



Screening For Contaminants of Emerging Concern in Surface Waters of the Great Lakes Network Parks

2013–2014

Natural Resource Data Series NPS/GLKN/NRDS—2015/786



ON THE COVER

Coldwater Spring, Mississippi National River and Recreation Area, Minnesota.
Photo by Gordon Dietzman/NPS.

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Abstract

In 2013, the National Park Service Great Lakes Inventory and Monitoring Network (GLKN) joined an ongoing collaborative project with the U.S. Environmental Protection Agency (EPA) Region 8 analytical laboratory in Golden, Colorado, NPS Water Resources Division (WRD), and six other NPS Inventory and Monitoring Networks to sample park surface waters for contaminants of emerging concern (CECs). The CECs for this study are generally classified as pesticides, pharmaceuticals and personal care products, and wastewater indicators. The purpose of this study is to document the occurrence of CECs in surface waters located in and near select GLKN park units, with the objectives of 1) sampling at locations where previous sampling or land use activities indicate CECs may be present, or at locations where baseline data are unavailable, 2) reporting data collected, analytical results and the distribution of detections among sites, and 3) providing NPS with CEC concentration information at low detection limits. GLKN and park staff monitored CECs at four Mississippi River sites and at Coldwater Spring at Mississippi National River and Recreation Area (MISS) in April, June, and November in 2013 and in July and August in 2014. In 2014 CECs were also monitored at seven Lake Superior sites within Apostle Islands National Lakeshore (APIS; June, July, and August) and at 19 sites at Indiana Dunes National Lakeshore (INDU; November). All samples were analyzed for pesticides, pharmaceuticals, and personal care products (212 total analytes); a subset of samples was evaluated for wastewater indicators (52 total analytes). The total number of pesticide, pharmaceutical, and personal care product analytes detected from river, stream, and ditch sites at MISS (44) and INDU (42) were much higher than those detected in the nearshore Lake Superior waters at APIS (5). For analytes detected in at least 50 percent of all samples, pesticides, mainly in the form of herbicides or associated breakdown products, were the most common class of analyte found at MISS and INDU. Gabapentin, a pharmaceutical used to treat epilepsy and neuropathic pain, was widespread in surface waters across all three parks sampled; it was detected in 80, 29, and 58 percent of samples collected at MISS, APIS, and INDU, respectively. Wastewater indicator samples were collected infrequently, but flame retardants, which are one type of wastewater indicator, were detected in samples from all water body types. This indicates that contamination from flame retardants is pervasive and widespread. There was a clear pattern of increasing numbers of CEC detections from upstream to downstream for Mississippi River sites, most likely as a result of contaminant contributions from the Minnesota River, Metro Wastewater Plant, and other point and nonpoint sources within Minneapolis and St. Paul, Minnesota. A seasonal pattern was also apparent for the Mississippi River sites at St. Paul, Newport, and Hastings, Minnesota, with a higher number of detections in early spring and late summer or fall and lower detections in early summer. GLKN is collaborating with the USGS Minnesota Water Science Center to compare concentrations of detected pesticides, pharmaceuticals and personal care products, and wastewater indicators against known environmental thresholds, and where possible, with CEC data from other studies. In 2015 GLKN will focus surface water CEC monitoring activities on up to nine inland lakes at Isle Royale National Park, where the number of CECs is hypothesized to be very low, as these lakes are isolated and generally are only exposed to CECs through atmospheric deposition.

Acknowledgments

In-kind sample analysis was provided by K. Dahlin and his colleagues at the EPA Region 8 analytical laboratory in Golden, Colorado. Project oversight was provided by K. Keteles from the EPA Region 8, and C. Flanagan, P. Penoyer, and K. King from the NPS in Ft. Collins, Colorado. This project would not have been possible without the primary NPS field staff: M. Prosser, R. Damstra, and J. Dickey. R. Key produced maps of the parks and monitoring locations. T. Gostomski assisted with final editing and formatting. B. Route and S. Elliott greatly improved this report with their helpful comments.

Introduction

The National Park Service (NPS) Great Lakes Inventory and Monitoring Network (GLKN) includes nine national park units in the upper Midwest: Voyageurs National Park (VOYA), Mississippi National River and Recreation Area (MISS), St. Croix National Scenic Riverway (SACN), Apostle Islands National Lakeshore (APIS), Grand Portage National Monument (GRPO), Isle Royale National Park (ISRO), Indiana Dunes National Lakeshore (INDU), Pictured Rocks National Lakeshore (PIRO), and Sleeping Bear Dunes National Lakeshore (SLBE) (Figure 1). As a program of the NPS, GLKN staff conduct long-term monitoring of critical park resources, or vital signs, at these national parks.

Fresh water is an internationally important recreational, commercial, political, and ecological resource shared by these nine parks. GLKN and park staff monitor a number of important water-related vital signs through implementation of monitoring protocols for inland lakes (Elias et al. 2008), large rivers (Magdalene et al. 2008), diatoms (Ramstack et al. 2008), and wadeable streams (in development). However, vital signs monitored through these protocols do not include monitoring surface waters for the presence of pesticides, pharmaceuticals, personal care products, and wastewater indicators (collectively referred to as “Contaminants of Emerging Concern” or “CECs” hereafter). Because many GLKN parks are potentially impacted by adjacent agriculture, mineral exploration and extraction, atmospheric deposition, and waste water treatment discharge, it is important to assess if anthropogenic contaminants are present in park waters and to identify potential sources of contamination to these protected areas.

In 2013, GLKN joined an ongoing collaborative project with the U.S. Environmental Protection Agency (EPA) Region 8 analytical laboratory in Golden, Colorado, NPS Water Resources Division (WRD), and six other NPS Inventory and Monitoring (I&M) Networks to sample park surface waters for CECs. For this project, I&M or park staff collected samples, the EPA Region 8 laboratory analyzed the samples and provided results, WRD staff managed and archived the data, and I&M staff interpreted data and reported results to parks within their respective networks.

Surface water sampling allows for the characterization and assessment of surface water quality conditions in relation to contaminant sources. Data support establishment of CEC baseline levels, impacts, impairments or levels of degradation from some potential sources and identification of the need for responsive public and environmental health decisions. The purpose of this study is to document the occurrence of CECs in surface waters located in and near select GLKN park units. Objectives include:

- Collect high quality surface water samples at locations where previous sampling or land use activities indicate CECs may be present, or at locations where baseline data are unavailable.
- Report data collected, analytical results and the distribution of detections among sites.
- Provide NPS with CEC concentration information at low detection limits.



Figure 1. Locations of nine national parks in the Great Lakes Inventory and Monitoring Network. Parks monitored for contaminants in surface waters (highlighted in yellow) include Mississippi National River and Recreation Area (2013 and 2014), Apostle Islands National Lakeshore (2014), and Indiana Dunes National Lakeshore (2014).

The number and type of samples allocated to GLKN for this project is negotiated annually by representatives from EPA, NPS WRD, and NPS I&M. Because the EPA sample analysis is provided in-kind, the overall number of samples allotted and the timing of sample collection assigned to each I&M Network in any given year is conservative. In 2013 GLKN was assigned to monitor CECs in April, June, and November. MISS was selected for monitoring because of 1) the availability of ice-free surface water in April and November, and 2) the existence of previous (Meade 1995, Fallon et al. 1997, Lee et al. 2008, Wiener and Sandheinrich 2010) and ongoing (Route et al. 2014a, 2014b; MCES 2015) studies of the same or similar CECs in or near MISS that might be comparable to those assessed in this project.

In 2014 GLKN targeted nearshore waters of Lake Superior within APIS for monitoring CECs in June, July, and August. APIS was selected for monitoring because 1) although Lake Superior is

influenced by both point (e.g., wastewater treatment plants) and nonpoint source (e.g., atmospheric deposition) contaminant inputs, CECs analyzed in this study are not routinely studied within park boundaries, and 2) fewer numbers of CECs were expected to be detected at APIS, which would provide a contrast to the relatively higher number of contaminants previously detected at MISS in 2013.

Despite the limited availability of information on contaminants in the nearshore waters of Lake Superior within APIS, some contaminants monitoring work in surface water, sediment, macroinvertebrates, and fish (WDNR 1977, Strachan and Glass 1978, Kraft et al. 2007, Hobbs et al. in press), as well as bald eagles (Route et al. 2014a, 2014b) has taken place, which might be comparable to results obtained from this project.

In 2014, some additional -and previously unplanned- monitoring also took place at MISS (July and August) and INDU (November) when samples and associated months opportunistically became available. Monitoring at MISS took place at the same sites as in 2013 to screen for CECs during previously unmonitored months. This was completed in order to obtain a better overall understanding of spatial and seasonal patterns for detected contaminants on the Mississippi River. INDU was chosen for monitoring because of its urban setting and close proximity to a number of industries that have the potential to introduce CECs to park surface waters (Arihood 1975, Hardy 1983, Egler et al. 2013) or to tributaries of the park (Lafrancois and Glase 2005).

Methods

The following paragraphs outline rationale used to select sites at each park, methods used to collect and analyze samples for contaminants, and data management and summarization. A complete description of field and laboratory methods can be found in the Quality Assurance Project Plan for this CEC screening effort (NPS 2014)

Site Selection

Four mainstem Mississippi River sites (identified by site names that begin with “UM” and include numbers) and one natural spring site were selected for monitoring at MISS (Figure 2) through consultations among GLKN, MISS, and U.S. Geological Survey (USGS) Minnesota Science Center staff, for the following reasons:

- UM883 (Elk River, MN) – site is just upstream of the park boundary and integrates all upstream conditions over a mix of different land uses.
- UM839 (St. Paul, MN) – site is downstream of the confluence of the Minnesota River but upstream of the Metropolitan Wastewater Treatment Plant (Metro Plant) and integrates all upstream conditions, including contributions from the Minnesota River and its associated agricultural inputs.
- UM831 (Newport, MN) – site is downstream of the Metro Plant and integrates all upstream conditions, including contributions from the Metro Plant, which potentially is a major point source of CECs.
- UM815 (Hastings, MN) – site is a few miles upstream of the park’s downstream boundary and integrates all upstream conditions at MISS just above the confluence of the St. Croix River
- Coldwater Spring (Fort Snelling, MN) – site is a natural spring on a recently acquired park property. The spring is culturally significant and has been a source of drinking water for hundreds of years.

Seven Lake Superior nearshore monitoring sites at APIS (Figure 3) were selected through consultation with GLKN and park staff, for the following reasons:

- APIS09-84 (Long Island) - site is within an active bald eagle territory where persistent and bioaccumulative CECs are being monitored in bald eagle nestlings (Route et al. 2014a, 2014b), and CECs in water, sediment, and fish were previously monitored by the EPA (Hobbs et al. in press). Collection of additional baseline contaminant data is warranted in the area prior to potential mining activities in the headwaters of the Bad River watershed.

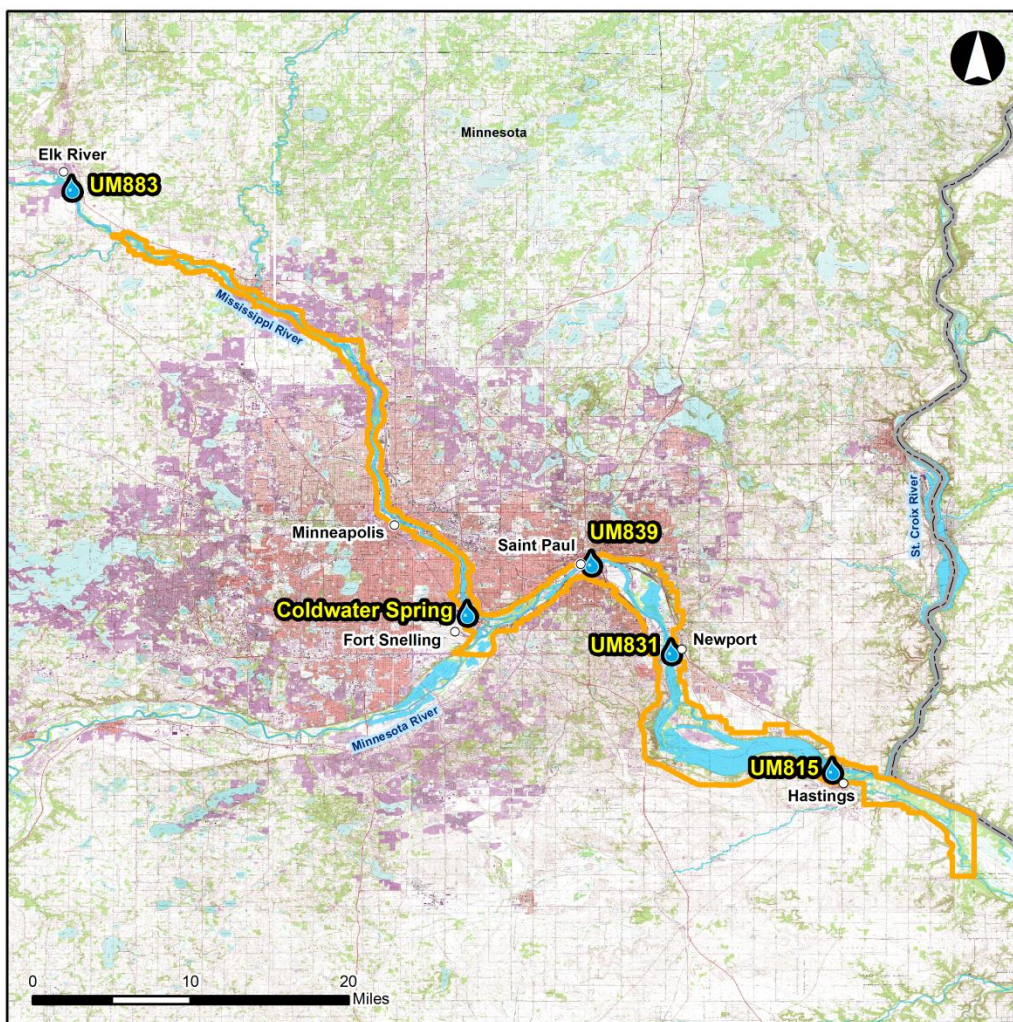


Figure 2. Surface water contaminant monitoring locations (water drops) at Mississippi National River and Recreation Area in 2013 and 2014. Park boundary shown in orange.

- APIS09-14 (Michigan Island) - site is within an active bald eagle territory where persistent and bioaccumulative CECs are being monitored. Higher levels of fat-soluble CECs have been documented in eagle nestlings from nests on islands farther from the mainland (Route et al. 2014a, 2014b), and CECs in water, sediment, and fish were previously monitored by the EPA. The site is also within an important lake trout spawning area (no fishing around the island).
- APIS09-03, 62, 30, 09 (Bear, Rocky, and Devils islands, and Little Sand Bay, respectively) – sites are within an active bald eagle territory where contaminants are being monitored. Higher levels of fat-soluble CECs have been documented in eagle nestlings compared to other eagle monitoring sites at APIS, and CECs in water, sediment, and fish have been previously monitored by the EPA.

- APIS-Outer (Outer Island) - site is within an active bald eagle territory where CECs are being monitored. The highest levels of fat-soluble CECs have been documented in eagle nestlings (Route et al. 2014a, 2014b) at APIS.



Figure 3. Surface water contaminant monitoring locations (water drops) at Apostle Islands National Lakeshore in 2014. Park boundary shown in orange.

Nineteen stream, river, or ditch sites at INDU (Figure 4) were selected and monitored by park staff for the following reasons (J. Dickey, pers. comm.):

- INDU029, 459, 475, 503, 539, 900, 901, 902, 903, 904, 905, and 906 - sites are part of an ongoing monitoring effort of the East Branch of the Little Calumet River (EBLC); sections of which run through INDU before entering Lake Michigan via Burns Waterway. Many of these sites have been sampled within the past three to four years for any combination of fish, macroinvertebrates, water chemistry, nutrient levels, stream flow, woody debris content, and sediment transport. Federal and state agencies, educational institutions, and non-profit

organizations have sampled at these sites because of their ease of access and relative contributions to the EBLC watershed.

- INDU082, 144, 164, 243, 262, 328, and 405 - sites are independent of the EBLC and feed directly into Lake Michigan. Sites include Brown Ditch, Derby Ditch, and Dunes Creek. Brown and Derby ditch discharge into Lake Michigan on INDU property. These sites may be potential sources of CECs because of contributions from nearby roads, residences, or other human activities (e.g., effluent discharge into Dunes Creek from a recently established waterpark) and are being monitored for at least fish and water chemistry.

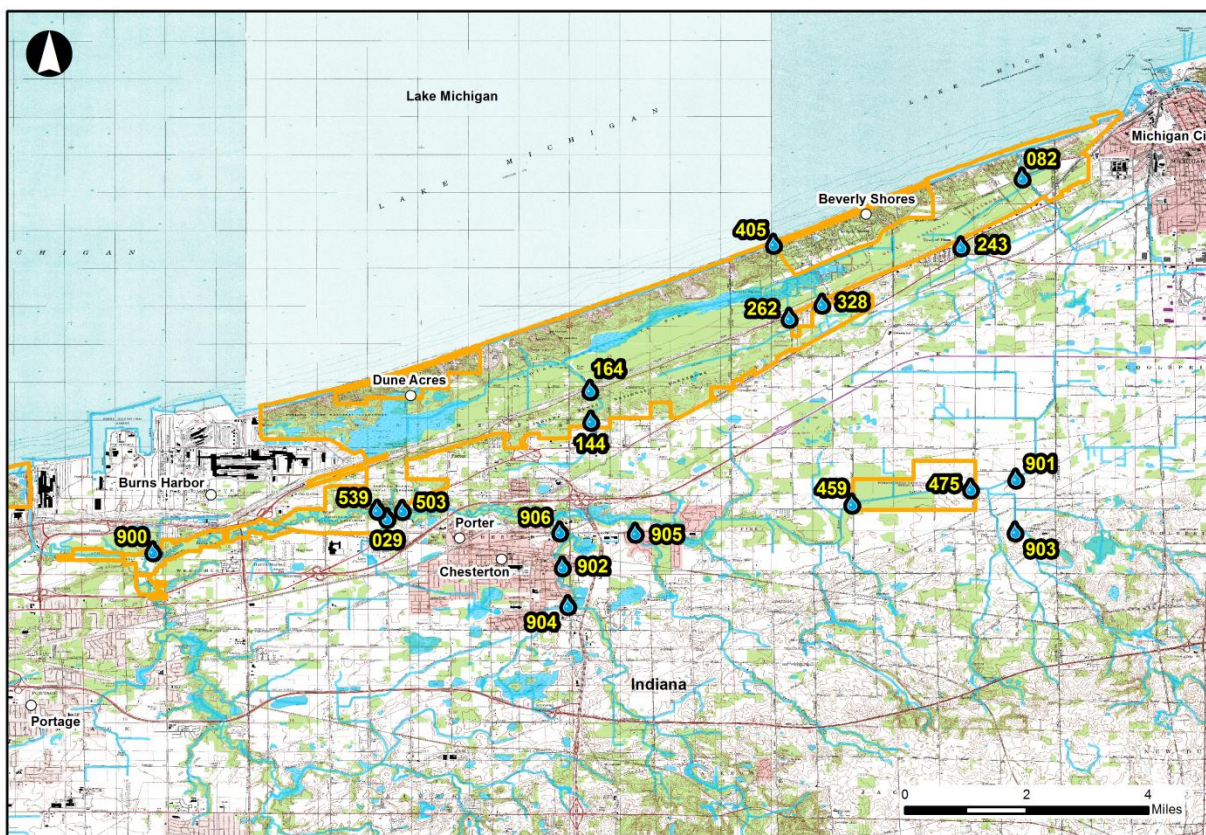


Figure 4. Surface water contaminant monitoring locations (water drops) at Indiana Dunes National Lakeshore in 2014. Park boundary shown in orange.

Field Sampling

In 2013, samples were collected at five locations within MISS in April, June, and November. In 2014, samples were collected at: seven locations within APIS in June, July, and August; the same five MISS locations that were sampled in 2013 in July and August; and 19 sites within INDU in November. Samples were collected for pesticides, pharmaceuticals, and personal care products at all sites during each monitoring event. Samples were also collected for wastewater indicator compounds at a subset of parks and sites (Table 1).

Table 1. Pesticide, pharmaceutical, and personal care product samples (shown with an “X”) collected at sampling sites in the Great Lakes Inventory and Monitoring Network parks in 2013 and 2014. Bold X’s indicate when wastewater indicator samples were also collected. Dash (-) indicates samples not collected. MISS = Mississippi National River and Recreation Area, APIS = Apostle Islands National Lakeshore, INDU = Indiana Dunes National Lakeshore.

Park	Year (s)	Site	Water body	April	June	July	Aug	Nov
			type					
MISS	2013/2014	Coldwater Spring	spring	X/-	X/-	-/X	-/X	X/-
		UM883B10	river	X/-	X/-	-/X	-/X	X/-
		UM839B10	river	X/-	X/-	-/X	-/X	X/-
		UM831B10	river	X/-	X/-	-/X	-/X	X/-
		UM815B10	river	X/-	X/-	-/X	-/X	X/-
APIS	2014	APIS_Outer	Great Lake	-	X	X	X	-
		APIS09-03	Great Lake	-	X	X	X	-
		APIS09-09	Great Lake	-	X	X	X	-
		APIS09-14	Great Lake	-	X	X	X	-
		APIS09-30	Great Lake	-	X	X	X	-
		APIS09-62	Great Lake	-	X	X	X	-
		APIS09-84	Great Lake	-	X	X	X	-
INDU	2014	INDU029	ditch	-	-	-	-	X
		INDU082	ditch	-	-	-	-	X
		INDU144	stream	-	-	-	-	X
		INDU164	stream	-	-	-	-	X
		INDU243	ditch	-	-	-	-	X
		INDU262	ditch	-	-	-	-	X
		INDU328	ditch	-	-	-	-	X
		INDU405	ditch	-	-	-	-	X
		INDU459	river	-	-	-	-	X
		INDU475	river	-	-	-	-	X
		INDU503	river	-	-	-	-	X
		INDU539	river	-	-	-	-	X
		INDU900	ditch	-	-	-	-	X
		INDU901	ditch	-	-	-	-	X
		INDU902	stream	-	-	-	-	X
		INDU903	ditch	-	-	-	-	X
		INDU904	ditch	-	-	-	-	X
		INDU905	stream	-	-	-	-	X
		INDU906	stream	-	-	-	-	X

Sample bottles, labels, and chain of custody forms were provided by the EPA laboratory. Field staff were instructed to minimize use of products like caffeine, fragrances, and insecticides prior to sampling, and clean gloves were donned for each sample. Bottles were rinsed with source water

twice before collecting a grab sample of the source water. The bottles were capped underwater to minimize exposure to air, when possible. Sample bottles were labeled, put on ice, refrigerated to 4°C, and shipped overnight to the EPA laboratory as soon as possible.

Field replicate pesticide and pharmaceutical and personal care product (PPCP) samples were collected during each round of monitoring and comprised approximately 10% of the total number of samples collected. Wastewater indicator field replicate samples and field blank samples were not collected due to the limited number of total samples allocated to GLKN. Other water quality parameters, including temperature, pH, dissolved oxygen, and specific conductivity, were collected at most of the sites across both years using a Hydrolab, Eureka, or YSI multiprobe sonde.

Laboratory Analysis

All samples were processed and analyzed by the EPA Region 8 Laboratory in Golden, CO. Laboratory analysis methods used to analyze the samples are shown in Table 2. Pesticide and PPCP samples were filtered and the dissolved fraction was analyzed. Whole water samples were analyzed for wastewater compounds. Samples were analyzed for 72 pesticide, 140 PPCP, and 53 wastewater analytes. All analytes and their respective detection and reporting limits are shown in Appendix A. Laboratory quality assurance and quality control checks for this project included analysis of laboratory matrix spikes, matrix spike duplicates, blanks, controls, and calibration samples, with results typically expressed as the relative percent difference (RPD) between two samples. For pesticides, the acceptance criteria are +/- 20% RPD for matrix blanks, spikes, and duplicates. For PPCPs and wastewater indicators, the acceptance criteria are +/- 30% RPD for matrix blanks, spikes, and duplicates.

Table 2. Analysis methods and sample requirements for the EPA Region 8 laboratory.

Parameter/Analytical Method	Sample Volume	Container Size and Type	Preservation	Maximum Holding Time	Method Reporting Limits (ng/L)
Pesticides by Liquid Chromatography – Mass Spectrometry (LCMS), EPA Method 8321A, EPA R8 Laboratory SOP ORGM-550	40 ml	Sterile 40 ml amber glass volatile organic analysis (VOA) vials	Ice to 2°C –6°C	7 Days	10–50
PPCPs by LCMS, EPA Method 1694, EPA R8 Laboratory SOP LCMS PPCPs Electrospray Ionization (ESI)+ and LCMS PPCPs ESI-	40 ml	Sterile 40 ml amber glass VOA vials	Ice to 2°C –6°C	7 Days	10–25
Waste Water Indicator Compounds by Gas Chromatography – Mass Spectrometry, EPA R8 Laboratory SOP ORGM-006	250 mL	Sterile 250 ml amber glass VOA vials	Ice to 2°C –6°C	7 days to extraction	50

Data Management and Analysis

Data were provided by the EPA lab to both the GLKN and to the NPS WRD. All data, including sample results and field and laboratory quality assurance/quality control (QA/QC) results, were reviewed by the GLKN. Site information including park, site description, GPS coordinates, and sample collection dates were provided to the NPS WRD. The NPS WRD imported the data and site metadata into the NPS's relational water quality database, NPSTORET, and transferred data from NPSTORET into the EPA STORET Data Warehouse for archival. The data were also provided by the NPS WRD to the GLKN in NPSTORET format. Additional data such as temperature, pH, dissolved oxygen, specific conductivity, and photographs were maintained internally at the GLKN's office in Ashland, Wisconsin.

Data were summarized by park, year and month sampled, detected vs. non-detected analytes, specific analyte, and by the number of analytes detected by site. Total number of detections by park, year sampled, and by site was tallied. We then calculated within-park percent occurrence by analyte and as a percentage of the total number of analytes analyzed. For MISS, the number of analytes detected was plotted by site and month across 2013 and 2014 to allow for qualitative analysis of seasonal and upstream to downstream spatial patterns.

Results

The methods used by EPA Region 8 Laboratory allow for detection of CECs at extremely low levels with reporting limits typically ranging from 10 to 50 ng/L (or parts per trillion), depending on the analyte (Table 2). Of the analytes detected in surface water samples across all three parks, herbicides were more commonly detected. Nearly all of the other detected analytes were at levels just above their respective reporting limits. Some exceptions were: gabapentin, a drug used to treat epilepsy and neuropathic pain; DEET, a commonly used insect repellent; and caffeine, a commonly used mild stimulant. When detected, concentrations of these three analytes often ranged from 100 to 1000 ng/L (1 part per billion).

Concentrations and associated reporting limits for all detected analytes are shown in Appendix B. GLKN is partnering with the USGS Minnesota Water Science Center to compare CEC results from this study to: 1) concentrations known to have chronic or acute impacts to aquatic organisms, 2) results from other similar studies within or near the three parks included in this monitoring effort, and 3) draft a manuscript for submission to a peer-reviewed journal if findings from #1 or #2 are noteworthy. Park-specific results focusing on total number of pesticide, PPCP, and wastewater indicator detections, as well as types and typical uses of detected analytes follow.

Mississippi National River and Recreation Area (MISS)

At MISS, a total of 44 pesticides and PPCPs (21 percent of the total number analyzed) were detected at all sites throughout the sampling periods (Table 3). Of the 44 detected analytes, only three (caffeine, DEET, and metolachlor ESA) were detected at Coldwater Spring. These three compounds were frequently found in samples collected at the four river sites (40, 48, and 100 percent, respectively). Twelve of the 44 detected pesticides and PPCPs were found in at least 50 percent of the samples spanning both years (Table 3). Metolachlor ESA, a breakdown product of the herbicide metolachlor, was detected in 100 percent of the samples collected across all five monitoring locations. Two herbicides, 2, 4-D and atrazine, and two pharmaceuticals, gabapentin and metformin, were detected in all of the samples collected at the four Mississippi River monitoring locations.

In general, the number of pesticides and PPCPs detected at the four river sites was high in the spring and fall and lower in the summer (Figure 5). This pattern was especially evident for the two downstream sites at Newport and Hastings, MN. The number of detections increased steadily from upstream to downstream for all the river sites across all sampling months, with the median number of detections ranging from 10 at Elk River, Minnesota (UM883), to 27 at Hastings, Minnesota (UM815). In comparison, the number of pesticides and PPCPs detected at Coldwater Spring was low and did not exhibit a seasonal pattern. The number of detections across all sites and both years, expressed as percent of the total number of contaminants analyzed, ranged from three to 14 percent for the river sites, and from 0 to 1 percent for Coldwater Spring.

Table 3. Analyte description, number of detections, and percent occurrence of pesticides, pharmaceuticals and personal care products from five sites at Mississippi National River and Recreation Area in 2013 and 2014. n = number of samples.

Analyte	Usage Description	Number of Detections		
		2013 (n=15)	2014 (n=10)	Percent Occurrence in Samples
Metolachlor ESA	Degradate of metolachlor, herbicide	15	10	100
2,4-D	Chlorophenoxy acid herbicide, in Weed-B-Gone®	12	8	80
Atrazine	Triazine herbicide for broad-leaf weeds	12	8	80
Gabapentin	Epileptic treatment and neuropathic pain reliever	12	8	80
Metformin	Anti-diabetic drug	12	8	80
Acetochlor OA	Acetochlor degradate, pre-emergent herbicide	11	8	76
Imazethapyr	Broad-spectrum weed herbicide	6	8	56
Metolachlor	Herbicide for grasses and broad-leaf weeds	7	7	56
DEET	Pest repellent	4	9	52
Desmethylvenlafaxine	Anti-depressant (Pristique®)	8	5	52
Hydrochlorothiazide	Diuretic	7	6	52
Lamotrigine	Anti-convulsant	9	4	52
Caffeine	Mild central nervous system stimulant	9	2	44
Sulfamethoxazole	Sulfonamide anti-bacterial	7	4	44
Acetochlor	Pre-emergent herbicide	6	4	40
Dimethenamid	Widely-used herbicide	6	3	36
Atenolol	Anti-hypertensive (beta blocker)	6	1	28
Dimethenamid ESA	Dimethenamide degradate, herbicide	4	3	28
Gemfibrozil	Lipid regulator	5	2	28
Atrazine de-ethyl	Degradate of atrazine, herbicide	3	3	24
Bentazon	Thiadiazine herbicide	1	5	24
Hydroxybupropion	Bupropion degradate, anti-depressant	6	0	24
Tramadol	Opioid analgesic for arthritis and fibromyalgia	4	2	24
Ranitidine	Heartburn medication (Zantac®)	5	0	20
Trimethoprim	Anti-biotic	3	2	20
Carbamazepine	Anti-convulsant	2	2	16
Furosemide	Diuretic	4	0	16
Levorphanol	Synthetic opioid, morphine-like analgesic	4	0	16
Metoprolol	Anti-hypertensive	2	2	16
Venlafaxine	Anti-depressant for anxiety attacks or major depressive disorder	2	2	16
Atrazine de-isopropyl	Degradate of atrazine, herbicide	0	3	12
Clothianidin	Neonicotinoid insecticide	0	3	12
Lidocaine	Local anesthetic, anti-arrhythmic	2	1	12
Naproxen	Nonsteroidal anti-inflammatory drug	3	0	12
Sotalol	Anti-arrhythmic medication	1	2	12
Theobromine	Found in chocolate and tea, similar to caffeine	3	0	12

Table 3 (continued). Analyte description, number of detections, and percent occurrence of pesticides, pharmaceuticals and personal care products from five sites at Mississippi National River and Recreation Area in 2013 and 2014. n = number of samples.

Analyte	Usage Description	Number of Detections		
		2013 (n=15)	2014 (n=10)	Percent Occurrence in Samples
Triclopyr	Pyridinyl foliar herbicide, defoliant	3	0	12
10,11-dihydro-10-hydroxycarbamazepine	Degradate of Oxcarbazepine (anti-epileptic drug)	0	2	8
Methylparaben	Antifungal preservative	0	2	8
Propachlor ESA	Propachlor degradate, herbicide	0	2	8
Modafinil acid	Modafinil degradate, wakefulness stimulant	1	0	4
Pregabalin	Anti-convulsant	1	0	4
Thiamethoxam	Neonicotinoid insecticide, banned in EU	0	1	4
Triamterene	Diuretic for treatment of hypertension and edema	1	0	4

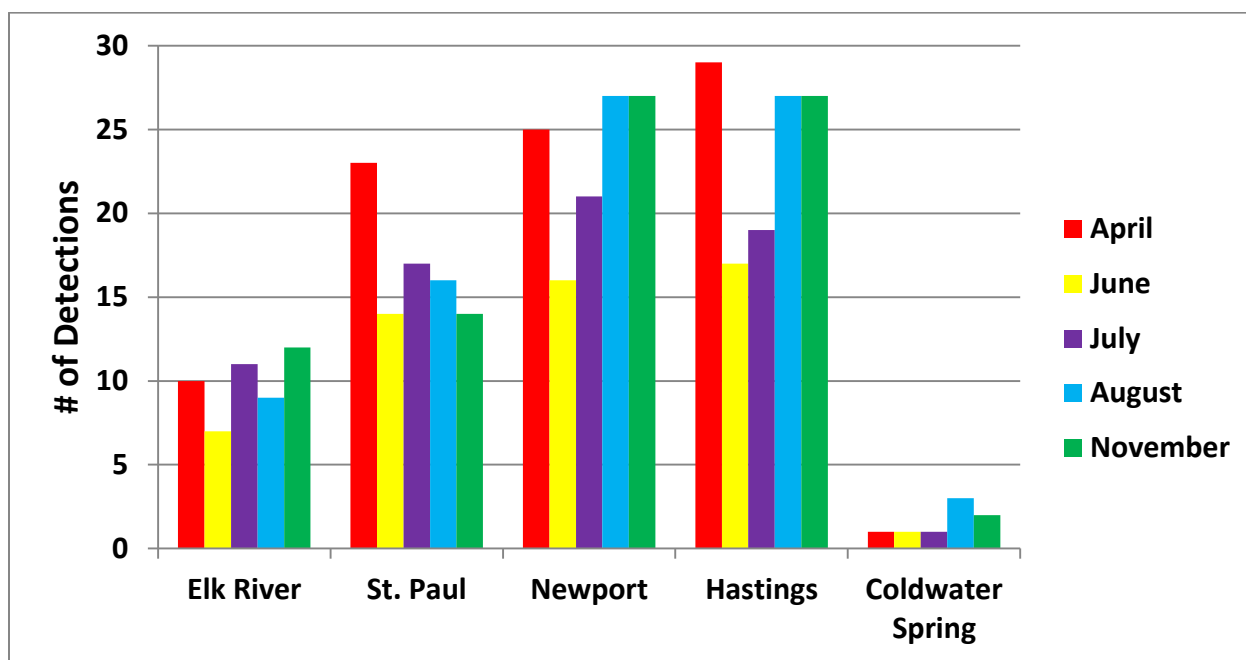


Figure 5. Number of detections for pesticides, pharmaceuticals and personal care products from five sites at Mississippi National River and Recreation Area in 2013 (April, June, and November) and 2014 (July and August). Mississippi River sites (Elk River, St. Paul, Newport, and Hastings) are arranged upstream to downstream, left to right.

Wastewater indicator samples were only collected in 2013 at the Mississippi River sites in Elk River and Newport, Minnesota, and Coldwater Spring. A total of six wastewater indicator analytes were detected across the three sites (Table 4). Tri(2-butoxyethyl) phosphate and tri(dichloroisopropyl) phosphate, two flame retardants, and bisphenol A, a substance commonly found in plastic, were found in 67, 44, and 33 percent of wastewater indicator samples, respectively. The total number of

detections was highest (6) at the Mississippi River site in Newport, Minnesota (Figure 6). The lowest numbers of detections were at the river site in Elk River, Minnesota (3) and Coldwater Spring (2). The number of detections expressed as percent of the total number of analytes analyzed ranged from two to 11 percent for the two river sites, and was at two percent all three months for Coldwater Spring. Seasonal and spatial patterns were not assessed for detected wastewater indicators due to the low number of monitoring sites and samples.

Table 4. Analyte description, number of detections, and percent occurrence of wastewater indicators from five sites at Mississippi National River and Recreation Area in 2013. n = number of samples.

Analyte	Usage Description	Number of Detections (n=9)	Percent Occurrence in Samples
Tri(2-butoxyethyl) Phosphate	flame retardant	6	67
Tri(dichloroisopropyl) Phosphate	flame retardant	4	44
Bisphenol A	plastics, plasticizer	3	33
Galaxolide	fragrance	2	22
Tributyl Phosphate	extractant, plasticizer, hydraulic fracturing	2	22
Triethyl Citrate	food additive, plasticizer	1	11

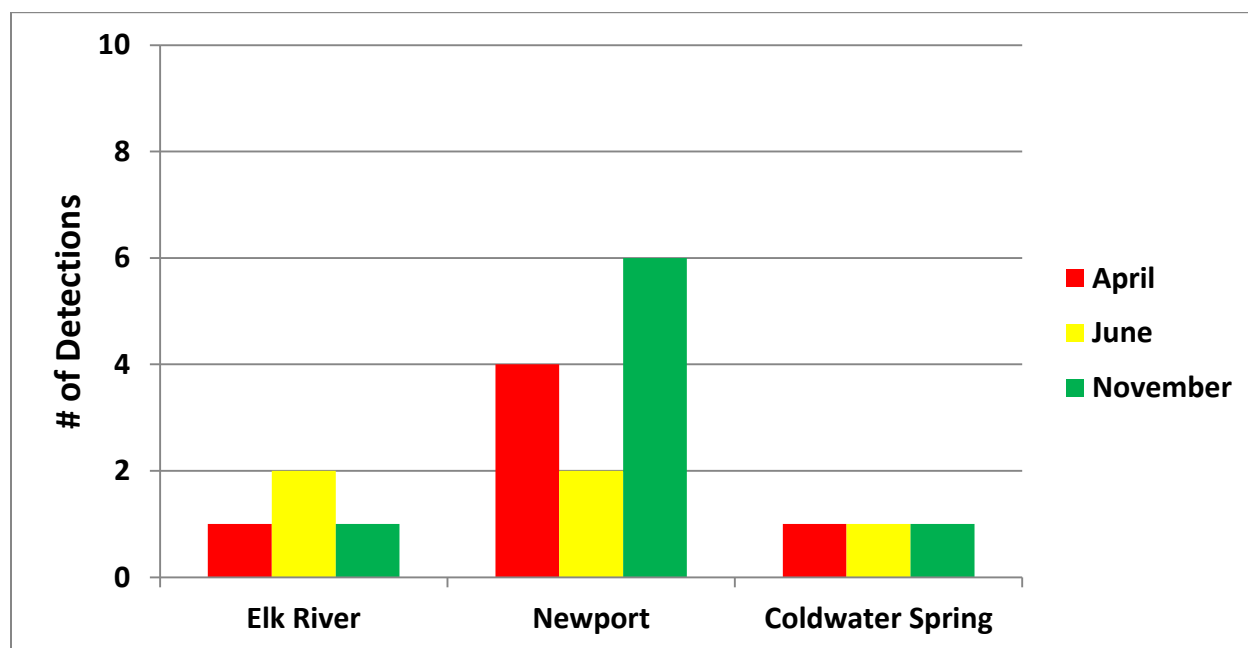


Figure 6. Number of detections for wastewater indicators from three sites at Mississippi National River and Recreation Area in 2013 (April, June, and November). Mississippi River sites (Elk River and Newport) are arranged upstream to downstream, left to right.

Apostle Islands National Lakeshore (APIS)

At APIS, a total of five pesticides and PPCPs (2 percent of the total number analyzed) were detected at all Lake Superior sites in 2014 (Table 5). Of the five detected analytes, Gabapentin and imazethapyr were found in 29 and 14 percent of samples, respectively. The remaining three pesticides and PPCPs were only detected once. A total of four wastewater indicators (8 percent of the total number analyzed) were detected. Of those detected, the flame retardant tri(2-butoxyethyl) phosphate was detected in 50 percent of the samples; the remaining wastewater indicators were only detected once. The number of pesticide, PPCP, and wastewater indicator detections by site and month are shown in Table 6; through qualitative assessment, seasonal and spatial patterns were not readily evident.

Table 5. Analyte description, number of detections, and percent occurrence of pesticides, pharmaceuticals and personal care products (PPCPs) and wastewater indicators at Apostle Islands National Lakeshore in 2014. Pesticide and PPCP samples collected at seven sites in June, July, and August. Wastewater indicator samples collected at four sites in June and July. n = number of samples.

	Analyte	Usage Description	Number of Detections	Percent Occurrence in Samples
Pesticides and PPCPs (n=21)	Gabapentin	Epileptic treatment and neuropathic pain reliever	6	29
	Imazethapyr	Broad-spectrum weed herbicide	3	14
	Caffeine	Mild central nervous system stimulant	1	5
	DEET	Pest repellent	1	5
	Metformin	Anti-diabetic drug	1	5
Wastewater Indicators (n=8)	Tri(2-butoxyethyl) Phosphate	Flame retardant component	4	50
	Bisphenol A	Used in plastic; contains hormone-properties	1	13
	Tri(dichloroisopropyl) Phosphate	Flame retardant component	1	13
	Triclosan	Antibacterial and antifungal	1	13

Table 6. Number of pesticides, pharmaceuticals, personal care products, and wastewater indicators (in parenthesis) detected each month at seven sites in Apostle Islands National Lakeshore in 2014.

Site ID	Site Description	June	July	August
APIS09-84	Lake Superior near Long Island	0 (4)	0 (3)	1
APIS09-14	Lake Superior near Michigan Island	2	0	0
APIS09-03	Lake Superior near Bear Island	0	0	1
APIS_Outter	Lake Superior near Outer Island	0 (2)	1 (3)	1
APIS09-62	Lake Superior near Rocky Island	1	0	1
APIS09-30	Lake Superior near Devils Island	0 (3)	0 (3)	1
APIS09-09	Lake Superior near Little Sand Bay	0 (2)	2 (3)	1

Indiana Dunes National Lakeshore (INDU)

At INDU, a total of 42 pesticides and PPCPs (20 percent of the total number analyzed) were detected at 19 stream, river, and ditch sites during a single monitoring event in November 2014 (Table 7).

Seven of the 42 pesticides and PPCPs were detected in at least 50 percent of the samples.

Metolachlor ESA, a breakdown product of the herbicide metolachlor, and DEET, an insect repellent, were detected in 95 percent of the samples collected across all 19 monitoring locations. Of the remaining five pesticides and PPCPs detected in at least 50% of the samples, four were herbicides or breakdown products of herbicides and one was the pharmaceutical gabapentin.

Table 7. Analyte description, number of detections, and percent occurrence of pesticides, pharmaceuticals and personal care products from 19 sites at Indiana Dunes National Lakeshore in November, 2014. n = number of samples.

Analyte	Usage Description	Number of Detections (n=19)	Percent Occurrence in Samples
DEET	Pest repellent	18	95
Metolachlor ESA	Degradate of metolachlor, herbicide	18	95
2,4-D	Chlorophenoxy acid herbicide, in Weed-B-Gone®	17	89
Imazethapyr	Broad-spectrum weed herbicide	16	84
Acetochlor OA	Acetochlor degradate, pre-emergent herbicide	13	68
Gabapentin	Epileptic treatment and neuropathic pain reliever	11	58
Simazine	Triazine herbicide	10	53
Atrazine	Triazine herbicide for broad-leaf weeds	8	42
Caffeine	Mild central nervous system stimulant	7	37
Triclopyr	Pyridinyl foliar herbicide, defoliant	6	32
Metformin	Anti-diabetic drug	5	26
Acetaminophen	Analgesic	4	21
Cotinine	Nicotine degradate, antidepressant	3	16
Dimethenamid ESA	Dimethenamide degradate, herbicide	3	16
Hydrochlorothiazide	Diuretic	3	16
MCP	2,4-D degradate, herbicide	3	16
Meprobamate	Anxiolytic drug, minor tranquilizer	3	16
Atrazine de-ethyl	Degradate of atrazine, herbicide	2	11
Atrazine de-isopropyl	Degradate of atrazine, herbicide	2	11
Desmethylvenlafaxine	Anti-depressant (Pristique®)	2	11
Furosemide	Diuretic	2	11
Hydroxybupropion	Bupropion degradate, anti-depressant	2	11
Lamotrigine	Anti-convulsant	2	11
Levorphanol	Synthetic opioid, morphine-like analgesic	2	11
Metolachlor	Herbicide for grasses and broad-leaf weeds	2	11
Naproxen	Nonsteroidal anti-inflammatory drug	2	11

Table 7 (continued). Analyte description, number of detections, and percent occurrence of pesticides, pharmaceuticals and personal care products from 19 sites at Indiana Dunes National Lakeshore in November, 2014. n = number of samples.

Analyte	Usage Description	Number of Detections (n=19)	Percent Occurrence in Samples
Sotalol	Anti-arrhythmic medication	2	11
Tebuthiuron	Dimethylurea herbicide	2	11
Tramadol	Opioid analgesic for arthritis and fibromyalgia	2	11
Valsartan	Treatment for high blood pressure/congestive heart failure	2	11
2,4,5-TP	Chlorophenoxy acid herbicide	1	5
Atenolol	Anti-hypertensive (beta blocker)	1	5
Clothianidin	Neonicotinoid insecticide	1	5
Dichloroprop	Chlorophenoxy acid herbicide for broad-leaf weeds	1	5
Diclofenac	Non-steroidal anti-inflammatory drug (NSAID)	1	5
Imazaquin	Broad-spectrum weed herbicide	1	5
Metalaxyl	Phenylamide systemic fungicide	1	5
Methylparaben	Antifungal preservative	1	5
Monuron	Phenylurea herbicide	1	5
Propachlor OA	Propachlor degradate, herbicide	1	5
Sulfamethoxazole	Sulfonamide anti-bacterial	1	5
Venlafaxine	Anti-depressant	1	5

The number of pesticide and PPCP analytes detected by site ranged from 20 at the East Branch of the Little Calumet River at Mineral Springs (INDU503) and Carver Ditch (INDU901) to a single analyte detected at Reynold's Creek (INDU903) (Table 8). The number of detections expressed as percent of the total number of pesticides and PPCPs analyzed ranged from 0.5 to 9.4 percent.

Table 8. Number of detections and percent of total number of pesticides, pharmaceuticals, and personal care products analyzed for 19 sites at Indiana Dunes National Lakeshore in November, 2014.

Site ID	Site Location	Number of Detections	Percent of Total Analyzed
INDU503	East Branch Little Calumet River @Mineral Springs	20	9.4
INDU901	Carver Ditch	20	9.4
INDU539	East Branch Little Calumet River	17	8.0
INDU459	East Branch Little Calumet River @ Heron Rookery-Downstream	13	6.1
INDU082	Kintzele Ditch	12	5.7
INDU144	Dunes Creek @ Hawley Rd.	11	5.2
INDU900	Samuelson Ditch	11	5.2
INDU905	Sand Creek	10	4.7
INDU029	Peterson Ditch	9	4.2

Table 8 (continued). Number of detections and percent of total number of pesticides, pharmaceuticals, and personal care products analyzed for 19 sites at Indiana Dunes National Lakeshore in November, 2014.

Site ID	Site Location	Number of Detections	Percent of Total Analyzed
INDU164	Dunes Creek @ US12	9	4.2
INDU243	Brown Ditch	9	4.2
INDU904	Pope O'Connor Ditch	9	4.2
INDU475	East Branch Little Calumet River @ Heron Rookery-Upstream	8	3.8
INDU405	Derby Ditch @ Beverly Dr.	7	3.3
INDU328	Derby Ditch @ US12 and Oxford	6	2.8
INDU262	Derby Ditch @ US12 and N375E	5	2.4
INDU906	Coffee Creek @Calumet Rd.	5	2.4
INDU902	Coffee Creek @Morgan Ave.	4	1.9
INDU903	Reynold's Creek	1	0.5

Wastewater indicator samples were collected at five out of the 19 sites. Eight wastewater indicator analytes were detected, with the highest number (5) detected at site INDU082 (Table 9). No analytes were detected in samples collected at sites INDU029 and INDU405.

Table 9. Number of detections and percent of total number of wastewater indicators analyzed for five sites at Indiana Dunes National Lakeshore in November, 2014

Site ID	Site Location	Number of Detections	Percent of Total Analyzed
INDU082	Kintzele Ditch	5	9.4
INDU475	East Branch Little Calumet River @ Heron Rookery-Upstream	3	5.7
INDU900	Samuelson Ditch	2	3.8
INDU029	Peterson Ditch	0	0.0
INDU405	Derby Ditch @ Beverly Dr.	0	0.0

Quality Assurance/Quality Control

Pesticide and PPCP replicate samples were collected concurrently with environmental samples for at least 10 percent of all sites in a given round of sampling at a park in order to determine variability introduced from collection, processing, shipping, and laboratory handling. Eight analytes did not meet the benchmark of <30% RPD between the environmental and replicate samples (Table 10). In seven out of eight instances, both the environmental and replicate sample concentrations were only slightly above their respective reporting limits, where relatively small differences in analyte levels produced larger RPDs. Because of this and the fact that for the purposes of this report, analyte concentrations are reported qualitatively and not subjected to statistical analyses, analytes not meeting the <30% RPD were not excluded when tallying number of detections by park, site, year, or any other variable. Moreover, many analyte results, reported as less than the reporting limit or as an actual concentration, were flagged by the laboratory to be considered estimates because they did not meet all of the laboratory QA/QC measures. Data flagged by the laboratory were not excluded when

tallying number of detections by park, site, or year, but will be considered for exclusion if analyzed statistically or if compared against known environmental thresholds. Data flagged as an estimate by the laboratory for detected analytes are included in Appendix B.

Table 10. Analytes with greater than 30% relative percent difference (RPD) between environmental and replicate samples. Results and reporting limits in ng/L. * indicates result less than reporting limit but set to reporting limit to calculate RPD. ** indicates two replicates collected

Park	Site ID	Date	Analyte	Environmental	Replicate	Reporting Limit	RPD
MISS	UM831	4/2/2013	Sulfamethoxazole	17	25	10	34
MISS	UM839	11/12/2013	Lamotrigine	20	35	10	53
MISS	UM831	7/8/2014	Methylparaben	25*	87	25	111
MISS	UM883	8/12/2014	DEET	50	192/163**	10	117/106
INDU	INDU164	11/12/2014	DEET	40	15.5	10	89
INDU	INDU539	11/12/2014	Sotalol	11	13.0	10	48
INDU	INDU539	11/12/2014	Desmethylvenlafaxine	25	29.7	10	37
INDU	INDU539	11/12/2014	Caffeine	36	25*	25	62

Discussion

The purpose of this project was to screen select surface waters across three national parks in the Great Lakes Inventory and Monitoring Network for detectable concentrations of pesticides, pharmaceuticals, personal care products, and wastewater indicators. Project objectives include:

- Collection of high quality surface water samples at locations where previous sampling or land use activities indicate CECs may be present, or at locations where baseline data are unavailable,
- Reporting data collected, analytical results and the distribution of detections between sites, and
- Providing NPS with CEC concentration information at low detection limits.

In general, parks were chosen for monitoring because 1) they are situated in an urban and/or agricultural setting with both point and nonpoint sources of contaminants, where at least some CEC monitoring had already taken place across different environmental media (e.g., water, sediment, or fish), and where CECs were likely to be detected (INDU and MISS), and 2) where CEC baseline information is scarce even though levels are perceived to be low (APIS). Within-park monitoring sites were chosen based on 1) sites of high interest to park staff (e.g., Coldwater Spring at MISS), 2) co-location with previous or ongoing CEC monitoring efforts (most sites across the three parks), or 3) where baseline data are scarce (e.g., Lake Superior near Outer Island at APIS).

This data summary focuses on basic metrics such as number of contaminants detected and frequency of occurrence by park, year, analyte, and monitoring site. GLKN is partnering with the USGS Minnesota Water Science Center to compare these results to concentrations known to have chronic or acute impacts to aquatic organisms and with results from other similar studies relevant to the three parks. Therefore, the discussion to follow focuses on noteworthy findings, particular CECs of interest, and future monitoring efforts.

As was expected, the total number of pesticide and PPCP detections from river, stream, and ditch sites at MISS (44 distinct analytes from 20 samples across four sites) and INDU (42 distinct analytes from 19 samples across 19 sites) were much higher than those detected in the nearshore Lake Superior waters at APIS (five distinct analytes from 21 samples across seven sites). For analytes detected in at least 50 percent of all samples, pesticides, mainly in the form of herbicides or associated breakdown products, were the most common class of analyte found at MISS and INDU. This is not surprising since the rivers, streams, and ditches at these two parks drain -or are within- watersheds where agriculture and pesticide use is widespread. Interestingly, simazine—an herbicide used to control broadleaf plants and grasses—was detected in 53 percent of samples at INDU, but was not detected at MISS, perhaps indicating a localized application in or near INDU or a regional preference in type of herbicide used.

Gabapentin, a pharmaceutical used to treat epilepsy and neuropathic pain (used on both humans and pets), was widespread in surface waters across all three parks sampled; it was detected in 80, 29, and

58 percent of samples collected at MISS, APIS, and INDU, respectively. Wastewater treatment plants have been shown to be a major source of neuro-active pharmaceuticals such as gabapentin in surface waters of Minnesota (Writer et al. 2013). All of the river monitoring sites at MISS, and several of the stream and river locations at INDU are located downstream of one or more wastewater treatment plants. Wastewater treatment plant effluent is also discharged into Lake Superior, but it is noteworthy that even for such a large water body with high dilution potential, gabapentin was still detected in nearly 30 percent (6 out of 21) of samples collected at APIS.

Wastewater indicator samples were collected infrequently. Therefore, even qualitative comparisons are challenging. However, it is notable that flame retardants (tri(2-butoxyethyl) phosphate and tri(dichloroisopropyl) phosphate) were detected in greater than 50% of wastewater indicator samples collected at MISS and APIS. Similarly, at least one flame retardant was detected in all three samples collected at Coldwater Spring, and in a single sample at INDU (triphenyl phosphate). These detections are from surface water samples collected from a large river, Great Lake, natural spring, and ditch, implying that contamination from flame retardants is pervasive and widespread. The same flame retardants detected in this project have even been detected in finished drinking water (Stackelberg et al. 2004). Other flame retardants such as polybrominated diphenyl ethers (PDBEs) have become ubiquitous in U.S. surface waters (Betts 2009) and have been detected in bald eagle nestlings from active nesting territories that directly overlap with CEC monitoring sites for this project at MISS and APIS (Route et al. 2014b).

A definite pattern of increasing numbers of CEC detections from upstream to downstream is evident for the Mississippi River sites, most likely as a result of contaminant contributions from the Minnesota River, Metro Wastewater Plant, and other point and nonpoint sources within Minneapolis and St. Paul, Minnesota, which are cumulative traveling downriver. For three of the four downstream sites, a seasonal pattern was also apparent with a higher number of detections in early spring and late summer or fall and lower detections in early summer. Possible explanations include the timing of herbicide applications, the influence of river flow, precipitation patterns, and water temperature. For example, water temperatures may directly affect chemical breakdown of analytes or indirectly decrease the metabolism or number of organisms that metabolize them.

The GLKN is partnering with toxicologists from the USGS Minnesota Water Science Center to compare concentrations of detected pesticides, PPCPs, and wastewater indicators against known environmental thresholds, and where possible, with CEC data from other studies. Noteworthy results, if found, will be published in a peer-reviewed journal and disseminated to park staff and partners. In 2015 GLKN will focus surface water CEC monitoring activities on up to nine inland lakes at Isle Royale National Park (where the number of CECs is hypothesized to be very low), as these lakes are isolated and generally are only exposed to CECs through atmospheric deposition.

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Appendix A. List of all analytes and associated reporting limits.

Table A-1. Target analytes and method reporting limits (MRL) for pesticides (EPA Region 8 SOP Analysis Method ORGM-550 and Modified Method EPA 8321A for Pesticides in Water by Light Chromatography- Mass Spectrometry (LCMS)). Lists are separated into positive (Pos) and negative (Neg) ion modes depending on the polarity of the pesticide.

Analysis: LCMS_Pest_2014_Pos	
Analyte	MRL (ng/L)
3-Hydroxycarbofuran	10.0
Acetochlor	20.0
Alachlor	20.0
Aldicarb	10.0
Aldicarb sulfone	20.0
Aldicarb sulfoxide	10.0
Atraton	10.0
Atrazine	10.0
Atrazine de-ethyl	20.0
Atrazine de-isopropyl	20.0
Azoxystrobin	20.0
Bromacil	50.0
Carbaryl	10.0
Carbofuran	10.0
Chlorimuron ethyl	20.0
Chlorsulfuron	20.0
Clothianidin	20.0
Cyanazine	10.0
Diazinon	20.0
Dimethachlor	10.0
Dimethenamid	10.0
Disulfoton sulfone	20.0
Diuron	20.0
Diuron Metabolite	20.0
EPTC	20.0
Ethoprop	10.0
Foramsulfuron	20.0
Hexazinone	10.0
Imazaquin	20.0
Imazethapyr	20.0
Imidacloprid	20.0
Linuron	20.0

Table A-1 (continued). Target analytes and method reporting limits (MRL) for pesticides (EPA Region 8 SOP Analysis Method ORGM-550 and Modified Method EPA 8321A for Pesticides in Water by Light Chromatography- Mass Spectrometry (LCMS)). Lists are separated into positive (Pos) and negative (Neg) ion modes depending on the polarity of the pesticide.

Analysis: LCMS_Pest_2014_Pos	
Analyte	MRL (ng/L)
Malathion	10.0
Metalaxyl	10.0
Methiocarb	20.0
Methomyl	20.0
Metolachlor	10.0
Metribuzin	20.0
Monuron	10.0
Neburon	50.0
Oxamyl	10.0
Prometon	20.0
Propachlor	10.0
Propazine	20.0
Propiconazole	50.0
Propoxur	10.0
Simazine	20.0
Sulfometuron methyl	20.0
Tebuconazole	50.0
Tebuthiuron	10.0
Terbuthylazine	10.0
Thiamethoxam	20.0
Analysis: LCMS_Pest_2014_Neg	
2,4,5-T	10.0
2,4,5-TP	10.0
2,4-D	10.0
Acetochlor OA	20.0
Alachlor OA	50.0
Bentazon	10.0
Bromoxynil	10.0
Butachlor ESA	10.0
Dacthal monoacid	20.0
Dichloroprop	10.0
Dimethenamid ESA	20.0
Fipronil	20.0
Fipronil sulfide	10.0
Fipronil sulfone	10.0

Table A-1 (continued). Target analytes and method reporting limits (MRL) for pesticides (EPA Region 8 SOP Analysis Method ORGM-550 and Modified Method EPA 8321A for Pesticides in Water by Light Chromatography- Mass Spectrometry (LCMS)). Lists are separated into positive (Pos) and negative (Neg) ion modes depending on the polarity of the pesticide.

Analysis: LCMS_Pest_2014_Neg	
Analyte	MRL (ng/L)
MCP	20.0
Metolachlor ESA	20.0
Oryzalin	20.0
Propachlor ESA	20.0
Propachlor OA	10.0
Triclopyr	20.0

Table A-2. Target analytes and method reporting limits (MRL) for pharmaceuticals and personal care products (PPCPs) (EPA Method 1694 for PPCPs in Water by LCMS and EPA Region 8 SOP Analysis Methods LCMS PPCPs ESI+ and LCMS PPCPs ESI-). Lists are separated into negative (-) and positive (+) ion modes depending on the polarity of the compound.

Analysis: PPCPs_ESI-_2014	
Analyte	MRL (ng/L)
(+/-)11-nor-9-carboxy-delta-THC	10.0
Bezafibrate	10.0
Celecoxib	10.0
Chloramphenicol	10.0
Diclofenac	10.0
Diclofenac 4-hydroxy	25.0
Fenbufen	25.0
Furosemide	10.0
Gemfibrozil	10.0
Hydrochlorothiazide	10.0
Ibuprofen	25.0
Methylparaben	25.0
Modafinil acid	25.0
Naproxen	25.0
n-Butylparaben	25.0
Phenobarbital	25.0
Phenytoin	10.0
Pravastatin	25.0
Sulfamethoxazole	10.0
Triclocarban	10.0
Triclosan	25.0
Warfarin	10.0
Analysis: PPCPs_ESI+_2014	
10,11-dihydro-10-hydroxycarbamazepine	10.0
6-Acetylmorphine	10.0
Acebutolol	10.0
Acetaminophen	10.0
Albuterol	10.0
Amitriptyline	10.0
Amitriptyline (+/-)-E-10-hydroxylated	10.0
Amphetamine	10.0
Aripiprazole	10.0
Atenolol	10.0
Atorvastatin	10.0

Table A-2 (continued). Target analytes and method reporting limits (MRL) for pharmaceuticals and personal care products (PPCPs) (EPA Method 1694 for PPCPs in Water by LCMS and EPA Region 8 SOP Analysis Methods LCMS PPCPs ESI+ and LCMS PPCPs ESI-). Lists are separated into negative (-) and positive (+) ion modes depending on the polarity of the compound.

Analysis: PPCPs_ESI+ _2014	
Analyte	MRL (ng/L)
Atrazine	10.0
Benzoylecgonine	10.0
Buprenorphine	10.0
Bupropion	10.0
Caffeine	25.0
Carbamazepine	10.0
Carbamazepine 10,11 epoxide	10.0
Carisoprodol	10.0
Chlorpheniramine	10.0
Clenbuterol	10.0
Clopidogrel carboxylic acid	10.0
Cocaethylene	10.0
Cocaine	10.0
Codeine	10.0
Cotinine	10.0
DEET	10.0
Dehydroaripiprazole	10.0
Desmethylcitalopram	10.0
Desmethylvenlafaxine	10.0
Dextromethorphan	10.0
Diltiazem	10.0
Diphenhydramine	10.0
Disopyramide	10.0
Donepezil	10.0
Duloxetine	25.0
Ecgonine Methyl Ester	10.0
EDDP	10.0
Erythromycin	10.0
Erythromycin-anhydro	10.0
Escitalopram	10.0
Famotidine	10.0
Fentanyl	10.0
Fluoxetine	10.0
Fluticasone propionate	10.0

Table A-2 (continued). Target analytes and method reporting limits (MRL) for pharmaceuticals and personal care products (PPCPs) (EPA Method 1694 for PPCPs in Water by LCMS and EPA Region 8 SOP Analysis Methods LCMS PPCPs ESI+ and LCMS PPCPs ESI-). Lists are separated into negative (-) and positive (+) ion modes depending on the polarity of the compound.

Analysis: PPCPs_ESI+ _2014	
Analyte	MRL (ng/L)
Gabapentin	10.0
Glyburide	10.0
Hydrocodone	10.0
Hydromorphone	10.0
Hydroxybupropion	10.0
Ketoprofen	10.0
Lamotrigine	10.0
Levorphanol	10.0
Lidocaine	10.0
Loratadine	10.0
Lorazepam	25.0
MDA	10.0
MDEA	10.0
MDMA	10.0
Mefenamic acid	10.0
Meperidine	10.0
Meprobamate	10.0
Metformin	10.0
Methadone	10.0
Methamphetamine	10.0
Methotrexate	10.0
Methylphenidate	10.0
Metoprolol	10.0
Mevastatin	10.0
m-Hydroxybenzoylecgonine	10.0
Modafinil	10.0
Monoethylglycinexylidide	10.0
Montelukast	10.0
Morphine	10.0
Nifedipine	10.0
Nifedipine oxidized	10.0
Norfentanyl	10.0
Norfluoxetine	10.0
Normeperidine	10.0

Table A-2 (continued). Target analytes and method reporting limits (MRL) for pharmaceuticals and personal care products (PPCPs) (EPA Method 1694 for PPCPs in Water by LCMS and EPA Region 8 SOP Analysis Methods LCMS PPCPs ESI+ and LCMS PPCPs ESI-). Lists are separated into negative (-) and positive (+) ion modes depending on the polarity of the compound.

Analysis: PPCPs_ESI+ _2014	
Analyte	MRL (ng/L)
Norquetiapine	10.0
Norsertaline	10.0
Norverapamil	10.0
Omeprazole	10.0
Oxazepam	10.0
Oxcarbazepine	10.0
Oxycodone	10.0
Oxymorphone	10.0
Oxymorphone glucuronide	10.0
Paroxetine	10.0
Phenmetrazine	10.0
Phentermine	25.0
Phenylpropanolamine	10.0
Pioglitazone	10.0
Pregabalin	10.0
Primidone	25.0
Propranolol	10.0
Pseudoephedrine	10.0
Quetiapine	10.0
Ritalinic acid	10.0
Sertraline	10.0
Sildenafil	10.0
Simvastatin	10.0
Sotalol	10.0
Sulfamethazine	10.0
Sumatriptan	10.0
Tadalafil	25.0
Temazepam	10.0
Thiabendazole	10.0
Tramadol	10.0
Trazodone	10.0
Triamterene	10.0
Trimethoprim	10.0
Tylosin	25.0

Table A-2 (continued). Target analytes and method reporting limits (MRL) for pharmaceuticals and personal care products (PPCPs) (EPA Method 1694 for PPCPs in Water by LCMS and EPA Region 8 SOP Analysis Methods LCMS PPCPs ESI+ and LCMS PPCPs ESI-). Lists are separated into negative (-) and positive (+) ion modes depending on the polarity of the compound.

Analysis: PPCPs_ESI+_2014	
Analyte	MRL (ng/L)
Valsartan	10.0
Venlafaxine	10.0
Verapamil	10.0
Zolpidem	10.0
Zolpidem phenyl-4-carboxylic acid	10.0

Table A-3. Target analytes and method reporting limits (MRL) for wastewater indicators (EPA Region 8 Laboratory SOP Analysis Method ORGM-006 for Wastewater Indicators in Water by GCMS).

Analyte	MRL (ng/L)
1,4-Dichlorobenzene	50.0
17beta-Estradiol	100
1-Methylnaphthalene	50.0
2,6-Dimethylnaphthalene	50.0
2-Methylnaphthalene	50.0
3-Methyl Indole	50.0
4-Cumylphenol	50.0
4-n-Octylphenol	50.0
4-tert-Octylphenol	50.0
Acetophenone	50.0
Benzophenone	50.0
Bisphenol A	50.0
Bromacil	50.0
Butylated Hydroxyanisole	100
Caffeine	50.0
Camphor	50.0
Carbaryl	50.0
Carbazole	50.0
Celestolide	50.0
Chlorpyrifos	50.0
Dacthal	50.0
Diazinon	50.0
Dichlorvos	50.0
Equilenin	100
Estrone	100
Ethinylestradiol	100
Fluoranthene	50.0
Galaxolide	50.0
Indole	50.0
Isoborneol	50.0
Isophorone	50.0
Isoquinoline	50.0
Menthol	50.0
Methyl Salicylate	50.0
Metolachlor	50.0
Musk ketone	50.0

Table A-3 (continued). Target analytes and method reporting limits (MRL) for wastewater indicators (EPA Region 8 Laboratory SOP Analysis Method ORGM-006 for Wastewater Indicators in Water by GCMS).

Analyte	MRL (ng/L)
Naphthalene	50.0
Nonylphenol	50.0
p-Cresol	50.0
Pentachlorophenol	100
Phantolide	50.0
Phenanthrene	50.0
Phenol	50.0
Prometon	50.0
Pyrene	50.0
Tonalide	50.0
Tri(2-butoxyethyl) Phosphate	50.0
Tri(2-chloroethyl) Phosphate	50.0
Tri(dichloroisopropyl) Phosphate	50.0
Tributyl Phosphate	50.0
Triclosan	50.0
Triethyl Citrate	50.0
Triphenyl phosphate	50.0

Appendix B. Concentrations of detected analytes.

Table B-1. Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
Coldwater Spring	11/12/2013	Caffeine	58-08-2	18.9	10.0
Coldwater Spring	8/11/2014	DEET	134-62-3	51.8 [1]	10.0
Coldwater Spring	4/2/2013	Metolachlor ESA	947601-85-6	50.2	20.0
Coldwater Spring	6/10/2013	Metolachlor ESA	947601-85-6	38.9	20.0
Coldwater Spring	11/12/2013	Metolachlor ESA	947601-85-6	38.0	20.0
Coldwater Spring	7/7/2014	Metolachlor ESA	947601-85-6	29.9	20.0
Coldwater Spring	8/11/2014	Metolachlor ESA	947601-85-6	29.7	20.0
Coldwater Spring	6/10/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	452 [1]	50.0
Coldwater Spring	11/12/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	270 [1]	50.0
Coldwater Spring	4/2/2013	Tri(dichloroisopropyl) Phosphate	13674-87-8	98.5 [1]	50.0
UM883B10	4/1/2013	2,4-D	94-75-7	31.2	10.0
UM883B10	6/11/2013	2,4-D	94-75-7	25.0	10.0
UM883B10	11/12/2013	2,4-D	94-75-7	22.9	10.0
UM883B10	7/7/2014	2,4-D	94-75-7	64.0	10.0
UM883B10	8/12/2014	2,4-D	94-75-7	24.5	10.0
UM883B10	7/7/2014	Acetochlor	34256-82-1	24.1	20.0
UM883B10	6/11/2013	Acetochlor OA	194992-44-4	43.5	20.0
UM883B10	11/12/2013	Acetochlor OA	194992-44-4	21.1	20.0
UM883B10	7/7/2014	Acetochlor OA	194992-44-4	92.4	20.0
UM883B10	8/12/2014	Acetochlor OA	194992-44-4	36.8	20.0
UM883B10	4/1/2013	Atenolol	29122-68-7	10.7	10.0
UM883B10	4/1/2013	Atrazine	1912-24-9	16.0	10.0
UM883B10	4/1/2013	Atrazine	1912-24-9	14.4	10.0
UM883B10	6/11/2013	Atrazine	1912-24-9	22.3	10.0
UM883B10	6/11/2013	Atrazine	1912-24-9	19.7	10.0
UM883B10	11/12/2013	Atrazine	1912-24-9	12.2	10.0
UM883B10	11/12/2013	Atrazine	1912-24-9	13.1 [1]	10.0
UM883B10	7/7/2014	Atrazine	1912-24-9	31.9	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM883B10	7/7/2014	Atrazine	1912-24-9	40.4	10.0
UM883B10	8/12/2014	Atrazine	1912-24-9	18.0	10.0
UM883B10	8/12/2014	Atrazine	1912-24-9	16.7 [1]	10.0
UM883B10	6/11/2013	Bisphenol A	80-05-7	150	50.0
UM883B10	4/1/2013	Caffeine	58-08-2	32.8	10.0
UM883B10	11/12/2013	Caffeine	58-08-2	22.5	10.0
UM883B10	11/12/2013	DEET	134-62-3	10.4	10.0
UM883B10	7/7/2014	DEET	134-62-3	166 [1]	10.0
UM883B10	8/12/2014	DEET	134-62-3	50.0 [1]	10.0
UM883B10	11/12/2013	Desmethylvenlafaxine	93413-62-8	11.1	10.0
UM883B10	4/1/2013	Gabapentin	60142-96-3	245	10.0
UM883B10	6/11/2013	Gabapentin	60142-96-3	112	10.0
UM883B10	11/12/2013	Gabapentin	60142-96-3	110	10.0
UM883B10	7/7/2014	Gabapentin	60142-96-3	104	10.0
UM883B10	8/12/2014	Gabapentin	60142-96-3	61.1 [1]	10.0
UM883B10	4/1/2013	Hydrochlorothiazide	58-93-5	12.7 [1]	10.0
UM883B10	7/7/2014	Imazethapyr	81335-77-5	31.9 [1]	20.0
UM883B10	8/12/2014	Imazethapyr	81335-77-5	24.7 [1]	20.0
UM883B10	11/12/2013	Lamotrigine	84057-84-1	16.2	10.0
UM883B10	4/1/2013	Metformin	657-24-9	69.3	10.0
UM883B10	6/11/2013	Metformin	657-24-9	24.9	10.0
UM883B10	11/12/2013	Metformin	657-24-9	15.8	10.0
UM883B10	7/7/2014	Metformin	657-24-9	20.0	10.0
UM883B10	8/12/2014	Metformin	657-24-9	13.6 [1]	10.0
UM883B10	7/7/2014	Metolachlor	51218-45-2	22.8	10.0
UM883B10	4/1/2013	Metolachlor ESA	947601-85-6	91.9	20.0
UM883B10	6/11/2013	Metolachlor ESA	947601-85-6	109	20.0
UM883B10	11/12/2013	Metolachlor ESA	947601-85-6	185	20.0
UM883B10	7/7/2014	Metolachlor ESA	947601-85-6	159	20.0
UM883B10	8/12/2014	Metolachlor ESA	947601-85-6	212	20.0
UM883B10	4/1/2013	Naproxen	22204-53-1	19.9 [1]	10.0
UM883B10	11/12/2013	Sulfamethoxazole	723-46-6	10.8	10.0
UM883B10	6/11/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	562 [1]	50.0
UM883B10	11/12/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	205 [1]	50.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM883B10	4/1/2013	Tri(dichloroisopropyl) Phosphate	13674-87-8	127 [1]	50.0
UM839B10	4/2/2013	2,4-D	94-75-7	172	10.0
UM839B10	6/10/2013	2,4-D	94-75-7	39.4	10.0
UM839B10	11/12/2013	2,4-D	94-75-7	25.9	10.0
UM839B10	7/8/2014	2,4-D	94-75-7	101	10.0
UM839B10	8/11/2014	2,4-D	94-75-7	164	10.0
UM839B10	4/2/2013	Acetaminophen	103-90-2	13.4	10.0
UM839B10	4/2/2013	Acetochlor	34256-82-1	195	20.0
UM839B10	6/10/2013	Acetochlor	34256-82-1	77.0	20.0
UM839B10	7/8/2014	Acetochlor	34256-82-1	125	20.0
UM839B10	4/2/2013	Acetochlor OA	194992-44-4	322	20.0
UM839B10	6/10/2013	Acetochlor OA	194992-44-4	270	20.0
UM839B10	11/12/2013	Acetochlor OA	194992-44-4	42.8	20.0
UM839B10	7/8/2014	Acetochlor OA	194992-44-4	896	20.0
UM839B10	8/11/2014	Acetochlor OA	194992-44-4	143	20.0
UM839B10	4/2/2013	Atenolol	29122-68-7	10.8	10.0
UM839B10	4/2/2013	Atrazine	1912-24-9	33.9	10.0
UM839B10	4/2/2013	Atrazine	1912-24-9	26.8	10.0
UM839B10	6/10/2013	Atrazine	1912-24-9	38.4	10.0
UM839B10	6/10/2013	Atrazine	1912-24-9	35.2 [1]	10.0
UM839B10	11/12/2013	Atrazine	1912-24-9	19.8	10.0
UM839B10	11/12/2013	Atrazine	1912-24-9	22.5 [1]	10.0
UM839B10	7/8/2014	Atrazine	1912-24-9	191	10.0
UM839B10	7/8/2014	Atrazine	1912-24-9	302	10.0
UM839B10	8/11/2014	Atrazine	1912-24-9	53.8	10.0
UM839B10	8/11/2014	Atrazine	1912-24-9	43.7 [1]	10.0
UM839B10	6/10/2013	Atrazine de-ethyl	6190-65-4	24.8	20.0
UM839B10	7/8/2014	Atrazine de-ethyl	6190-65-4	56.7	20.0
UM839B10	7/8/2014	Atrazine de-isopropyl	1007-28-9	33.6	20.0
UM839B10	7/8/2014	Bentazon	25057-89-0	12.6	10.0
UM839B10	8/11/2014	Bentazon	25057-89-0	11.0	10.0
UM839B10	4/2/2013	Caffeine	58-08-2	16.6	10.0
UM839B10	11/12/2013	Caffeine	58-08-2	18.9	10.0
UM839B10	8/11/2014	Caffeine	58-08-2	41.8 [1]	25.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM839B10	7/8/2014	Clothianidin	210880-92-5	37.5	20.0
UM839B10	4/2/2013	Cotinine	486-56-6	11.1	10.0
UM839B10	11/12/2013	DEET	134-62-3	11.0	10.0
UM839B10	7/8/2014	DEET	134-62-3	85.7 [1]	10.0
UM839B10	8/11/2014	DEET	134-62-3	168 [1]	10.0
UM839B10	11/12/2013	Desmethylvenlafaxine	93413-62-8	20.3	10.0
UM839B10	8/11/2014	Desmethylvenlafaxine	93413-62-8	12.6 [1]	10.0
UM839B10	4/2/2013	Dimethenamid	87674-68-8	65.1	10.0
UM839B10	6/10/2013	Dimethenamid	87674-68-8	19.6	10.0
UM839B10	7/8/2014	Dimethenamid	87674-68-8	108	10.0
UM839B10	4/2/2013	Dimethenamid ESA	205939-58-8	24.6	20.0
UM839B10	6/10/2013	Dimethenamid ESA	205939-58-8	21.2	20.0
UM839B10	7/8/2014	Dimethenamid ESA	205939-58-8	104	20.0
UM839B10	4/2/2013	Gabapentin	60142-96-3	350	10.0
UM839B10	6/10/2013	Gabapentin	60142-96-3	152 [1]	10.0
UM839B10	11/12/2013	Gabapentin	60142-96-3	122	10.0
UM839B10	7/8/2014	Gabapentin	60142-96-3	45.0	10.0
UM839B10	8/11/2014	Gabapentin	60142-96-3	49.0 [1]	10.0
UM839B10	4/2/2013	Gemfibrozil	25812-30-0	11.3 [1]	10.0
UM839B10	4/2/2013	Hydrochlorothiazide	58-93-5	10.8 [1]	10.0
UM839B10	11/12/2013	Hydrochlorothiazide	58-93-5	16.5	10.0
UM839B10	8/11/2014	Hydrochlorothiazide	58-93-5	11.4	10.0
UM839B10	4/2/2013	Hydroxybupropion	92264-81-8	11.6	10.0
UM839B10	11/12/2013	Hydroxybupropion	92264-81-8	11.0	10.0
UM839B10	4/2/2013	Imazethapyr	81335-77-5	37.8	10.0
UM839B10	6/10/2013	Imazethapyr	81335-77-5	26.5	10.0
UM839B10	7/8/2014	Imazethapyr	81335-77-5	189 [1]	20.0
UM839B10	8/11/2014	Imazethapyr	81335-77-5	29.1 [1]	20.0
UM839B10	4/2/2013	Lamotrigine	84057-84-1	20.6	10.0
UM839B10	11/12/2013	Lamotrigine	84057-84-1	20.2	10.0
UM839B10	8/11/2014	Lamotrigine	84057-84-1	27.4 [1]	10.0
UM839B10	4/2/2013	Metformin	657-24-9	49.2	10.0
UM839B10	6/10/2013	Metformin	657-24-9	41.9 [1]	10.0
UM839B10	11/12/2013	Metformin	657-24-9	43.6	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM839B10	7/8/2014	Metformin	657-24-9	17.5	10.0
UM839B10	8/11/2014	Metformin	657-24-9	26.6 [1]	10.0
UM839B10	4/2/2013	Metolachlor	51218-45-2	702	10.0
UM839B10	6/10/2013	Metolachlor	51218-45-2	16.6	10.0
UM839B10	7/8/2014	Metolachlor	51218-45-2	328	10.0
UM839B10	8/11/2014	Metolachlor	51218-45-2	19.8	10.0
UM839B10	4/2/2013	Metolachlor ESA	947601-85-6	369	20.0
UM839B10	6/10/2013	Metolachlor ESA	947601-85-6	273	20.0
UM839B10	11/12/2013	Metolachlor ESA	947601-85-6	168	20.0
UM839B10	7/8/2014	Metolachlor ESA	947601-85-6	555	20.0
UM839B10	8/11/2014	Metolachlor ESA	947601-85-6	211	20.0
UM839B10	6/10/2013	Ranitidine	66357-35-5	20.4 [1]	10.0
UM839B10	4/2/2013	Sulfamethoxazole	723-46-6	10.7	10.0
UM839B10	11/12/2013	Sulfamethoxazole	723-46-6	20.4	10.0
UM839B10	8/11/2014	Sulfamethoxazole	723-46-6	13.0	10.0
UM839B10	4/2/2013	Theobromine	83-67-0	13.7	10.0
UM839B10	4/2/2013	Triclopyr	55335-06-3	49.4	20.0
UM831B10	8/11/2014	10,11-dihydro-10-hydroxycarbamazepine	29331-92-8	24.2 [1]	10.0
UM831B10	4/2/2013	2,4-D	94-75-7	155	10.0
UM831B10	6/10/2013	2,4-D	94-75-7	46.3	10.0
UM831B10	11/13/2013	2,4-D	94-75-7	32.1	10.0
UM831B10	7/8/2014	2,4-D	94-75-7	98.9	10.0
UM831B10	8/11/2014	2,4-D	94-75-7	119	10.0
UM831B10	4/2/2013	Acetaminophen	103-90-2	10.5	10.0
UM831B10	4/2/2013	Acetochlor	34256-82-1	179	20.0
UM831B10	6/10/2013	Acetochlor	34256-82-1	71.9	20.0
UM831B10	7/8/2014	Acetochlor	34256-82-1	97.9	20.0
UM831B10	4/2/2013	Acetochlor OA	194992-44-4	277	20.0
UM831B10	6/10/2013	Acetochlor OA	194992-44-4	252	20.0
UM831B10	11/13/2013	Acetochlor OA	194992-44-4	43.0	20.0
UM831B10	7/8/2014	Acetochlor OA	194992-44-4	784	20.0
UM831B10	8/11/2014	Acetochlor OA	194992-44-4	133	20.0
UM831B10	4/2/2013	Atenolol	29122-68-7	22.2	10.0
UM831B10	11/13/2013	Atenolol	29122-68-7	26.0	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM831B10	8/11/2014	Atenolol	29122-68-7	10.1 [1]	10.0
UM831B10	4/2/2013	Atrazine	1912-24-9	32.1	10.0
UM831B10	4/2/2013	Atrazine	1912-24-9	22.1	10.0
UM831B10	6/10/2013	Atrazine	1912-24-9	38.5	10.0
UM831B10	6/10/2013	Atrazine	1912-24-9	25.1 [1]	10.0
UM831B10	11/13/2013	Atrazine	1912-24-9	20.3	10.0
UM831B10	11/13/2013	Atrazine	1912-24-9	23.9 [1]	10.0
UM831B10	7/8/2014	Atrazine	1912-24-9	225	10.0
UM831B10	7/8/2014	Atrazine	1912-24-9	241	10.0
UM831B10	8/11/2014	Atrazine	1912-24-9	61.2	10.0
UM831B10	8/11/2014	Atrazine	1912-24-9	53.8 [1]	10.0
UM831B10	6/10/2013	Atrazine de-ethyl	6190-65-4	24.8	20.0
UM831B10	7/8/2014	Atrazine de-ethyl	6190-65-4	53.6	20.0
UM831B10	7/8/2014	Atrazine de-isopropyl	1007-28-9	26.2	20.0
UM831B10	7/8/2014	Bentazon	25057-89-0	10.2	10.0
UM831B10	8/11/2014	Bentazon	25057-89-0	10.1	10.0
UM831B10	6/10/2013	Bisphenol A	80-05-7	156	50.0
UM831B10	11/13/2013	Bisphenol A	80-05-7	232 [1]	50.0
UM831B10	4/2/2013	Caffeine	58-08-2	18.4	10.0
UM831B10	11/13/2013	Caffeine	58-08-2	29.2	10.0
UM831B10	8/11/2014	Caffeine	58-08-2	43.3 [1]	25.0
UM831B10	11/13/2013	Carbamazepine	298-46-4	16.0	10.0
UM831B10	8/11/2014	Carbamazepine	298-46-4	11.2 [1]	10.0
UM831B10	7/8/2014	Clothianidin	210880-92-5	25.7	20.0
UM831B10	11/13/2013	DEET	134-62-3	12.4	10.0
UM831B10	7/8/2014	DEET	134-62-3	251 [1]	10.0
UM831B10	8/11/2014	DEET	134-62-3	102 [1]	10.0
UM831B10	4/2/2013	Desmethylvenlafaxine	93413-62-8	15.3	10.0
UM831B10	6/10/2013	Desmethylvenlafaxine	93413-62-8	19.0 [1]	10.0
UM831B10	11/13/2013	Desmethylvenlafaxine	93413-62-8	72.1	10.0
UM831B10	7/8/2014	Desmethylvenlafaxine	93413-62-8	21.3	10.0
UM831B10	8/11/2014	Desmethylvenlafaxine	93413-62-8	37.7 [1]	10.0
UM831B10	4/2/2013	Dimethenamid	87674-68-8	47.8	10.0
UM831B10	6/10/2013	Dimethenamid	87674-68-8	19.1	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM831B10	7/8/2014	Dimethenamid	87674-68-8	77.9	10.0
UM831B10	6/10/2013	Dimethenamid ESA	205939-58-8	24.5	20.0
UM831B10	7/8/2014	Dimethenamid ESA	205939-58-8	90.6	20.0
UM831B10	4/2/2013	Furosemide	54-31-9	14.5 [1]	10.0
UM831B10	11/13/2013	Furosemide	54-31-9	51.8	10.0
UM831B10	4/2/2013	Gabapentin	60142-96-3	705	10.0
UM831B10	6/10/2013	Gabapentin	60142-96-3	322 [1]	10.0
UM831B10	11/13/2013	Gabapentin	60142-96-3	343	10.0
UM831B10	7/8/2014	Gabapentin	60142-96-3	88.2	10.0
UM831B10	8/11/2014	Gabapentin	60142-96-3	228 [1]	10.0
UM831B10	4/2/2013	Galaxolide	1222-05-5	77.4	50.0
UM831B10	11/13/2013	Galaxolide	1222-05-5	115	50.0
UM831B10	4/2/2013	Gemfibrozil	25812-30-0	23.3 [1]	10.0
UM831B10	11/13/2013	Gemfibrozil	25812-30-0	24.0	10.0
UM831B10	8/11/2014	Gemfibrozil	25812-30-0	13.6	10.0
UM831B10	4/2/2013	Hydrochlorothiazide	58-93-5	51.8 [1]	10.0
UM831B10	11/13/2013	Hydrochlorothiazide	58-93-5	67.8	10.0
UM831B10	7/8/2014	Hydrochlorothiazide	58-93-5	16.6	10.0
UM831B10	8/11/2014	Hydrochlorothiazide	58-93-5	26.1	10.0
UM831B10	4/2/2013	Hydroxybupropion	92264-81-8	13.6	10.0
UM831B10	11/13/2013	Hydroxybupropion	92264-81-8	35.5	10.0
UM831B10	8/11/2014	Hydroxybupropion	92264-81-8	11.6 [1]	10.0
UM831B10	4/2/2013	Imazethapyr	81335-77-5	32.4	10.0
UM831B10	6/10/2013	Imazethapyr	81335-77-5	24.9	10.0
UM831B10	7/8/2014	Imazethapyr	81335-77-5	158 [1]	20.0
UM831B10	8/11/2014	Imazethapyr	81335-77-5	36.9 [1]	20.0
UM831B10	4/2/2013	Lamotrigine	84057-84-1	31.7	10.0
UM831B10	6/10/2013	Lamotrigine	84057-84-1	10.8 [1]	10.0
UM831B10	11/13/2013	Lamotrigine	84057-84-1	81.0	10.0
UM831B10	7/8/2014	Lamotrigine	84057-84-1	18.9	10.0
UM831B10	8/11/2014	Lamotrigine	84057-84-1	43.9 [1]	10.0
UM831B10	4/2/2013	Levorphanol	77-07-6	10.3	10.0
UM831B10	11/13/2013	Levorphanol	77-07-6	28.4	10.0
UM831B10	11/13/2013	Lidocaine	137-58-6	14.5	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM831B10	4/2/2013	Metformin	657-24-9	84.8	10.0
UM831B10	6/10/2013	Metformin	657-24-9	57.3 [1]	10.0
UM831B10	11/13/2013	Metformin	657-24-9	150	10.0
UM831B10	7/8/2014	Metformin	657-24-9	56.8	10.0
UM831B10	8/11/2014	Metformin	657-24-9	68.6 [1]	10.0
UM831B10	4/2/2013	Metolachlor	51218-45-2	813	50.0
UM831B10	4/2/2013	Metolachlor	51218-45-2	633	10.0
UM831B10	6/10/2013	Metolachlor	51218-45-2	13.4	10.0
UM831B10	7/8/2014	Metolachlor	51218-45-2	236	10.0
UM831B10	8/11/2014	Metolachlor	51218-45-2	23.3	10.0
UM831B10	4/2/2013	Metolachlor ESA	947601-85-6	316	20.0
UM831B10	6/10/2013	Metolachlor ESA	947601-85-6	254	20.0
UM831B10	11/13/2013	Metolachlor ESA	947601-85-6	181	20.0
UM831B10	7/8/2014	Metolachlor ESA	947601-85-6	446	20.0
UM831B10	8/11/2014	Metolachlor ESA	947601-85-6	212	20.0
UM831B10	11/13/2013	Metoprolol	37350-58-6	24.8	10.0
UM831B10	8/11/2014	Metoprolol	37350-58-6	13.1 [1]	10.0
UM831B10	4/2/2013	Naproxen	22204-53-1	23.6 [1]	10.0
UM831B10	11/13/2013	Naproxen	22204-53-1	13.2	10.0
UM831B10	8/11/2014	Propachlor ESA	947601-88-9	459	20.0
UM831B10	6/10/2013	Ranitidine	66357-35-5	28.6 [1]	10.0
UM831B10	11/13/2013	Ranitidine	66357-35-5	33.1	10.0
UM831B10	11/13/2013	Sotalol	959-24-0	11.2	10.0
UM831B10	8/11/2014	Sotalol	959-24-0	11.0 [1]	10.0
UM831B10	4/2/2013	Sulfamethoxazole	723-46-6	17.4	10.0
UM831B10	11/13/2013	Sulfamethoxazole	723-46-6	74.5	10.0
UM831B10	7/8/2014	Sulfamethoxazole	723-46-6	12.8	10.0
UM831B10	8/11/2014	Sulfamethoxazole	723-46-6	45.4	10.0
UM831B10	4/2/2013	Theobromine	83-67-0	12.1	10.0
UM831B10	11/13/2013	Tramadol	27203-92-5	26.4	10.0
UM831B10	8/11/2014	Tramadol	27203-92-5	18.2 [1]	10.0
UM831B10	6/10/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	733 [1]	50.0
UM831B10	11/13/2013	Tri(2-butoxyethyl) Phosphate	78-51-3	350 [1]	50.0
UM831B10	4/2/2013	Tri(dichloroisopropyl) Phosphate	13674-87-8	105 [1]	50.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM831B10	11/13/2013	Tri(dichloroisopropyl) Phosphate	13674-87-8	79.3 [1]	50.0
UM831B10	4/2/2013	Tributyl Phosphate	126-73-8	50.1	50.0
UM831B10	11/13/2013	Tributyl Phosphate	126-73-8	68.6 [1]	50.0
UM831B10	4/2/2013	Triclopyr	55335-06-3	53.9	20.0
UM831B10	11/13/2013	Triethyl Citrate = 1	77-93-0	146 [1]	50.0
UM831B10	11/13/2013	Trimethoprim	738-70-5	23.7	10.0
UM831B10	8/11/2014	Trimethoprim	738-70-5	13.8 [1]	10.0
UM831B10	11/13/2013	Venlafaxine	93413-44-6	27.6	10.0
UM831B10	8/11/2014	Venlafaxine	93413-44-6	11.5 [1]	10.0
UM815B10	8/11/2014	10,11-dihydro-10-hydroxycarbamazepine	29331-92-8	22.2 [1]	10.0
UM815B10	4/1/2013	2,4-D	94-75-7	160	10.0
UM815B10	6/10/2013	2,4-D	94-75-7	61.5	10.0
UM815B10	11/13/2013	2,4-D	94-75-7	34.1	10.0
UM815B10	7/7/2014	2,4-D	94-75-7	56.5	10.0
UM815B10	8/11/2014	2,4-D	94-75-7	37.3	10.0
UM815B10	4/1/2013	Acetaminophen	103-90-2	20.8	10.0
UM815B10	4/1/2013	Acetochlor	34256-82-1	265	20.0
UM815B10	6/10/2013	Acetochlor	34256-82-1	84.0	20.0
UM815B10	7/7/2014	Acetochlor	34256-82-1	130	20.0
UM815B10	4/1/2013	Acetochlor OA	194992-44-4	217	20.0
UM815B10	6/10/2013	Acetochlor OA	194992-44-4	263	20.0
UM815B10	11/13/2013	Acetochlor OA	194992-44-4	48.7	20.0
UM815B10	7/7/2014	Acetochlor OA	194992-44-4	817	20.0
UM815B10	8/11/2014	Acetochlor OA	194992-44-4	168	20.0
UM815B10	4/1/2013	Atenolol	29122-68-7	30.5	10.0
UM815B10	11/13/2013	Atenolol	29122-68-7	17.4	10.0
UM815B10	4/1/2013	Atrazine	1912-24-9	29.9	10.0
UM815B10	4/1/2013	Atrazine	1912-24-9	26.2	10.0
UM815B10	6/10/2013	Atrazine	1912-24-9	41.4	10.0
UM815B10	6/10/2013	Atrazine	1912-24-9	33.0 [1]	10.0
UM815B10	11/13/2013	Atrazine	1912-24-9	19.2	10.0
UM815B10	11/13/2013	Atrazine	1912-24-9	19.6 [1]	10.0
UM815B10	7/7/2014	Atrazine	1912-24-9	237	10.0
UM815B10	7/7/2014	Atrazine	1912-24-9	289	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM815B10	8/11/2014	Atrazine	1912-24-9	63.9	10.0
UM815B10	8/11/2014	Atrazine	1912-24-9	67.3 [1]	10.0
UM815B10	6/10/2013	Atrazine de-ethyl	6190-65-4	26.4	20.0
UM815B10	7/7/2014	Atrazine de-ethyl	6190-65-4	56.8	20.0
UM815B10	7/7/2014	Atrazine de-isopropyl	1007-28-9	25.1	20.0
UM815B10	4/1/2013	Bentazon	25057-89-0	10.2	10.0
UM815B10	8/11/2014	Bentazon	25057-89-0	11.1	10.0
UM815B10	4/1/2013	Caffeine	58-08-2	26.2	10.0
UM815B10	11/13/2013	Caffeine	58-08-2	21.7	10.0
UM815B10	11/13/2013	Carbamazepine	298-46-4	16.5	10.0
UM815B10	8/11/2014	Carbamazepine	298-46-4	11.9 [1]	10.0
UM815B10	7/7/2014	Clothianidin	210880-92-5	41.5	20.0
UM815B10	4/1/2013	Cotinine	486-56-6	13.0	10.0
UM815B10	11/13/2013	DEET	134-62-3	11.5	10.0
UM815B10	7/7/2014	DEET	134-62-3	68.7 [1]	10.0
UM815B10	8/11/2014	DEET	134-62-3	138 [1]	10.0
UM815B10	4/1/2013	Desmethylvenlafaxine	93413-62-8	18.1	10.0
UM815B10	6/10/2013	Desmethylvenlafaxine	93413-62-8	13.1 [1]	10.0
UM815B10	11/13/2013	Desmethylvenlafaxine	93413-62-8	74.7	10.0
UM815B10	7/7/2014	Desmethylvenlafaxine	93413-62-8	10.8	10.0
UM815B10	8/11/2014	Desmethylvenlafaxine	93413-62-8	37.5 [1]	10.0
UM815B10	4/1/2013	Dimethenamid	87674-68-8	13.0	10.0
UM815B10	6/10/2013	Dimethenamid	87674-68-8	21.8	10.0
UM815B10	7/7/2014	Dimethenamid	87674-68-8	119	10.0
UM815B10	6/10/2013	Dimethenamid ESA	205939-58-8	22.0	20.0
UM815B10	7/7/2014	Dimethenamid ESA	205939-58-8	107	20.0
UM815B10	4/1/2013	Furosemide	54-31-9	11.2 [1]	10.0
UM815B10	11/13/2013	Furosemide	54-31-9	26.7	10.0
UM815B10	4/1/2013	Gabapentin	60142-96-3	1080	10.0
UM815B10	6/10/2013	Gabapentin	60142-96-3	378 [1]	10.0
UM815B10	11/13/2013	Gabapentin	60142-96-3	304	10.0
UM815B10	7/7/2014	Gabapentin	60142-96-3	66.1	10.0
UM815B10	8/11/2014	Gabapentin	60142-96-3	186 [1]	10.0
UM815B10	4/1/2013	Gemfibrozil	25812-30-0	27.5 [1]	10.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM815B10	11/13/2013	Gemfibrozil	25812-30-0	18.4	10.0
UM815B10	8/11/2014	Gemfibrozil	25812-30-0	18.6	10.0
UM815B10	4/1/2013	Hydrochlorothiazide	58-93-5	57.1 [1]	10.0
UM815B10	11/13/2013	Hydrochlorothiazide	58-93-5	54.1	10.0
UM815B10	8/11/2014	Hydrochlorothiazide	58-93-5	29.1	10.0
UM815B10	4/1/2013	Hydroxybupropion	92264-81-8	23.2	10.0
UM815B10	11/13/2013	Hydroxybupropion	92264-81-8	42.6	10.0
UM815B10	8/11/2014	Hydroxybupropion	92264-81-8	14.2 [1]	10.0
UM815B10	4/1/2013	Imazethapyr	81335-77-5	33.9	10.0
UM815B10	6/10/2013	Imazethapyr	81335-77-5	19.8	10.0
UM815B10	7/7/2014	Imazethapyr	81335-77-5	198 [1]	20.0
UM815B10	8/11/2014	Imazethapyr	81335-77-5	57.9 [1]	20.0
UM815B10	4/1/2013	Lamotrigine	84057-84-1	37.2	10.0
UM815B10	6/10/2013	Lamotrigine	84057-84-1	13.2 [1]	10.0
UM815B10	11/13/2013	Lamotrigine	84057-84-1	85.2	10.0
UM815B10	8/11/2014	Lamotrigine	84057-84-1	71.3 [1]	10.0
UM815B10	4/1/2013	Levorphanol	77-07-6	16.2	10.0
UM815B10	11/13/2013	Levorphanol	77-07-6	27.8	10.0
UM815B10	11/13/2013	Lidocaine	137-58-6	11.6	10.0
UM815B10	8/11/2014	Lidocaine	137-58-6	12.4 [1]	10.0
UM815B10	4/1/2013	Metformin	657-24-9	111	10.0
UM815B10	6/10/2013	Metformin	657-24-9	73.9 [1]	10.0
UM815B10	11/13/2013	Metformin	657-24-9	127	10.0
UM815B10	7/7/2014	Metformin	657-24-9	28.4	10.0
UM815B10	8/11/2014	Metformin	657-24-9	76.2 [1]	10.0
UM815B10	7/7/2014	Methylparaben	99-76-3	35.9	25.0
UM815B10	8/11/2014	Methylparaben	99-76-3	45.0	25.0
UM815B10	4/1/2013	Metolachlor	51218-45-2	867	10.0
UM815B10	6/10/2013	Metolachlor	51218-45-2	21.8	10.0
UM815B10	7/7/2014	Metolachlor	51218-45-2	323	10.0
UM815B10	8/11/2014	Metolachlor	51218-45-2	26.5	10.0
UM815B10	4/1/2013	Metolachlor ESA	947601-85-6	313	20.0
UM815B10	6/10/2013	Metolachlor ESA	947601-85-6	264	20.0
UM815B10	11/13/2013	Metolachlor ESA	947601-85-6	193	20.0

Table B-1 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Mississippi National River and Recreation Area in 2013 and 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
UM815B10	7/7/2014	Metolachlor ESA	947601-85-6	513	20.0
UM815B10	8/11/2014	Metolachlor ESA	947601-85-6	239	20.0
UM815B10	11/13/2013	Metoprolol	37350-58-6	20.7	10.0
UM815B10	8/11/2014	Metoprolol	37350-58-6	11.7 [1]	10.0
UM815B10	11/13/2013	Modafinil acid	112111-45-2	11.5	10.0
UM815B10	4/1/2013	Pregabalin	148553-50-8	15.4	10.0
UM815B10	8/11/2014	Propachlor ESA	947601-88-9	485	20.0
UM815B10	6/10/2013	Ranitidine	66357-35-5	32.0 [1]	10.0
UM815B10	11/13/2013	Ranitidine	66357-35-5	20.3	10.0
UM815B10	8/11/2014	Sotalol	959-24-0	10.7 [1]	10.0
UM815B10	4/1/2013	Sulfamethoxazole	723-46-6	31.8	10.0
UM815B10	11/13/2013	Sulfamethoxazole	723-46-6	77.7	10.0
UM815B10	8/11/2014	Sulfamethoxazole	723-46-6	59.2	10.0
UM815B10	4/1/2013	Theobromine	83-67-0	20.7	10.0
UM815B10	7/7/2014	Thiamethoxam	153719-23-4	23.2	20.0
UM815B10	4/1/2013	Tramadol	27203-92-5	14.7	10.0
UM815B10	6/10/2013	Tramadol	27203-92-5	11.3 [1]	10.0
UM815B10	11/13/2013	Tramadol	27203-92-5	28.2	10.0
UM815B10	8/11/2014	Tramadol	27203-92-5	21.2 [1]	10.0
UM815B10	11/13/2013	Triamterene	396-01-0	10.6	10.0
UM815B10	4/1/2013	Triclopyr	55335-06-3	58.5	20.0
UM815B10	4/1/2013	Trimethoprim	738-70-5	12.1	10.0
UM815B10	11/13/2013	Trimethoprim	738-70-5	18.5	10.0
UM815B10	8/11/2014	Trimethoprim	738-70-5	16.0 [1]	10.0
UM815B10	11/13/2013	Venlafaxine	93413-44-6	26.6	10.0
UM815B10	8/11/2014	Venlafaxine	93413-44-6	10.8 [1]	10.0

Table B-2. Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Apostle Islands National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
APIS_Outer	8/4/2014	Gabapentin	60142-96-3	11.9 [1]	10.0
APIS_Outer	7/7/2014	Imazethapyr	81335-77-5	20.4 [1]	20.0
APIS_Outer	7/7/2014	Tri(2-butoxyethyl) Phosphate	78-51-3	299 [1]	50.0
APIS09-03	8/4/2014	Gabapentin	60142-96-3	12.5 [1]	10.0
APIS09-09	7/8/2014	Gabapentin	60142-96-3	13.7	10.0
APIS09-09	8/5/2014	Gabapentin	60142-96-3	11.1 [1]	10.0
APIS09-09	7/8/2014	Metformin	657-24-9	15.2	10.0
APIS09-09	7/8/2014	Tri(2-butoxyethyl) Phosphate	78-51-3	832 [1]	50.0
APIS09-14	6/9/2014	Caffeine	58-08-2	31.0	25.0
APIS09-14	6/9/2014	DEET	134-62-3	15.8 [1]	10.0
APIS09-30	8/4/2014	Gabapentin	60142-96-3	10.1 [1]	10.0
APIS09-30	7/8/2014	Tri(2-butoxyethyl) Phosphate	78-51-3	387 [1]	50.0
APIS09-30	6/9/2014	Tri(dichloroisopropyl) Phosphate	13674-87-8	50.9	50.0
APIS09-62	8/4/2014	Gabapentin	60142-96-3	12.3 [1]	10.0
APIS09-62	6/9/2014	Imazethapyr	81335-77-5	65.3	20.0
APIS09-84	6/3/2014	Bisphenol A	80-05-7	56.2 [1]	50.0
APIS09-84	8/4/2014	Imazethapyr	81335-77-5	22.9 [1]	20.0
APIS09-84	7/7/2014	Tri(2-butoxyethyl) Phosphate	78-51-3	405 [1]	50.0
APIS09-84	6/3/2014	Triclosan	3380-34-5	74.9 [1]	50.0

Table B-3. Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU029	11/12/2014	2,4-D	94-75-7	166	10.0
INDU029	11/12/2014	Caffeine	58-08-2	86.3 [1]	25.0
INDU029	11/12/2014	Cotinine	486-56-6	16.7 [1]	10.0
INDU029	11/12/2014	DEET	134-62-3	48.6 [1]	10.0
INDU029	11/12/2014	Imazethapyr	81335-77-5	22.1	20.0
INDU029	11/12/2014	MCP	7085-19-0	56.2	20.0
INDU029	11/12/2014	Metformin	657-24-9	15.8 [1]	10.0
INDU029	11/12/2014	Metolachlor ESA	947601-85-6	44.4	20.0
INDU029	11/12/2014	Monuron	150-68-5	15.8	10.0
INDU082	11/12/2014	2,4-D	94-75-7	65.0	10.0
INDU082	11/12/2014	Acetaminophen	103-90-2	18.8 [1]	10.0
INDU082	11/12/2014	Acetophenone	98-86-2	67.7	50.0
INDU082	11/12/2014	Atrazine	1912-24-9	12.2	10.0
INDU082	11/12/2014	Bisphenol A	80-05-7	295	50.0
INDU082	11/12/2014	Caffeine	58-08-2	200 [1]	25.0
INDU082	11/12/2014	Caffeine	58-08-2	215	50.0
INDU082	11/12/2014	Camphor	76-22-2	89.6	50.0
INDU082	11/12/2014	Cotinine	486-56-6	18.7 [1]	10.0
INDU082	11/12/2014	DEET	134-62-3	43.0 [1]	10.0
INDU082	11/12/2014	Gabapentin	60142-96-3	45.8 [1]	10.0
INDU082	11/12/2014	Imazethapyr	81335-77-5	37.8	20.0
INDU082	11/12/2014	Metformin	657-24-9	10.5 [1]	10.0
INDU082	11/12/2014	Methyl Salicylate	119-36-8	53.4	50.0
INDU082	11/12/2014	Metolachlor ESA	947601-85-6	74.3	20.0
INDU082	11/12/2014	Naproxen	22204-53-1	25.5	25.0
INDU082	11/12/2014	Simazine	122-34-9	24.1	20.0
INDU144	11/12/2014	2,4-D	94-75-7	60.9	10.0
INDU144	11/12/2014	Acetaminophen	103-90-2	22.2 [1]	10.0
INDU144	11/12/2014	Acetochlor OA	194992-44-4	28.3	20.0
INDU144	11/12/2014	Cotinine	486-56-6	10.3 [1]	10.0
INDU144	11/12/2014	DEET	134-62-3	18.0 [1]	10.0
INDU144	11/12/2014	Gabapentin	60142-96-3	220 [1]	10.0
INDU144	11/12/2014	Imazethapyr	81335-77-5	46.9	20.0
INDU144	11/12/2014	Metolachlor ESA	947601-85-6	27.4	20.0
INDU144	11/12/2014	Simazine	122-34-9	35.5	20.0
INDU144	11/12/2014	Tebuthiuron	34014-18-1	86.5	10.0

Table B-3 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU144	11/12/2014	Triclopyr	55335-06-3	33.5	20.0
INDU164	11/12/2014	2,4-D	94-75-7	42.5	10.0
INDU164	11/12/2014	Acetaminophen	103-90-2	12.4 [1]	10.0
INDU164	11/12/2014	Caffeine	58-08-2	34.4 [1]	25.0
INDU164	11/12/2014	DEET	134-62-3	40.2 [1]	10.0
INDU164	11/12/2014	Gabapentin	60142-96-3	218 [1]	10.0
INDU164	11/12/2014	Metolachlor ESA	947601-85-6	23.1	20.0
INDU164	11/12/2014	Simazine	122-34-9	35.3	20.0
INDU164	11/12/2014	Tebuthiuron	34014-18-1	40.2	10.0
INDU164	11/12/2014	Triclopyr	55335-06-3	41.1	20.0
INDU243	11/12/2014	2,4-D	94-75-7	20.8	10.0
INDU243	11/12/2014	Acetochlor OA	194992-44-4	21.5	20.0
INDU243	11/12/2014	DEET	134-62-3	50.9 [1]	10.0
INDU243	11/12/2014	Gabapentin	60142-96-3	255 [1]	10.0
INDU243	11/12/2014	Imazethapyr	81335-77-5	102	20.0
INDU243	11/12/2014	MCP	7085-19-0	23.4	20.0
INDU243	11/12/2014	Meprobamate	57-53-4	19.2 [1]	10.0
INDU243	11/12/2014	Metolachlor ESA	947601-85-6	122	20.0
INDU243	11/12/2014	Naproxen	22204-53-1	27.1	25.0
INDU262	11/12/2014	2,4-D	94-75-7	19.2	10.0
INDU262	11/12/2014	Acetochlor OA	194992-44-4	32.6	20.0
INDU262	11/12/2014	DEET	134-62-3	17.3 [1]	10.0
INDU262	11/12/2014	Gabapentin	60142-96-3	51.2 [1]	10.0
INDU262	11/12/2014	Metolachlor ESA	947601-85-6	103	20.0
INDU328	11/12/2014	2,4,5-TP	93-72-1	12.3	10.0
INDU328	11/12/2014	2,4-D	94-75-7	23.3	10.0
INDU328	11/12/2014	Acetochlor OA	194992-44-4	54.6	20.0
INDU328	11/12/2014	DEET	134-62-3	25.7 [1]	10.0
INDU328	11/12/2014	Imazethapyr	81335-77-5	32.1	20.0
INDU328	11/12/2014	Metolachlor ESA	947601-85-6	437	20.0
INDU405	11/12/2014	2,4-D	94-75-7	15.1	10.0
INDU405	11/12/2014	Acetochlor OA	194992-44-4	32.7	20.0
INDU405	11/12/2014	DEET	134-62-3	27.1 [1]	10.0
INDU405	11/12/2014	Gabapentin	60142-96-3	20.7 [1]	10.0
INDU405	11/12/2014	Imazethapyr	81335-77-5	33.2	20.0
INDU405	11/12/2014	Metolachlor ESA	947601-85-6	104	20.0

Table B-3 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU405	11/12/2014	Valsartan	137862-53-4	14.2 [1]	10.0
INDU459	11/12/2014	2,4-D	94-75-7	42.9	10.0
INDU459	11/12/2014	Acetochlor OA	194992-44-4	87.2	20.0
INDU459	11/12/2014	Atrazine	1912-24-9	10.8	10.0
INDU459	11/12/2014	Atrazine de-isopropyl	1007-28-9	50.4	20.0
INDU459	11/12/2014	DEET	134-62-3	115 [1]	10.0
INDU459	11/12/2014	Dimethenamid ESA	205939-58-8	26.8	20.0
INDU459	11/12/2014	Gabapentin	60142-96-3	10.9 [1]	10.0
INDU459	11/12/2014	Imazethapyr	81335-77-5	82.5	20.0
INDU459	11/12/2014	Methylparaben	99-76-3	84.2	25.0
INDU459	11/12/2014	Metolachlor	51218-45-2	14.0	10.0
INDU459	11/12/2014	Metolachlor ESA	947601-85-6	348	20.0
INDU459	11/12/2014	Simazine	122-34-9	1350 [1]	20.0
INDU459	11/12/2014	Triclopyr	55335-06-3	23.3	20.0
INDU475	11/12/2014	2,4-D	94-75-7	22.6	10.0
INDU475	11/12/2014	Acetochlor OA	194992-44-4	71.1	20.0
INDU475	11/12/2014	Bisphenol A	80-05-7	350	50.0
INDU475	11/12/2014	DEET	134-62-3	13.5 [1]	10.0
INDU475	11/12/2014	Dimethenamid ESA	205939-58-8	30.2	20.0
INDU475	11/12/2014	Equilenin	517-09-9	187	100
INDU475	11/12/2014	Estrone	53-16-7	121	100
INDU475	11/12/2014	Imazethapyr	81335-77-5	92.4	20.0
INDU475	11/12/2014	Metolachlor ESA	947601-85-6	298	20.0
INDU475	11/12/2014	Simazine	122-34-9	421	20.0
INDU475	11/12/2014	Triclopyr	55335-06-3	20.1	20.0
INDU503	11/12/2014	2,4-D	94-75-7	25.0	10.0
INDU503	11/12/2014	Acetochlor OA	194992-44-4	46.0	20.0
INDU503	11/12/2014	DEET	134-62-3	19.2 [1]	10.0
INDU503	11/12/2014	Desmethylvenlafaxine	93413-62-8	45.9 [1]	10.0
INDU503	11/12/2014	Diclofenac	15307-86-5	12.6	10.0
INDU503	11/12/2014	Furosemide	54-31-9	16.3	10.0
INDU503	11/12/2014	Gabapentin	60142-96-3	76.1 [1]	10.0
INDU503	11/12/2014	Hydrochlorothiazide	58-93-5	64.1	10.0
INDU503	11/12/2014	Hydroxybupropion	92264-81-8	20.9 [1]	10.0
INDU503	11/12/2014	Imazethapyr	81335-77-5	53.4	20.0
INDU503	11/12/2014	Lamotrigine	84057-84-1	32.1 [1]	10.0

Table B-3 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU503	11/12/2014	Levorphanol	77-07-6	22.3 [1]	10.0
INDU503	11/12/2014	Meprobamate	57-53-4	13.8 [1]	10.0
INDU503	11/12/2014	Metformin	657-24-9	35.4 [1]	10.0
INDU503	11/12/2014	Metolachlor ESA	947601-85-6	204	20.0
INDU503	11/12/2014	Simazine	122-34-9	594	20.0
INDU503	11/12/2014	Sotalol	959-24-0	22.2 [1]	10.0
INDU503	11/12/2014	Sulfamethoxazole	723-46-6	11.5	10.0
INDU503	11/12/2014	Tramadol	27203-92-5	29.6 [1]	10.0
INDU503	11/12/2014	Venlafaxine	93413-44-6	15.5 [1]	10.0
INDU539	11/12/2014	2,4-D	94-75-7	35.2	10.0
INDU539	11/12/2014	Acetochlor OA	194992-44-4	33.6	20.0
INDU539	11/12/2014	Caffeine	58-08-2	36.3 [1]	25.0
INDU539	11/12/2014	DEET	134-62-3	17.9 [1]	10.0
INDU539	11/12/2014	Desmethylvenlafaxine	93413-62-8	25.1 [1]	10.0
INDU539	11/12/2014	Furosemide	54-31-9	11.3	10.0
INDU539	11/12/2014	Gabapentin	60142-96-3	52.2 [1]	10.0
INDU539	11/12/2014	Hydrochlorothiazide	58-93-5	32.2	10.0
INDU539	11/12/2014	Hydroxybupropion	92264-81-8	12.8 [1]	10.0
INDU539	11/12/2014	Imazethapyr	81335-77-5	38.4	20.0
INDU539	11/12/2014	Lamotrigine	84057-84-1	22.7 [1]	10.0
INDU539	11/12/2014	Levorphanol	77-07-6	16.1 [1]	10.0
INDU539	11/12/2014	Metformin	657-24-9	24.3 [1]	10.0
INDU539	11/12/2014	Metolachlor ESA	947601-85-6	196	20.0
INDU539	11/12/2014	Simazine	122-34-9	348	20.0
INDU539	11/12/2014	Sotalol	959-24-0	10.5 [1]	10.0
INDU539	11/12/2014	Tramadol	27203-92-5	16.8 [1]	10.0
INDU900	11/12/2014	Acetophenone	98-86-2	50.3	50.0
INDU900	11/12/2014	Atrazine	1912-24-9	32.3	10.0
INDU900	11/12/2014	Atrazine	1912-24-9	28.3 [1]	10.0
INDU900	11/12/2014	Atrazine de-ethyl	6190-65-4	39.0	20.0
INDU900	11/12/2014	Caffeine	58-08-2	31.6 [1]	25.0
INDU900	11/12/2014	DEET	134-62-3	10.8 [1]	10.0
INDU900	11/12/2014	Gabapentin	60142-96-3	18.1 [1]	10.0
INDU900	11/12/2014	Hydrochlorothiazide	58-93-5	12.7	10.0
INDU900	11/12/2014	Imazethapyr	81335-77-5	25.0	20.0
INDU900	11/12/2014	Metformin	657-24-9	39.1 [1]	10.0

Table B-3 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU900	11/12/2014	Metolachlor ESA	947601-85-6	27.8	20.0
INDU900	11/12/2014	Propachlor OA	70628-36-3	15.2	10.0
INDU900	11/12/2014	Triphenyl phosphate	115-86-6	82.0	50.0
INDU901	11/12/2014	2,4-D	94-75-7	106	10.0
INDU901	11/12/2014	Acetaminophen	103-90-2	53.2 [1]	10.0
INDU901	11/12/2014	Acetochlor OA	194992-44-4	413	20.0
INDU901	11/12/2014	Atrazine	1912-24-9	49.7	10.0
INDU901	11/12/2014	Atrazine	1912-24-9	36.7 [1]	10.0
INDU901	11/12/2014	Atrazine de-ethyl	6190-65-4	38.6	20.0
INDU901	11/12/2014	Atrazine de-isopropyl	1007-28-9	116	20.0
INDU901	11/12/2014	Caffeine	58-08-2	25.8 [1]	25.0
INDU901	11/12/2014	Clothianidin	210880-92-5	74.7	20.0
INDU901	11/12/2014	DEET	134-62-3	17.0 [1]	10.0
INDU901	11/12/2014	Dichloroprop	120-36-5	10.9	10.0
INDU901	11/12/2014	Dimethenamid ESA	205939-58-8	230	20.0
INDU901	11/12/2014	Gabapentin	60142-96-3	70.2 [1]	10.0
INDU901	11/12/2014	Imazaquin	81335-37-7	20.2	20.0
INDU901	11/12/2014	Imazethapyr	81335-77-5	492	20.0
INDU901	11/12/2014	Metolachlor	51218-45-2	50.8	10.0
INDU901	11/12/2014	Metolachlor ESA	947601-85-6	1550 [1]	20.0
INDU901	11/12/2014	Simazine	122-34-9	3140 [1]	20.0
INDU901	11/12/2014	Triclopyr	55335-06-3	87.6	20.0
INDU901	11/12/2014	Valsartan	137862-53-4	27.7 [1]	10.0
INDU902	11/12/2014	2,4-D	94-75-7	18.9	10.0
INDU902	11/12/2014	Acetochlor OA	194992-44-4	21.9	20.0
INDU902	11/12/2014	Imazethapyr	81335-77-5	27.0	20.0
INDU902	11/12/2014	Metolachlor ESA	947601-85-6	91.1	20.0
INDU903	11/12/2014	DEET	134-62-3	23.9 [1]	10.0
INDU904	11/12/2014	2,4-D	94-75-7	41.5	10.0
INDU904	11/12/2014	Acetochlor OA	194992-44-4	105	20.0
INDU904	11/12/2014	Atenolol	29122-68-7	11.9 [1]	10.0
INDU904	11/12/2014	Atrazine	1912-24-9	14.0	10.0
INDU904	11/12/2014	DEET	134-62-3	24.6 [1]	10.0
INDU904	11/12/2014	Imazethapyr	81335-77-5	22.9	20.0
INDU904	11/12/2014	Meprobamate	57-53-4	18.0 [1]	10.0
INDU904	11/12/2014	Metolachlor ESA	947601-85-6	125	20.0

Table B-3 (continued). Pesticides, pharmaceuticals, personal care products, and wastewater indicators detected at Indiana Dunes National Lakeshore in 2014. Results flagged with [1] should be considered estimates due to failure of one or more laboratory quality assurance/quality control measures. The chemical abstracts services (CAS) number is a unique numerical identifier for chemicals described in the open scientific literature. MRL = method reporting limit.

Site ID	Date Sampled	Analyte	CAS Number	Result (ng/L)	MRL (ng/L)
INDU904	11/12/2014	Simazine	122-34-9	29.7	20.0
INDU905	11/12/2014	2,4-D	94-75-7	80.6	10.0
INDU905	11/12/2014	Acetochlor OA	194992-44-4	77.8	20.0
INDU905	11/12/2014	Atrazine	1912-24-9	10.1	10.0
INDU905	11/12/2014	DEET	134-62-3	16.7 [1]	10.0
INDU905	11/12/2014	Imazethapyr	81335-77-5	45.5	20.0
INDU905	11/12/2014	MCPPP	7085-19-0	33.1	20.0
INDU905	11/12/2014	Metolachlor	57837-19-1	13.3	10.0
INDU905	11/12/2014	Metolachlor ESA	947601-85-6	158	20.0
INDU905	11/12/2014	Simazine	122-34-9	36.4	20.0
INDU905	11/12/2014	Triclopyr	55335-06-3	35.7	20.0
INDU906	11/12/2014	2,4-D	94-75-7	18.5	10.0
INDU906	11/12/2014	Caffeine	58-08-2	49.7 [1]	25.0
INDU906	11/12/2014	DEET	134-62-3	46.6 [1]	10.0
INDU906	11/12/2014	Imazethapyr	81335-77-5	29.0	20.0
INDU906	11/12/2014	Metolachlor ESA	947601-85-6	89.8	20.0

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