54	**−.88**
	Fire retardant	PAH	−.58	**−.88**
	Fire retardant	Solvent	−.56	**−.88**
	Fuel	Insecticide	−.55	−.81
	Fuel	PAH	−.57	−.82
	Fuel	Solvent	**−.64**	−.86
	Insecticide	PAH	−.5	−.82
	Insecticide	Solvent	−.52	−.86
	PAH	Solvent	−.57	**−.88**
Fire retardant			−.32	−.42
	Fuel		**−.69**	**−.88**
	Insecticide		−.53	**−.87**
	PAH		−.56	−.86
	Solvent		−.26	−.33
	Fuel	Insecticide	**−.59**	−.85
	Fuel	PAH	**−.61**	−.86
	Fuel	Solvent	**−.74**	**−.92**
	Insecticide	PAH	−.52	−.85
	Insecticide	Solvent	−.58	**−.95**
	PAH	Solvent	**−.64**	**−.97**
Fuel			**−.59**	−.75
	Insecticide		−.52	−.77
	PAH		−.54	−.78
	Solvent		**−.62**	−.78
	Insecticide	PAH	−.51	−.78
	Insecticide	Solvent	−.56	−.82
	PAH	Solvent	−.58	−.84
Insecticide			−.43	−.77
	PAH		−.45	−.78
	Solvent		−.47	−.84
	PAH	Solvent	−.5	−.85
PAH			−.47	−.77
	Solvent		−.54	**−.87**
Solvent			−.03	−.02

Summary and Conclusions

During 2006–9, the U.S. Geological Survey, in cooperation with the Milwaukee Metropolitan Sewerage District, sampled a suite of 69 organic waste compounds (OWCs) in different stream compartments and flow regimes in Milwaukee area streams and in the Milwaukee Harbor. Three stream compartments were represented: water column, streambed pore water, and streambed sediment. Water-column samples were divided by flow regime into surface water during base-flow conditions and surface water during stormflow conditions. OWC data should be interpreted cautiously as laboratory recovery rates vary (see Quality Assurance/Quality Control section).

OWCs were detected in all 196 samples collected, with as many as 41 different compounds detected in a single sample. Of the 69 compounds analyzed, 64 were detected at least once. The lowest detection rates were in the base-flow samples, with a median of 12 compounds detected per sample. Median detection rates for stormflow, streambed pore-water, and streambed-sediment samples were approximately double those of the base-flow samples. The compound classes with the highest detection rates were the polycyclic aromatic hydrocarbons (PAHs), insecticides, herbicides, and dyes/pigments.

The compound classes with the highest detected concentrations were the PAHs (water and sediment samples) and the herbicides (water samples only). Sterols also had relatively high detection frequencies and concentrations in both water and sediment samples, but some of those compounds are naturally occurring and not necessarily human-derived. The PAH concentrations show a clear urban gradient in stormflow samples, with concentrations increasing with increasing urban area. However, the urban gradient is less clear in base-flow PAH concentrations, indicating that urban runoff is an important source of PAHs to the streams. Total concentrations of the 20 domestic wastewater-indicator compounds also show an urban gradient in stormflow samples, indicating increased sanitary and (or) septic system leaks or releases during stormflow conditions, as compared to base flow.

The Kinnickinnic River (KRM)—one of the most urban sites—had some of the highest detection frequencies and concentrations in all sample types. Sites with the lowest detection frequencies and concentrations were the least urban stream sites: Milwaukee River near Cedarburg (MRC), and Middle Outside Harbor Breakwater Lake Site (OH-14). The Milwaukee River at the mouth at Jones Island (MRJ) site had some of the highest detection frequencies and concentrations of endocrine-disrupting chemicals and domestic wastewater-indicator compounds, likely in part because of its proximity to the Jones Island Water Reclamation Facility (wastewater-treatment plant) effluent discharge point.

Aquatic-toxicity benchmarks were exceeded for 12 of the 25 compounds with known benchmarks. The compounds with the greatest benchmark exceedances were the PAHs, both in terms of exceedance frequency (up to 93 percent for some compounds in sediment samples) and magnitude (concentrations up to 1,024 times greater than the benchmark value). Other compounds with toxicity-benchmark exceedances include Bis(2-ethylhexyl) phthalate (a plasticizer), 2-Methylnapthalene (a component of fuel and oil), phenol (an antimicrobial disinfectant with diverse uses), and 4-Nonylphenol (sum of all isomers; a detergent metabolite, among other uses). Analyzed as a mixture, the suite of PAH compounds were found to be potentially toxic in stormflow samples from three of four sites, in pore-water samples from four of nine sites, and in sediment samples from seven of nine sites. Base-flow samples showed low potential for PAH toxicity when those compounds were analyzed as a mixture. These results indicate that the likely source of PAHs to the stream is urban nonpoint runoff during storm events, and that the PAHs then collect in the streambed sediments.

Bioassay tests were conducted on base-flow samples, pore-water samples, and sediment samples, with the greatest adverse effect in *Chironomus tentans* assays from sediment samples. The sediment bioassay results were highly correlated with OWC results. There was a relation between PAH results and bioassay results for the majority of samples; however, solvents and flame retardants appeared to be important for one site each as well.

From an aquatic toxicity standpoint, PAHs are prominent in this study as compounds that likely have an adverse effect on aquatic organisms. This is evident from evaluation of the computed potency factors as well as results from direct bioassay testing. Additional insight on impacts to aquatic organisms may be gained if more aquatic-life criteria were available to help evaluate these data. In addition, if longer duration, chronic-toxicity information were made available, it may be valuable to perform a long-term assessment of the impacts of these compounds on aquatic life.

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Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Antioxidants	3-tert-Butyl-4-hydroxyanisole (BHA)	25013-16-5	0.2	150	Antioxidant, general preservative
	5-Methyl-1H-benzotriazole	136-85-6	1.22, 1.6, 2.0	na	Antioxidant in antifreeze and deicers
	Bisphenol A	80-05-7	0.4, 1.0	50	Manufacturing polycarbonate resins, antioxidant, flame retardant
Dyes/ pigments	9, 10-Anthraquinone	84-65-1	0.2, 0.5	50	Manufacturing dye/textiles, seed treatment, bird repellant
Fire retardants	BDE congener 47 (tetrabromodiphenyl ether)*	5436-43-1	0.2, 0.3, 0.39, 0.5	50	Flame retardant
	Tributyl phosphate*	126-73-8	0.2, 0.5	50	Antifoaming agent, flame retardant
	Tris(2-butoxyethyl) phosphate*	78-51-3	0.2, 0.21, 0.22, 0.26, 0.28, 0.3, 0.49, 0.5, 0.54,	150	Flame retardant
	Tris(dichloroisopropyl) phosphate (Fyrol FR 2)*	13674-87-8	0.2, 0.5	100	Flame retardant
PAH	Anthracene	120-12-7	0.2, 0.5	50	Wood preservative, component of tar, diesel, crude oil, combustion product
	Benzo[a]pyrene	50-32-8	0.2	50	Regulated PAH, used in cancer research, combustion product
	Fluoranthene	206-44-0	0.2, 0.3	50	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), combustion product
	Naphthalene	91-20-3	0.2	50	Fumigant, moth repellent, major component (about 10 percent) of gasoline
	Phenanthrene	85-01-8	0.2	50	Manufacturing explosives, component of tar, diesel fuel, or crude oil, combustion product
	Pyrene	129-00-0	0.2, 0.4	50	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), combustion product

Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Plasticizers	Bis(2-ethylhexyl) phthalate (DEHP)	117-81-7	0.22, 2.0, 2.1, 2.62, 9.8	250	Plasticizer for polymers and resins; major component of vinyl
	Diethyl phthalate	84-66-2	0.2, 0.22, 0.26, 0.32, 0.39, 0.5	100	Makes plastics more flexible; also cosmetics, insecticides, aspirin
	Triethyl citrate	77-93-0	0.2, 0.5	na	Cosmetics, pharmaceuticals
	Triphenyl phosphate	115-86-6	0.2, 0.5	50	Plasticizer, resin, wax, finish, roofing paper, flame retardant
	Tris(2-chloroethyl) phosphate (Fyrol CEF)	115-96-8	0.2, 0.5	100	Plasticizer, flame retardant
Fuels	1-Methylnaphthalene	90-12-0	0.2, 0.5	50	2-5 percent of gasoline, diesel fuel, or crude oil
	2,6-Dimethylnaphthalene	581-42-0	0.2, 0.5	50	Present in diesel/kerosene (trace in gasoline)
	2-Methylnaphthalene	91-57-6	0.2, 0.5	50	2-5 percent of gasoline, diesel fuel, or crude oil
	Isopropylbenzene (cumene)	98-82-8	0.2, 0.5	100	Manufacturing phenol/acetone, fuels and paint thinner
Solvents	Isophorone	78-59-1	0.2, 0.5	50	Solvent for lacquer, plastic, oil, silicon, resin
	Tetrachloroethene (PERC)	127-18-4	0.4, 0.5	na	Solvent, degreaser, veterinary anthelmintic

Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Herbicides	Atrazine	1912-24-9	0.2	100	Herbicide, selective for weed control and used on cropland and highway and railroad rights of way
	Bromacil	314-40-9	0.2, 0.26, 0.5, 0.8	500	Herbicide (general use pesticide), >80 percent non-crop usage on grass/brush
	Dichlorophenyl isocyanate	102-36-3	0.5, 1.6, 2.0	na	Degradate of diuron, a non-crop herbicide
	Metalaxyl	57837-19-1	0.2, 0.5	na	Herbicide, fungicide (general use pesticide), mildew, blight, pathogens, golf/turf
	Metolachlor	51218-45-2	0.2, 0.5	50	Herbicide (general use pesticide), indicator of agricultural drainage
	Pentachlorophenol (PCP)	87-86-5	0.8, 1.6, 1.71, 2.0, 3.2	na	Herbicide, fungicide, wood preservative, termite control
	Prometon	1610-18-0	0.2, 0.5, 1.6	50	Herbicide (noncrop only), applied prior to b lacktop
Insecticides	Carbaryl	63-25-2	0.2, 1.0	na	Insecticide, crop and garden uses, low persistence
	Carbazole	86-74-8	0.2, 0.5	50	Insecticide, Manufacturing dyes, explosives, and lubricants
	Chlorpyrifos	2921-88-2	0.2, 0.5	50	Insecticide, domestic pest and termite control (domestic use restricted as of 2001)
	Diazinon	333-41-5	0.2, 0.5	50	Insecticide, >40 percent nonagricultural usage, ants, flies
	Dichlorvos	62-73-7	0.2, 1.0	na	Insecticide, control of parasites on livestock and pets (pet collars)
	N, N-diethyl-*meta*-toluamide (DEET)	134-62-3	0.2, 0.25	100	Insecticide, urban uses, mosquito repellent
Antimicrobial disinfectants	p-Cresol (*para*-Cresol)	106-44-5	0.2, 1.0	250	Wood preservative, dissolve other chemicals, disinfectant
	Phenol	108-95-2	0.2, 0.5	50	Disinfectant, slimicide, manufacturing of synthetic fibers, mouthwash
	Triclosan*	3380-34-5	0.2, 1.0	50	Disinfectant, antimicrobial (concern for acquired microbial resistance)

Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Detergent metabolites	4-tert-Octylphenol monoethoxylate*	NA	1	250	Nonionic detergent metabolite
	4-Cumylphenol*	599-64-4	0.2, 1.0	50	Nonionic detergent metabolite
	4-Nonylphenol (sum of all isomers)*	84852-15-3	1.6, 5.0	750	Nonionic detergent metabolite
	4-Nonylphenol diethoxylate*	20427-84-3	3.2, 5.0	1,000	Nonionic detergent metabolite
	4-Nonylphenol monoethoxylate*	104-35-8	1.6, 2.0	500	Nonionic detergent metabolite
	4-Octylphenol (4-*normal*-Octylphenol)*	1806-26-4	0.2, 1.0	50	Nonionic detergent metabolite
	4-tert-Octylphenol diethoxylate*	NA	0.32, 0.5, 1.0	50	Nonionic detergent metabolite
	4-tert-Octylphenol*	140-66-9	0.2, 0.4, 1.0	50	Nonionic detergent metabolite

Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Flavors and fragrances	3-Methyl-1H-indole (skatol)	83-34-1	0.2, 1.0	50	Fragrance, stench in feces and coal tar
	Acetophenone	98-86-2	0.2, 0.3, 0.32, 0.37, 0.4, 0.5	150	Fragrance in detergent and tobacco, flavor in beverages
	Acetyl hexamethyl tetrahydro naphthalene (AHTN)*	21145-77-7	0.2, 0.5	50	Musk fragrance (widespread usage), persistent in groundwater
	Benzophenone*	119-61-9	0.2, 0.4, 0.5	50	Fixative for perfumes and soaps
	Camphor*	76-22-2	0.2, 0.5	50	Flavor, odorant, ointments
	d-Limonene*	5989-27-5	0.2, 0.5	50	Citrus flavoring and fragrance in food and houshold products, fungicide, antimicrobial, antiviral
	Hexahydrohexamethyl cyclopentabenzopyran (HHCB)*	1222-05-5	0.2, 0.5	50	Musk fragrance (widespread usage) persistent in ground water
	Indole	120-72-9	0.2, 0.5	100	Pesticide inert ingredient, fragrance in coffee
	Isoborneol*	124-76-5	0.2, 0.5	50	Fragrance in perfumery and disinfectants
	Isoquinoline*	119-65-3	0.2, 0.5	100	Flavors and fragrances
Human drugs, nonprescription	Caffeine	58-08-2	0.2, 0.5	na	Stimulant, beverages, diuretic, very mobile/ biodegradable
	Cotinine	486-56-6	0.8, 1.0	na	Primary nicotine metabolite
	Menthol	89-78-1	0.2, 0.29, 0.32, 0.4, 0.5	50	Cigarettes, cough drops, liniment, mouthwash

Table 2. Organic waste compounds sampled, their classes, and possible uses, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; CAS, Chemical Abstracts Service; µg/L, micrograms per liter; µg/kg, micrograms per kilogram; na, not analyzed; PAH, polycyclic aromatic hydrocarbons; >, greater than; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	CAS registry number	Reporting level		Possible compound uses or sources†
			Water samples (µg/L)	Sediment samples (µg/kg)‡	
Sterols	3-beta-Coprostanol	360-68-9	0.8, 1.2, 1.5, 1.6, 2.0	500	Carnivore fecal indicator
	beta-Sitosterol	83-46-5	0.8, 1.5, 1.6, 2.0, 2.09, 2.15, 3.2	500	Plant sterol
	beta-Stigmastanol	19466-47-8	0.8, 0.94, 1.05, 1.3, 1.6, 1.7, 2.0, 2.4	500	Plant sterol, pulp-mill waste, decomposing paper
	Cholesterol	57-88-5	0.8, 0.85, 0.91, 1.08, 1.1, 1.4, 1.6, 2.5	250	Often a fecal indicator, also a plant sterol
Miscellaneous	1,4-Dichlorobenzene	106-46-7	0.2, 0.5	50	Moth repellant, fumigant, deodorant
	Methyl salicylate	119-36-8	0.2, 0.45, 0.5	na	Liniment, food, beverage, UV-absorbing lotion
	Tribromomethane (bromoform)	75-25-2	0.2, 0.5	na	Wastewater ozination byproduct, military/explosives

‡ Reporting levels of sediment samples were variously elevated because of interferences.

† Zaugg and others, 2007.

* Compounds considered by the authors to be domestic wastewater-indicator compounds likely from sanitary sewer sources.

Table 4a. Organic waste compounds sampled and summary of analytical results of water samples, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; µg/L, micrograms per liter; ND, not detected; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequencies (percent)			Median detected concentration (µg/L)‡			Maximum detected concentration (µg/L), and site*		
		Base flow	Storm-flow	Pore water	Base flow	Storm-flow	Pore water	Base flow	Stormflow	Pore water
Antioxidants	3-tert-Butyl-4-hydroxyanisole (BHA)	0	0	0	ND	ND	ND	ND	ND	ND
	5-Methyl-1H-benzotriazole	32	33	40	e0.239	e1.25	e0.278	5.36, KRM	e3.42, KRM	e0.379, LCM
	Bisphenol A	24	77	100	e0.063	e0.144	e0.269	e0.311, LCM	e1.12, KRM	1.47, LMM
Dyes/pigments	9,10-Anthraquinone	46	95	100	e0.091	0.45	e0.30	0.416, LCM	e2.98, KRM	2.13, KRM
Fire retardants	BDE congener 47 (tetrabromodiphenyl ether)**	0	0	0	ND	ND	ND	ND	ND	ND
	Tributyl phosphate**	32	74	50	e0.0539	0.35	e0.0414	4.2, KRM	e4.69, KRM	e0.144, KRM
	Tris(2-butoxyethyl) phosphate**	50	64	60	e0.268	e0.389	e0.982	13.4, RRG	e1.5, KRM	e1.3, UCW
	Tris(dichloroisopropyl) phosphate (Fyrol FR 2)**	30	49	60	e0.0612	e0.0784	e0.0462	0.268, UCW	0.23, KRM	e0.0642, LCM
PAHs	Anthracene	25	74	90	e0.0142	e0.118	e0.182	e0.0556, UCW	5.16, KRM	1.0, KRM
	Benzo[a]pyrene	23	95	100	e0.0447	0.42	0.40	e0.14, UCW	e8.88, KRM	7.23, KRM
	Fluoranthene	76	100	90	e0.061	e0.927	e1.2	0.651, UCW	e25.6, KRM	11.6, KRM
	Naphthalene	9	67	70	e0.0281	e0.0707	e0.0871	e0.14, OH-14	0.26, KRM	e0.187, RRG
	Phenanthrene	51	100	90	e0.0382	e0.519	e0.551	0.343, UCW	e26.3, KRM	6.59, KRM
	Pyrene	71	100	90	e0.0479	e0.705	e0.927	0.413, UCW	e25.6, KRM	10.1, KRM

Table 4a. Organic waste compounds sampled and summary of analytical results of water samples, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; µg/L, micrograms per liter; ND, not detected; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequencies (percent)			Median detected concentration (µg/L)‡			Maximum detected concentration (µg/L), and site*		
		Base flow	Storm-flow	Pore water	Base flow	Storm-flow	Pore water	Base flow	Stormflow	Pore water
Plasticizers	Bis(2-ethylhexyl) phthalate (DEHP)	2	23	0	e0.248	e1.33	ND	e0.309, OH-14	2.4, KRM	ND
	Diethyl phthalate	5	26	0	0.21	e0.251	ND	0.3, OH-03	0.46, MRW	ND
	Triethyl citrate	15	15	0	e0.0326	e0.0546	ND	e0.151, MRJ	0.282, MRW	ND
	Triphenyl phosphate	24	44	30	e0.0246	e0.0431	e0.0198	e0.0555, HCW	e0.116, KRM	e0.0481, HCW
	Tris(2-chloroethyl) phosphate (Fyrol CEF)	42	90	90	e0.0522	e0.125	e0.0492	0.87, HCW	0.418, OSM	e0.0787, OSM
Fuels	1-Methylnaphthalene	18	59	60	e0.0101	e0.0312	e0.0389	e0.132, OH-14	0.349, KRM	e0.0794, KRM
	2,6-Dimethylnaphthalene	20	54	100	e0.01	e0.0232	e0.0159	e0.159, OH-14	0.379, KRM	e0.0733, KRM
	2-Methylnaphthalene	16	62	60	e0.0132	e0.0378	e0.0322	e0.184, OH-14	0.377, KRM	e0.105, KRM
	Isopropylbenzene (cumene)	0	3	0	ND	e0.0081	ND	ND	e0.0081, KRM	ND
Solvents	Isophorone	22	100	60	e0.0127	e1.42	e0.0331	e0.0386, MRW	e18.2, OSM	e0.0613, HCW
	Tetrachloroethene (PERC)	22	18	40	e0.0082	e0.0095	e0.0078	e0.0281, LCM	e0.0169, MRW	e0.0116, KRM
Herbicides	Atrazine	69	46	90	e0.0472	e0.123	e0.0358	0.795, OH-03	1.4, MRC	e0.447, LMM
	Bromacil	16	28	30	e0.212	e0.163	0.62	e6.2, LMM	e0.379, KRM	e2.62, LMM
	Dichlorophenyl isocyanate	64	67	90	e0.190	e0.28	e7.51	e115, LMM	e14.7, MRW	e24.3, LMM
	Metalaxyl	0	0	10	ND	ND	e0.061	ND	ND	e0.061, RRG

Table 4a. Organic waste compounds sampled and summary of analytical results of water samples, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; µg/L, micrograms per liter; ND, not detected; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequencies (percent)			Median detected concentration (µg/L)‡			Maximum detected concentration (µg/L), and site*		
		Base flow	Storm-flow	Pore water	Base flow	Storm-flow	Pore water	Base flow	Stormflow	Pore water
Herbicides	Metolachlor	38	46	40	e0.0206	e0.0277	e0.0583	0.706, OH-03	1.38, MRC	e0.242, LMM
	Pentachlorophenol (PCP)	25	77	40	e0.726	e0.457	e0.0666	e3.01, LCM	e3.26, KRM	e0.0796, KRM
	Prometon	16	15	50	e0.0306	e0.0496	e0.0204	e0.0495, OSM	e0.0792, KRM	e0.0409, OSM
Insecticides	Carbaryl	3	18	0	e0.0989	e0.0804	ND	e0.225, UCW	e0.109, MRW	ND
	Carbazole	30	87	100	e0.0208	e0.139	e0.177	e0.0973, UCW	1.56, KRM	1.36, RRG
	Chlorpyrifos	0	3	0	ND	e0.018	ND	ND	e0.018, OSM	ND
	Diazinon	0	0	0	ND	ND	ND	ND	ND	ND
	Dichlorvos	2	3	0	e0.117	e0.0245	ND	e0.216, RRG	e0.0245, OSM	ND
	N, N-diethyl-*meta*-toluamide (DEET)	58	82	90	e0.0538	e0.0996	e0.105	0.293, OSM	e0.776, KRM	e0.165, OSM, RRG
Antimicrobial disinfectants	p-Cresol (*para*-Cresol)	16	33	50	e0.0481	e0.0668	e0.196	0.272, KRM	0.245, KRM	e119, HCW
	Phenol	10	31	30	0.26	e0.162	e0.171	0.511, OH-14	0.549, KRM	6.33, HCW
	Triclosan**	12	3	0	e0.0557	e0.0672	ND	e0.162, LMM	e0.0672, MRC	ND
Detergent metabolites	4-tert-Octylphenol monoethoxylate**	2	10	0	e0.411	e0.664	ND	e0.421, KRM	e1.0, KRM	ND
	4-Cumylphenol**	8	46	60	e0.0299	e0.0559	e0.0959	e0.0436, OSM	0.569, KRM	4.65, LMM
	4-Nonylphenol (sum of all isomers)**	22	74	70	e0.195	e0.71	e0.396	e0.442, MRJ	e3.81, KRM	e12.5, LMM

Table 4a. Organic waste compounds sampled and summary of analytical results of water samples, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; µg/L, micrograms per liter; ND, not detected; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequencies (percent)			Median detected concentration (µg/L)‡			Maximum detected concentration (µg/L), and site*		
		Base flow	Storm-flow	Pore water	Base flow	Storm-flow	Pore water	Base flow	Stormflow	Pore water
Detergent metabolites	4-Nonylphenol diethoxylate**	5	10	30	e1.9	e1.06	e0.662	e3.03, MRJ	e1.96, KRM	e0.749, KRM
	4-Nonylphenol monoethoxylate**	10	0	0	e0.291	ND	ND	e0.568, MRJ	ND	ND
	4-Octylphenol (4-normal-Octylphenol)**	0	0	0	ND	ND	ND	ND	ND	ND
	4-tert-Octylphenol diethoxylate**	3	21	0	e0.496	e0.426	ND	e0.779, KRM	e1.77, KRM	ND
	4-tert-Octylphenol**	1	5	0	e0.032	e0.0454	ND	e0.032, OH-03	e0.0587, KRM	ND
Flavors and fragrances	3-Methyl-1H-indole (skatol)	3	8	60	e0.0055	e0.0071	e0.0354	e0.0115, OSM	e0.0072, KRM	1.5, HCW
	Acetophenone	1	5	0	e0.0382	0.40	ND	e0.0382, KRM	0.482, KRM	ND
	Acetyl hexamethyl tetrahydro naphthalene (AHTN)**	7	0	0	e0.0203	ND	ND	e0.0605, MRJ	ND	ND
	Benzophenone**	7	31	50	e0.0769	e0.0566	e0.0565	e0.116, MRJ	e0.0937, OSM	e0.0781, MRC
	Camphor**	15	49	100	e0.0413	e0.0773	e0.0596	0.266, LCM	e0.171, KRM	0.575, RRG
	d-Limonene**	1	3	20	e0.131	e0.141	e0.0664	e0.131, UCW	e0.141, KRM	e0.0812, KRM
	Hexahydrohexamethyl cyclopentabenzopyran (HHCB)**	31	13	30	e0.0615	e0.0244	e0.0254	0.54, MRJ	e0.0444, MRC	e0.0292, KRM
	Indole	10	10	30	e0.0091	e0.0337	0.99	e0.018, KRM	e0.06, KRM	1.02, MRC
	Isoborneol**	0	0	0	ND	ND	ND	ND	ND	ND
	Isoquinoline**	0	3	13	ND	e0.095	e0.0689	ND	e0.095, KRM	e0.0689, RRG

Table 4a. Organic waste compounds sampled and summary of analytical results of water samples, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; µg/L, micrograms per liter; ND, not detected; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequencies (percent)			Median detected concentration (µg/L)‡			Maximum detected concentration (µg/L), and site*		
		Base flow	Storm-flow	Pore water	Base flow	Storm-flow	Pore water	Base flow	Stormflow	Pore water
Human drugs, nonprescription	Caffeine	52	77	0	e0.105	e0.149	ND	0.66, LCM	0.711, KRM	ND
	Cotinine	2	10	0	e0.0299	e0.0853	ND	e0.0315, MRJ	e0.349, KRM	ND
	Menthol	5	36	0	e0.0401	e0.160	ND	0.31, HCW	0.317, KRM	ND
Sterols	3-beta-Coprostanol	18	31	10	e0.442	e0.569	e1.03	e4.26, LMM	e2.16, MRW	e1.03, KRM
	beta-Sitosterol	24	71	70	e0.924	e1.4	e1.07	e3.15, MRC	e5.08, KRM	e2.45, KRM
	beta-Stigmastanol	7	3	50	e0.664	e0.793	e0.47	1.15, OSM	e0.793, KRM	e0.921, KRM
	Cholesterol	73	77	90	e0.586	e0.917	e1.59	e6.18, LMM	e4.55, KRM	e6.63, RRG
Miscellaneous	1,4-Dichlorobenzene	11	8	50	e0.0395	e0.0093	e0.0297	e0.108, MRJ	e0.0245, OSM	e0.073, LMM
	Methyl salicylate	1	3	0	e0.0206	e0.0158	ND	e0.0206, MRW	e0.0158, OSM	ND
	Tribromomethane (bromoform)	25	8	20	e0.014	e0.0114	e0.0117	e0.0299, KRM	e0.0131, OSM	e0.014, HCW

[*] Site abbreviations are defined in table 1.

[**] Indicates compounds considered by the authors to be domestic wastewater-indicator compounds, likely from sanitary sewer sources.

[‡] Determined from only those samples with detections.

Table 4b. Organic waste compounds with known aquatic toxicity benchmarks for water samples, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; µg/L, micrograms per liter; EPA, U.S. Environmental Protection Agency; --, no data; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Aquatic toxicity benchmarks from EPA		Aquatic toxicity benchmarks from other sources		Percentage of samples exceeding lowest aquatic toxicity benchmark		
		Acute (µg/L)	Chronic (µg/L)	Acute (µg/L)	Chronic (µg/L)	Base flow	Stormflow	Pore water
PAHs	Anthracene	86.1[B]	20.7[B]	13[F]	.012[D]	17	72	90
	Benzo[a]pyrene	3.98[B]	.957[B]	.24[F]	.015[D]	19	92	90
	Fluoranthene	29.6[B]	7.11[B]	3,980[F]	.04[D]	46	95	90
	Naphthalene	803[B]	193[B]	190[F]	1.1[D]	0	0	0
	Phenanthrene	79.7[B]	19.1[B]	30[F]	.4[D]	0	51	50
	Pyrene	42[B]	10.1[B]	--	.025[D]	53	95	90
Plasticizers	Bis(2-ethylhexyl) phthalate (DEHP)	--	--	400[F]	.3[F]	1	23	0
	Diethyl phthalate	--	--	1,800[F]	110[F]	0	0	0
Fuels	1-Methylnaphthalene	--	--	37[F]	2.1[F]	0	0	0
	2-Methylnaphthalene	--	--	--	330[F]	0	0	0
	Isopropylbenzene (cumene)	2,140[A]	420[A]	--	--	0	0	0
Solvents	Isophorone	--	--	117,000[F]	920[F]	0	0	0
	Tetrachloroethene (PERC)	--	--	830[F]	45[F]	0	0	0
Herbicides	Atrazine	--	--	--	1.8[D]	0	0	0
	Bromacil	--	--	--	5.0[D]	1	0	0
	Metolachlor	--	--	--	7.8[D]	0	0	0
	Pentachlorophenol (PCP)	19[C]	15[C]	--	--	0	0	0
Insecticides	Carbaryl	2.1[C]	2.1[C]	3.3[D]	.2[D]	1	0	0
	Chlorpyrifos	.083[C]	.041[C]	.02[D]	.02[D]	0	0	0
	Diazinon	.17[C]	.17[C]	.2[E]	.07[E]	0	0	0
Antimicrobial disinfectants	Phenol	--	--	10,200[F]	180[F]	0	0	0
Detergent metabolites	4-Nonylphenol (sum of all isomers)**	28[C]	6.6[C]	--	1.0[D]	0	28	20
Miscellaneous	1,4-Dichlorobenzene	--	--	180[F]	9.4[F]	0	0	0
	Tribromomethane (bromoform)	--	--	2,300[F]	320[F]	0	0	0

** Indicates compounds considered by the authors to be domestic wastewater-indicator compounds, likely from sanitary sewer sources.

[A] U.S. Environmental Protection Agency, 2008.

[B] U.S. Environmental Protection Agency, 2003.

[C] U.S. Environmental Protection Agency, 2012a.

[D] Canadian Council of Ministers of the Environment, 2012.

[E] Palumbo and others, 2012.

[F] Buchman, 2008.

Table 5a. Organic waste compounds sampled and summary of analytical results of sediment samples, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; µg/kg, micrograms per kilogram; ND, not detected; -, not applicable; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequency (percent)	Median detected concentration (µg/kg)‡	Maximum detected concentration	
				Value (µg/kg)	Site*
Antioxidants	3-tert-Butyl-4-hydroxyanisole (BHA)	0	ND	ND	-
	Bisphenol A	81	e98.42	e1,500	LMM
Dyes/pigments	9,10-Anthraquinone	96	625	1,630	LMM
Fire retardants	BDE congener 47 (tetrabromodiphenyl ether)**	0	ND	ND	-
	Tributyl phosphate**	4	e20.4	e20.4	OSM
	Tris(2-butoxyethyl) phosphate**	11	e161	e324	HCW
	Tris(dichloroisopropyl) phosphate (Fyrol FR 2)**	4	e3.13	e3.13	MRW
PAHs	Anthracene	93	364	1,690	KRM
	Benzo[a]pyrene	96	1,230	3,360	KRM
	Fluoranthene	100	4,370	14,900	MRW
	Naphthalene	71	88.5	278	KRM
	Phenanthrene	100	2,320	7,630	MRW
	Pyrene	100	3,460	10,600	MRW
Plasticizers	Bis(2-ethylhexyl) phthalate (DEHP)	75	567	1,930	KRM
	Diethyl phthalate	4	88.9	88.9	LMM
	Triphenyl phosphate	4	e37.7	e37.7	KRM
	Tris(2-chloroethyl) phosphate (Fyrol CEF)	0	ND	ND	-
Fuels	1-Methylnaphthalene	75	e25.4	129	KRM
	2,6-Dimethylnaphthalene	93	e30.3	e136	KRM
	2-Methylnaphthalene	68	48.9	167	KRM
	Isopropylbenzene (cumene)	4	e2.34	e2.34	LMM
Solvents	Isophorone	18	e13.6	e21.5	LCM
Herbicides	Atrazine	0	ND	ND	-
	Bromacil	0	ND	ND	-
	Metolachlor	0	ND	ND	-
	Prometon	0	ND	ND	-
Insecticides	Carbazole	93	324	810	KRM
	Chlorpyrifos	0	ND	ND	-
	Diazinon	0	ND	ND	-
	N, N-diethyl-*meta*-toluamide (DEET)	0	ND	ND	-
Antimicrobial disinfectants	p-Cresol (*para*-Cresol)	96	e192	2,670	KRM
	Phenol	61	e105	e753	UCW
	Triclosan**	0	ND	ND	-

Table 5a. Organic waste compounds sampled and summary of analytical results of sediment samples, southeast Wisconsin, 2006–9.—Continued

[OWC, organic waste compound; µg/kg, micrograms per kilogram; ND, not detected; -, not applicable; e, estimated; PAH, polycyclic aromatic hydrocarbon; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	Detection frequency (percent)	Median detected concentration (µg/kg)‡	Maximum detected concentration	
				Value (µg/kg)	Site*
Detergent metabolites	4-tert-Octylphenol monoethoxylate**	4	e114	e114	KRM
	4-Cumylphenol**	54	112	1,270	LMM
	4-Nonylphenol (sum of all isomers)**	82	e907	e3,100	MRW
	4-Nonylphenol diethoxylate**	14	e606	e1,520	MRW
	4-Nonylphenol monoethoxylate**	11	e373	e463	MRW
	4-Octylphenol (4-*normal*-Octylphenol)**	0	ND	ND	-
	4-tert-Octylphenol diethoxylate**	0	ND	ND	-
	4-tert-Octylphenol**	21	e29.4	e48.5	KRM
Flavors and fragrances	3-Methyl-1H-indole (skatol)	86	e21.4	74.9	KRM
	Acetophenone	50	e46.6	e220	KRM
	Acetyl hexamethyl tetrahydro naphthalene (AHTN)**	0	ND	ND	-
	Benzophenone**	0	ND	ND	-
	Camphor**	0	ND	ND	-
	d-Limonene**	18	e29.7	e59.5	LCM
	Hexahydrohexamethyl cyclopentabenzopyran (HHCB)**	11	e15.4	e16.3	LCM
	Indole	89	237	717	LCM
	Isoborneol**	0	ND	ND	-
	Isoquinoline**	4	e6.0	e6.0	HCW
Human drugs, nonprescription	Menthol	0	ND	ND	-
Sterols	3-beta-Coprostanol	57	e229	e937	KRM
	beta-Sitosterol	96	e3,670	e13,200	LCM
	beta-Stigmastanol	82	e577	e2,450	MRW
	Cholesterol	96	e1,380	e4,420	KRM
Miscellaneous	1,4-Dichlorobenzene	18	e57.2	e72.3	LCM

‡ Determined from only those samples with detections.

* Site abbreviations are defined in table 1.

** Indicates compounds considered by the authors to be domestic wastewater-indicator compounds likely from sanitary sewer sources.

Table 5b. Organic waste compounds with known aquatic toxicity benchmarks for sediment samples, southeast Wisconsin, 2006–9.

[OWC, organic waste compound; µg/kg, micrograms per kilogram; EPA, U.S. Environmental Protection Agency; --, no data; PAH, polycyclic aromatic hydrocarbon; ISQG, interim sediment quality guideline; AET, apparent effects threshold; PEL, probable effect level; red compound names indicate known or suspected endocrine disruptors, according to The Endocrine Disruption Exchange, Inc. (2011)]

OWC class	Compound name	EPA freshwater sediment screening benchmark† (µg/kg)	Sediment toxicity benchmarks from other sources		Percentage of samples exceeding lowest benchmark
			Value (µg/kg)	Criteria type and source	
PAHs	Anthracene	57.2	46.9	ISQG[D]	82
	Benzo[a]pyrene	150	31.9	ISQG[D]	89
	Fluoranthene	423	111	ISQG[D]	93
	Naphthalene	176	34.6	ISQG[D]	64
	Phenanthrene	204	41.9	ISQG[D]	93
	Pyrene	195	53	ISQG[D]	93
Plasticizers	Bis(2-ethylhexyl) phthalate (DEHP)	180	750	AET[F]	54
	Diethyl phthalate	603	--	--	0
Fuels	2-Methylnaphthalene	20.2	201	PEL[D]	61
	Isopropylbenzene (cumene)	86	--	--	0
Herbicides	Atrazine	6.62	--	--	0
Insecticides	Chlorpyrifos	0.52	--	--	0
	Diazinon	2.39	--	--	0
Antimicrobial disinfectants	p-Cresol (*para*-Cresol)	670	--	--	14
	Phenol	420	48	AET[F]	46
Detergent metabolites	4-Nonylphenol (sum of all isomers)**	--	1,400	ISQG[D]	29
Miscellaneous	1,4-Dichlorobenzene	599	--	--	0

† U.S. Environmental Protection Agency, 2012b.

** Indicates compounds considered by the authors to be domestic wastewater-indicator compounds likely from sanitary sewer sources.

[D] Canadian Council of Ministers of the Environment, 2012.

[F] Buchman, 2008.

Appendix 1.

Appendix 1. Summary of Test Conditions for Conducting Sediment-Toxicity Tests[1].

Parameter	Conditions
Test type	Whole sediment toxicity test with renewal of overlying water
Temperature	23 ± 1 degree Celsius
Light quality	Wide-spectrum fluorescent lights
Illuminance	About 500 to 1,000 lux
Photoperiod	16 light : 8 dark
Test chamber	400 milliliter polypropylene beaker
Sediment volume	100 milliliter
Overlying water volume	175 milliliter
Renewal of overlying water	2 volume additions per day
Age of organisms	Third instar larvae or 9 to 12 days (*Chironomus tentans*)
	7 to 14 days (*Hyallela azteca*)
Number of organisms/ chamber	10
Number of replicates/ treatment	8
Feeding	1.5 milliliter yeast/fish food/cereal daily to each test chamber (*Hyallela azteca*)
	1 milliliter Tetramin flake fish food to each test chamber (1 milliliter contains 4 milligrams of dry solids) (*Chironomus tentans*)
Aeration	None, unless dissolved oxygen in overlying water drops below 40 percent of saturation
Overlying water	Dechlorinated tap water
Test chamber cleaning	If screens become clogged during a test, gently brush the outside of the screen
Overlying water quality	Hardness, alkalinity, conductivity, pH, and ammonia at the beginning and end of a test. Temperature, pH, and dissolved oxygen daily.
Test duration	10 days
Endpoints	Survival and growth (dry weight)
Test acceptability	Minimum mean control survival of 70 percent (*Chironomus tentans*)
	Minimum mean control survival of 80 percent (*Hyallela azteca*)

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[1] U.S. Environmental Protection Agency, 1994.

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