

Prepared in cooperation with the U.S. Environmental Protection Agency

# **Methods Used to Characterize the Chemical Composition and Biological Activity of Environmental Waters Throughout the United States, 2012–14**

Open-File Report 2017–1011

**Cover.** Sycamore Slough at County Line Road near Dunnigan, California. Photograph by Jim Orlando, USGS, June 18, 2013.

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By Kristin M. Romanok, Timothy J. Reilly, Larry Barber, Scott Boone, Herbert T. Buxton, William T. Foreman, Edward T. Furlong, Michelle Hladik, Luke R. Iwanowicz, Celeste Journey, Dana W. Kolpin, Kathryn Kuivila, Keith A. Loftin, Marc A. Mills, Michael T. Meyer, James L. Orlando, Kelly L. Smalling, Daniel L. Villeneuve, and Paul M. Bradley

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

## **U.S. Department of the Interior**

RYAN K. ZINKE, Secretary

## **U.S. Geological Survey**

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# Contents

Acknowledgments .....	iii
Abstract .....	1
Introduction.....	1
Purpose and Scope .....	2
Methods.....	2
Sampling Site Selection.....	2
Sampling Methods.....	2
Analytical Methods.....	3
National Water Quality Laboratory.....	3
Ambient Purgeable Volatile Organic Compounds .....	8
Heat Purgeable Volatile Organic Compounds.....	8
Steroid Hormones and Related Compounds .....	8
Wastewater Indicator Compounds and Selected Pharmaceuticals.....	8
Pesticides and Pesticide Metabolites.....	8
Human-Use Pharmaceuticals, Pharmaceutical Metabolites, and Selected Polar Organic Compounds.....	8
Selected Halogenated Organic Compounds.....	8
Selected Trace Elements, Cations, Anions, and Physical Properties .....	9
Selected Nutrients and Total Organic Carbon .....	9
Organic Geochemistry Research Laboratory .....	9
Acetamide Herbicides and Degradation Products .....	10
Glyphosate, Glufosinate, and Aminomethylphosphonic Acid .....	10
Steroid Hormones and Phytoestrogens .....	10
Triazine and Phenylurea Herbicides.....	10
Selected Antibiotics .....	10
Pesticide Fate Research Laboratory .....	10
Pesticide and Pesticide Degradates .....	10
Diuron and Diuron Degradates and Neonicotinoid Insecticides.....	10
Disinfection By-Products .....	10
National Research Program .....	11
USGS National Fish Health Research Laboratory.....	11
Bioluminescent Yeast Estrogen Screen.....	11
Transgenic Zebrafish Embryo Estrogen Bioassay.....	11
U.S. Environmental Protection Agency Laboratories .....	12
Perfluorinated Compounds .....	12
Bioassays .....	12
Quality Assurance/Quality Control.....	13
Field.....	13
Laboratory.....	13
National Water Quality Laboratory.....	13
Pesticides and Pesticide Metabolites.....	13

Human-Use Pharmaceuticals, Pharmaceutical Metabolites, and Selected Polar Organic Compounds .....	13
Steroid Hormones and Related Compounds .....	13
Selected Halogenated Organic Compounds .....	13
Heat Purgeable Volatile Organic Compounds.....	14
Ambient Purgeable Volatile Organic Compounds .....	14
Organic Geochemistry Research Laboratory .....	14
Pesticide Fate Research Group.....	15
U.S. Environmental Protection Agency Laboratories .....	15
National Research Program .....	15
Geographic Information System Methods .....	15
Results .....	17
References Cited.....	17
Appendix 1. Example of protocols provided to field personnel for the preparation, collection, processing, and shipping of samples collected for the Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.....	23
Appendix 2. Site-specific pages containing site, sampling, and other basin information as well as physical parameter and inorganic chemical data collected at each site .....	29

## Figure

1. Map showing site locations and 2011 National Land Cover for sites where samples were collected for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.....3

## Tables

1. Station information and dates samples were collected for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014 .....
2. Analyzing laboratory and method, bottle type, and treatment required for sampling conducted nationwide as part of the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.....6
3. Compound information for analyses performed by various laboratories for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014 (Available in a separate Excel file.)
4. Aminocarboxylic acid complexing agents and non-ionic surfactant degradate compound information and spiked concentrations of surrogate and internal standards analyzed using gas chromatography with mass spectrometry at the National Research Program laboratory in Boulder, Colorado .....
5. Compound information for analyses of perfluorinated compounds performed by the U.S. Environmental Protection Agency, Research and Development laboratories in the John C. Stennis Space Center in Mississippi, November 2012–August 2013 .....
6. Summary of field quality-assurance samples collected from sites nationwide for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014 .....

7.	Quality-control procedures for perfluorinated compounds analyzed at the U.S. Environmental Protection Agency, Environmental Chemistry Branch at John C. Stennis Space Center in Mississippi, for surface-water samples collected as part of the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–December 2013 .....	15
8.	Landscape metric data types and source information .....	16



## Conversion Factors

U.S. customary units to International System of Units

Multiply	By	To obtain
Length		
inch (in.)	2.54	centimeter (cm)
foot (ft)	0.3048	meter (m)
mile (mi)	1.609	kilometer (km)
yard (yd)	0.9144	meter (m)
Area		
acre	4,047	square meter (m <sup>2</sup> )
acre	0.004047	square kilometer (km <sup>2</sup> )
Volume		
ounce, fluid (fl. oz)	0.02957	liter (L)
cubic inch (in <sup>3</sup> )	16.39	cubic centimeter (cm <sup>3</sup> )
cubic inch (in <sup>3</sup> )	0.01639	liter (L)
cubic foot (ft <sup>3</sup> )	0.02832	cubic meter (m <sup>3</sup> )
cubic yard (yd <sup>3</sup> )	0.7646	cubic meter (m <sup>3</sup> )
acre-foot (acre-ft)	1,233	cubic meter (m <sup>3</sup> )
Flow rate		
cubic foot per second (ft <sup>3</sup> /s)	0.02832	cubic meter per second (m <sup>3</sup> /s)
Mass		
ounce, avoirdupois (oz)	28.35	gram (g)
pound, avoirdupois (lb)	0.4536	kilogram (kg)
ton per day (ton/d)	0.9072	metric ton per day
ton per day per square mile [(ton/d)/mi <sup>2</sup> ]	0.3503	megagram per day per square kilometer [(Mg/d)/km <sup>2</sup> ]
ton per year (ton/yr)	0.9072	metric ton per year
Application rate		
pounds per acre per year [(lb/acre)/yr]	1.121	kilograms per hectare per year [(kg/ha)/yr]

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

$$^{\circ}\text{F} = (1.8 \times ^{\circ}\text{C}) + 32.$$

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

$$^{\circ}\text{C} = (^{\circ}\text{F} - 32) / 1.8.$$

## Datum

Horizontal coordinate information is referenced to the North American Datum of 1983 (NAD 83).

## Supplemental Information

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius ( $\mu\text{S}/\text{cm}$  at 25 °C).

Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L), micrograms per liter ( $\mu\text{g}/\text{L}$ ), or nanograms per liter (ng/L).

## Abbreviations

ANC	acid neutralizing capacity
BLYES	bioluminescent yeast estrogen screen
°C	Celsius
DCM	dichloromethane
DEE	diethyl ether
DMSO	dimethyl sulfoxide
DTPA	diethylenetriaminepentaacetic acid
ECNIM	electron-capture negative ionization mode
EDTA	ethylenediaminetetraacetic acid
EPA	U.S. Environmental Protection Agency
ESI	electrospray ionizations
g	grams
GC	gas chromatography
GIS	Geographic Information System
HPLC	high-performance liquid chromatography
mg/L	milligrams per liter
mL	milliliter
MRM	multiple reaction monitoring
MS	mass spectrometry
MS–MS	Tandem Mass Spectrometry
N	Normality
NaCl	sodium chloride
NAWQA	National Water-Quality Assessment Project
ng/L	nanograms per liter
NFHRL	National Fish Health Research Laboratory
NHD	National Hydrography Dataset
NIEHS	National Institute of Environmental Health Sciences

NPDES	National Pollutant Discharge Elimination System
NP1EC	nonylphenolmonothoxycarboxylate
NP2EC	4-nonylphenoldiethoxycarboxylate
NRP	National Research Program
NTA	nitrilotriacetic acid
NTU	nephelometric turbidity units
NWQL	National Water Quality Laboratory
OGRL	Organic Geochemistry Research Laboratory
PCB	polychlorinated biphenyl
PFRG	Pesticide Fate Research Group
pg/ml	picograms per milliliter
PTV	programmer-temperature vaporization
QA/QC	quality assurance and quality control
REACH	Registration, Evaluation, and Authorization of Chemicals
RNA	ribonucleic acid
RTP	Research Triangle Park
SIM	selected ion monitoring
SPE	solid phase extraction
TIC	tentatively identified compounds
TRI	Toxic Release Inventory
µg/L	micrograms per liter
µL	microliters
µm	micrometer
µS/cm	microsiemens per centimeter
USGS	U.S. Geological Survey
VOC	volatile organic compound
v:v	volume to volume
WBD	Watershed Boundary Dataset

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By Kristin M. Romanok,<sup>1</sup> Timothy J. Reilly,<sup>1</sup> Larry Barber,<sup>1</sup> Scott Boone,<sup>2</sup> Herbert T. Buxton,<sup>1</sup> William T. Foreman,<sup>1</sup> Edward T. Furlong,<sup>1</sup> Michelle Hladik,<sup>1</sup> Luke R. Iwanowicz,<sup>1</sup> Celeste Journey,<sup>1</sup> Dana W. Kolpin,<sup>1</sup> Kathryn Kuivila,<sup>1</sup> Keith A. Loftin,<sup>1</sup> Marc A. Mills,<sup>2</sup> Michael T. Meyer,<sup>1</sup> James L. Orlando,<sup>1</sup> Kelly L. Smalling,<sup>1</sup> Daniel L. Villeneuve,<sup>2</sup> and Paul M. Bradley<sup>1</sup>

## Abstract

A vast array of chemical compounds are in wide commercial use in the United States, and the potential ecological and human-health effect of exposure to chemical mixtures has been identified as a high priority in environment health science. Awareness of the potential effects of low-level chemical exposures is rising. The U.S. Geological Survey, in cooperation with the U.S. Environmental Protection Agency, conducted a study in which samples were collected from 38 streams in 25 States to provide an overview of contaminants found in stream water across the Nation. Additionally, biological screening assays were used to help determine any potential ecological and human-health effects of these chemical mixtures and to prioritize target chemicals for future toxicological studies. This report describes the site locations and the sampling and analytical methods and quality-assurance procedures used in the study.

## Introduction

More than 30,000 chemicals are in wide commercial use in the United States (Judson and others, 2009). About 143,000 substances were preregistered in the European Union's Registration, Evaluation, and Authorization of Chemicals (REACH) program in December 2008 (European Chemicals Agency [ECHA], 2011). The potential ecological and human-health effect of exposure to chemical mixtures has been identified as a high priority in environment health science. Furthermore, awareness of the potential effects of low-level chemical exposures is rising (Birnbau, 2012). The National Institute of Environmental Health Sciences (NIEHS) states,

The evaluation of human-health effects from multiple environmental exposures represents a special challenge to the research community due to the inherent complexity of the topic. ... Continuous human exposure to complex and dynamic mixtures precludes directly testing the toxicity of each possible exposure combination. Therefore, predictive models of mixture toxicity must be developed and validated in order to characterize the hazard associated with complex exposures. In order to develop these models, a better understanding is required of both the composition of real-world exposures and the fundamental principles of chemical interactions (NIEHS, 2011).

Chemical analytical capabilities have advanced substantially over the past decade. Chemical contaminant concentrations detected in environmental samples commonly reach below the nanogram-per-liter level. Advancements in detection technologies also have broadened the types of chemicals that can typically be separated from environmental matrices and accurately quantified (Ballesteros-Gómez and Rubio, 2011; Richardson and Ternes, 2011). At the same time, forensic approaches to identify unknown contaminants in samples of environmental media have also advanced. Phillips and others (2010) used an analysis of unknowns and subsequently tentatively identified compounds (TIC) to determine that pharmaceutical manufacturing facilities were releasing substantial amounts of opioids and muscle relaxants, most of which had never before been measured in the environment. Thurman and Ferrer (2012) used liquid chromatography/quadrupole-time-of-flight mass spectrometry (LC/Q-TOF-MS) and metabolic profiling of human urine to identify dextromethorphan metabolites at levels 5 to 10 times greater than the parent compound; neither dextromethorphan nor its metabolites had been reported previously in streams. Aided by improved TIC analysis and new analytical technologies for chemical separation and identification, these new capabilities provide an

<sup>1</sup>U.S. Geological Survey.

<sup>2</sup>U.S. Environmental Protection Agency.

unprecedented ability to identify and quantify chemical mixtures in environmental samples as well as a unique opportunity to characterize ecological and human exposures in a wide range of environmental settings. Settings affected by multiple contaminant sources include industrial and municipal wastewater discharges, crop and animal agriculture, mining, land application of manures and bio-solids, urban runoff, and other point and nonpoint sources.

At the same time, biological screening assays, such as those being applied to assess the biological activity of a wide range of chemicals by the U.S. Environmental Protection Agency (EPA), NIEHS, and other Federal agencies through the Toxicology in the 21st Century (Tox21) program (EPA, 2011), provide an improved approach for testing large numbers of chemicals, identifying potential toxicity pathways, and prioritizing chemicals for more detailed toxicological studies. An approach for testing the biological activity of contaminants on a chemical-by-chemical basis, however, is limited by the facts that ecological and human-health impacts are affected by the level of environmental exposure, and environmental exposures typically occur to complex mixtures of synthetic and naturally occurring chemicals. Furthermore, those chemical mixtures include environmental degradation by-products, disinfection by-products, and metabolites, which are often unknown and require deductive reasoning and analysis to identify.

## Purpose and Scope

The purpose of this report is to describe the methods and procedures used to determine the chemical composition and associated biological activity of environmental waters from selected sites throughout the United States. This pilot study combines the important strengths and capabilities of the U.S. Geological Survey (USGS) in environmental chemistry, a history of studying emerging contaminants in a wide range of environmental settings across the Nation, and a national cadre of field technicians trained to collect and process environmental samples using consistent protocols, a description of which can be found in Buxton and others (2015). Health risks associated with environmental contamination are associated with both the toxicity of a chemical or chemical mixture and the level of exposure to that chemical or mixture. Incorporation of field-based testing using actual environmental samples and an aggressive chemical characterization approach to define contaminant mixtures and levels provides an important opportunity to complement existing contaminant prioritization processes. The USGS is incorporating a forensic chemical characterization capability into its process for prioritizing chemicals for methods development and environmental studies. Because USGS capabilities to apply biological screening techniques are limited, a partnership with the EPA has been established, and the descriptions of those analyses are included herein.

## Methods

Sampling and analytical methods used to determine the chemical composition and associated biological activity of environmental water samples by USGS field and laboratory personnel are described. Quality-assurance and quality-control (QA/QC) procedures are discussed.

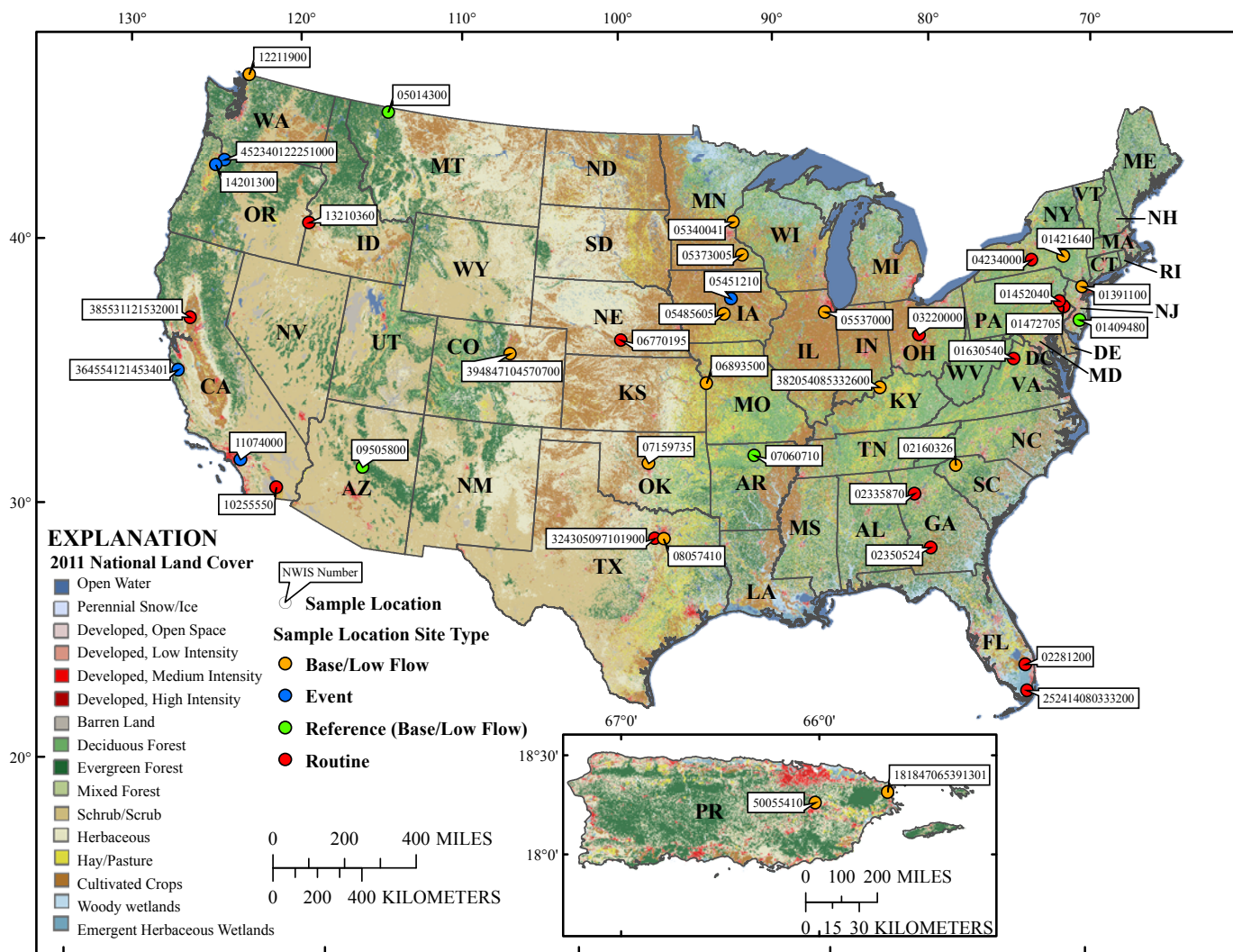
### Sampling Site Selection

Nationally, 38 sampling sites were identified and prioritized to evaluate the chemical composition and associated biological activity of surface waters from environmental settings that are vulnerable to a range of environmental contaminants from a spectrum of point and nonpoint contamination sources (fig. 1; table 1). Sites were selected from past sites at which USGS has characterized environmental contaminants and has knowledge of both the types of chemicals that affect the site as well as the types of upstream contamination sources. Four sites were selected and sampled to represent minimally affected environmental settings based on the diversity and sensitivity of fish species present. These sites were identified with input from USGS biologists and local stakeholders and are referred to as “reference” sites in table 1. Prior results and experience with all the sites allowed sampling times and conditions to be optimized to represent periods when the broadest chemical mixtures were expected to be present.

### Sampling Methods

Water-quality samples were collected by USGS staff at 38 sites throughout the United States and Puerto Rico, using standard USGS methods (U.S. Geological Survey, variously dated). Each station was sampled one time during the November 2012–June 2014 period. Water samples were collected at a single location in the center of flow, either into a Teflon churn splitter or directly into bottles ready for laboratory analysis. Samples that were collected into the Teflon churn were then processed onsite into appropriate bottles for analyses (table 2), chilled to below 4 degrees Celsius (°C), and prepared for shipment the same day, with appropriate paperwork and (or) chain of custody. Samples were shipped overnight to participating laboratories. When possible, field measurements (pH, specific conductance, dissolved oxygen, and water and air temperature) and instantaneous discharge measurements were made. Discharge measurements not made during sampling were determined from rating curves, when available. To ensure consistency in sampling methods and bottle filling order, sampling instructions (for each laboratory) were sent to each team along with all of the necessary supplies for sample collection, processes, preservation, and shipment (bottles, sampling processes and preservation supplies, pre-printed laboratory request and chain-of-custody forms, shipping labels and cooler packing diagrams). An example of the sampling instructions is provided in appendix 1.





**Figure 1.** Site locations and 2011 National Land Cover for sites where samples were collected for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014 (from Multi-Resolution Land Characteristics Consortium, 2011).

## Analytical Methods

The following section describes analytical methods performed at the USGS National Water Quality Laboratory (NWQL) in Denver, Colorado; the USGS Organic Geochemistry Research Laboratory (OGRL) in Lawrence, Kansas; the USGS Pesticide Fate Research Group (PFRG) laboratory in Sacramento, California; the USGS National Research Program (NRP), Central Branch, in Boulder, Colorado; the USGS Leetown Science Center, National Fish Health Research Laboratory (NFHRL) in Kearneysville, West Virginia; and the EPA Research and Development laboratories located in the John C. Stennis Space Center, Mississippi, the Research Triangle Park (RTP), Durham, North Carolina, Duluth, Minnesota, and Cincinnati, Ohio. Samples were shipped unfiltered to the laboratories. Filtering required for analysis was performed in the laboratory using a Whatman

0.7-micron glass-fiber filter (Florham Park, New Jersey) unless otherwise noted. Compound information and reporting limits are described in tables 3–5.

## National Water Quality Laboratory

The USGS NWQL, in Denver, Colo., analyzed 38 surface-water samples using the following methods: volatile organic compounds, steroid hormones and related compounds, wastewater indicator compounds, human-use pharmaceuticals and pharmaceutical metabolites and polar organic compounds, pesticides and pesticide metabolites, halogenated organic compounds, and selected trace elements, cations, anions, nutrients, and physical properties. Method descriptions including digestion (where applicable), analytical instrumentation, reporting limits, and units are described below.

**Table 1.** Station information and dates samples were collected for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.—Continued

[NWIS, National Water Information System; DD, decimal degrees; mi<sup>2</sup>, square mile; WWTP, wastewater-treatment plant. Latitude and longitude are referenced to the North American Datum of 1983 (NAD83)]

NWIS site number	Station name	Site code	Date sampled (mm/dd/yyyy)	Site type	Latitude (DD)	Longitude (DD)	Drainage area (mi <sup>2</sup> )
02350524	Abrams Creek near Oakfield, Georgia	Abrams Ck, GA	7/23/2013	Routine	31.7188	–83.9885	80.2
06893500	Blue River at Kansas City, Missouri	Blue River, MO	2/11/2014	Base/Low Flow	38.9570	–94.5589	188
252414080333200	C-111 Canal 100 feet above S-177 near Homestead, Florida	C-111 Canal, FL	4/9/2013	Routine	25.4043	–80.5587	51.0
05537000	Chicago Sanitary and Ship Canal at Lockport, Illinois	Chicago SSC, IL	2/11/2014	Base/Low Flow	41.5703	–88.0794	740
07159735	Chisholm Creek near Edmond, Oklahoma	Chisholm Ck, OK	11/28/2012	Base/Low Flow	35.7256	–97.5273	38.8
452340122251000	Deep Creek at Highway 224, Oregon	Deep Ck, OR	1/13/2014	Event	45.3943	–122.4206	49.0
01472705	East Branch Perkiomen Creek near Derstines, Pennsylvania	EB Perkio-men Ck, PA	1/14/2014	Routine	40.3540	–75.3133	29.4
02160326	Enoree River at Pelham, South Carolina	Enoree R, SC	5/19/2014	Base/Low Flow	34.8565	–82.2262	84.2
04234000	Fall Creek near Ithaca, New York	Fall Ck, NY	6/9/2014	Routine	42.4533	–76.4728	126
12211900	Fishtrap Creek at 1B near Lyndon, Washington	Fishtrap Ck, WA	12/10/2013	Base/Low Flow	49.0026	–122.4074	16.5
05485605	Fourmile Creek below Ankeny WWTP outfall, Iowa	Fourmile Ck, IA	11/19/2012	Base/Low Flow	41.7174	–93.5701	60.9
01630540	Hawksbill Creek at Route 648 at Springfield, Virginia	Hawksbill Ck, VA	4/15/2014	Routine	38.7082	–78.4564	68.2
02281200	Hillsboro Canal at S-6 near Shawano, Florida	Hillsboro Canal, FL	3/12/2013	Routine	26.4720	–80.4459	311
382054085332600	Hite Creek at Sleepy Hollow Road near Prospect, Kentucky	Hite Ck, KY	5/20/2014	Base/Low Flow	38.3483	–85.5572	5.55
01391100	Hohokus Brook at mouth at Paramus, New Jersey	Hohokus Br, NJ	11/28/2012	Base/Low Flow	40.9553	–74.1006	20.2
01452040	Jordan Creek at mouth at Allentown, Pennsylvania	Jordan CK, PA	6/17/2013	Routine	40.6018	–75.4621	82.3
03220000	Mill Creek near Bellepoint, Ohio	Mill Ck, OH	6/4/2013	Routine	40.2484	–83.1738	178
10255550	New River near Westmorland, California	New R, CA	3/5/2013	Routine	33.1048	–115.6644	1,471
06770195	North Dry Creek 2.0 miles southwest of Bridge South of Kearny, Nebraska	N. Dry Ck, NE	6/10/2013	Routine	40.6411	–99.1159	77.7
07060710	North Sylamore Creek near Fifty Six, Arkansas	N. Sylamore Ck, AR	1/7/2014	Reference (Base/Low Flow)	35.9917	–92.2139	58.1

**Table 1.** Station information and dates samples were collected for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.—Continued

[NWIS, National Water Information System; DD, decimal degrees; mi<sup>2</sup>, square mile; WWTP, wastewater-treatment plant. Latitude and longitude are referenced to the North American Datum of 1983 (NAD83)]

NWIS site number	Station name	Site code	Date sampled (mm/dd/yyyy)	Site type	Latitude (DD)	Longitude (DD)	Drainage area (mi <sup>2</sup> )
01409480	Penn Swamp Branch near Batsto, New Jersey	Penn Swamp Br, NJ	12/10/2012	Reference (Base/Low Flow)	39.6842	−74.6503	4.80
50055410	Rio Bairoa at mouth, Puerto Rico	Rio Bairoa, PR	3/19/2013	Base/Low Flow	18.2611	−66.0188	7.70
181847065391301	Rio Fajardo below Fajardo WWTP Outfall, Puerto Rico	Rio Fajardo, PR	3/25/2013	Base/Low Flow	18.3130	−65.6536	20.4
324305097101900	Rush Creek at Highway 303 near Arlington, Texas	Rush Ck, TX	5/13/2013	Routine	32.7179	−97.1720	28.9
13210360	Sand Run Gulch at Highway 95 crossing near Parma, Idaho	Sand Run Gulch, ID	6/18/2013	Routine	43.7641	−116.9121	79.5
11074000	Santa Ana River below Prado Dam, California	Santa Ana R, CA	3/25/2014	Event	33.8833	−117.6453	2,258
02335870	Sope Creek near Marietta, Georgia	Sope Ck, GA	4/16/2013	Routine	33.9539	−84.4433	30.8
05451210	South Fork Iowa River near New Providence, Iowa	SF Iowa R, IA	5/20/2013	Event	42.3150	−93.1524	266
05373005	South Fork Zumbro River below WWTP near Rochester Minnesota	SF Zumbro R, MN	12/5/2012	Base/Low Flow	44.0739	−92.4677	312
394847104570700	South Platte River near Commerce, Colorado	S. Platte R, CO	12/11/2012	Base/Low Flow	39.8130	−104.9525	4,082
05340041	Sunrise River tributary near Lindstrom, Minnesota	Sunrise Trib, MN	12/4/2012	Base/Low Flow	45.4076	−92.8806	6.60
05014300	Swiftcurrent Creek above Swiftcurrent Lake at Many Glacier, Montana	Swiftcurrent Ck, MT	8/28/2013	Reference (Base/Low Flow)	48.7954	−113.6806	15.8
385531121532001	Sycamore Slough a County Line Road near Dunnigan, California	Sycamore Slough, CA	6/18/2013	Routine	38.9253	−121.8889	64.3
364554121453401	Tembladero Slough at Castroville, California	Tembladero Slough, CA	3/3/2014	Event	36.7650	−121.7594	154
08057410	Trinity River below Dallas, Texas	Trinity R, TX	12/4/2012	Base/Low Flow	32.7076	−96.7358	6,265
01421640	West Branch Delaware River at South Kortright, New York	WB Delaware R, NY	11/19/2012	Base/Low Flow	42.3436	−74.7200	48.2
09505800	West Clear Creek near Camp Verde, Arizona	W. Clear Ck, AZ	1/15/2014	Reference (Base/Low Flow)	34.5386	−111.6940	241
14201300	Zollner Creek near Mt. Angel, Oregon	Zollner Ck, OR	5/19/2014	Event	45.1004	−122.8218	15.0

**Table 2.** Analyzing laboratory and method, bottle type, and treatment required for sampling conducted nationwide as part of the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.

[USGS, U.S. Geological Survey; NWQL, National Water Quality Laboratory, Denver, Colorado; PFRG, Pesticide Fate Research Group, Sacramento, California; OGRL, Organic Geochemistry Research Laboratory, Lawrence, Kansas; NRP, National Research Program, Boulder, Colorado; NFHRL, National Fish Health Research Laboratory, Kearneysville, West Virginia; EPA, U.S. Environmental Protection Agency; ECB, Environmental Chemistry Branch; TOC, total organic carbon; DBP, disinfectant by-products; FHM, fathead minnow (*Pimephales promelas*); APEO, alkylphenol ethoxylates; mL, milliliter; L, liter; HDPE, high density polyethylene; N, normal; H<sub>2</sub>SO<sub>4</sub>, sulfuric acid; HNO<sub>3</sub>, nitric acid; HCl, hydrochloric acid; H<sub>2</sub>O, water]

Analyzing laboratory	Laboratory method, schedule (S), or laboratory code (LC)	Bottle type	Preservation required
USGS NWQL	S2440	1- 40 mL, amber, glass	Lab filter, chill
	S2521	1- 125 mL, clear, polyethylene	Acidify (1 mL of 4.5N H <sub>2</sub> SO <sub>4</sub> ), chill
	S2521 (TOC)	1- 125 mL, baked, amber, glass	Chill
	S2524	1- 250 mL, acid rinsed, clear, polyethylene	Acidify (2 mL of 7.5N Ultrex HNO <sub>3</sub> )
	S2524	1- 250 mL, clear, polyethylene	Lab filter
	S2524	1- 250 mL, clear, polyethylene	None
	S2524	1- 500 mL, clear polyethylene	None, chill
	S4434, LC8170	1- 500 mL, clear polyethylene	Chill
	LC8170	2- 500 mL, clear polyethylene	Chill
	S4433, LC8244, LC2753	1- 1 L, baked, amber, glass	Chill
	LC8099	1- 1 L, baked, amber, glass	Chill
	S2033, S2060	2- 1 L, baked, amber, glass	Lab filter, chill
	S4440, S4437, LC8252	6- 40 mL, amber, vial	Acidify (1:1 HCl:H <sub>2</sub> O solution), chill
USGS PFRG	Gas chromatography pesticides, Liquid chromatography pesticides, DBP, forensics	3- 1 L, baked, amber, glass	Chill
USGS OGRL	Liquid chromatography pesticides, Antibiotics, Hormones	2- 1 L, baked, amber, glass	Chill
USGS NRP	Surfactants	1- 1 L, baked, amber, glass	Chill
USGS NFHRL	BioAssay (Estrogen Receptor Activation)	1- 1 L, baked, amber, glass	Chill
EPA, Ohio	Endocrine disrupting compounds	1- 600 mL, glass	Copper sulfate, chill
	Perfluorocarbons	1- 1 L, HDPE	Chill
	FHM larva exposure	3- 1 L, baked, amber, glass	Chill
	Alkylphenol	1- 250 mL, amber, glass	1 mL of 2N HCl, chill
	Bisphenol A	1- 250 mL, amber, glass	1 mL of 2N HCl, chill
	Steroid hormones	1- 1 L, baked, amber, glass	Chill
	APEOs	1- 125 mL, amber, glass	3 mL of 35 percent formaldehyde, chill
	Perchlorate	1- 40 mL, amber, glass	Chill
EPA, Minnesota	BioAssay	4- 1 L, baked, amber, glass	Chill
EPA, North Carolina	BioAssay	8- 1 L, baked, amber, glass	Chill
EPA ECB	Perfluorocarbons	2- 1 L, Nalgene <sup>1</sup> , wide-mouth	Chill

<sup>1</sup>Nalge Nunc International Registered Trademark.

**Table 4.** Aminocarboxylic acid complexing agents and non-ionic surfactant degradate compound information (Barber and others, 2000) and spiked concentrations of surrogate and internal standards analyzed using gas chromatography with mass spectrometry (GC/MS) at the National Research Program laboratory in Boulder, Colorado. Also shown are the molecular ions [ $M^+$ ] and qualifier ions ( $Q_1$ ,  $Q_2$ ) used in the selected ion monitoring (SIM) GC/MS analysis.

[CAS, Chemical Abstracts Service Registry; MDL, method detection limit;  $\mu\text{g/L}$ , microgram per liter; MW, molecular weight; value in parenthesis is the molecular weight of the propyl ester derivative;  $M^+$ , molecular ion;  $Q_1$ ,  $Q_2$ , qualifier ions; m/z, mass to charge ratio; SS, surrogate standard; IS, internal standard; —, not applicable]

Compound	CAS number	MDL ( $\mu\text{g/L}$ )	Units	MW (grams)	[ $M^+$ ] (m/z)	$Q_1$ (m/z)	$Q_2$ (m/z)
Nitrilotriacetic acid (NTA)	139-13-9	0.012	$\mu\text{g/L}$	191.1 (317.4)	317.4	230.3	144.1
Ethylenediaminetetraacetic acid (EDTA)	60-00-4	0.018	$\mu\text{g/L}$	292.2 (460.5)	460.6	230.3	144.1
Diethyltriaminepentaacetic acid (DTPA)	67-43-6	0.018	$\mu\text{g/L}$	393.4 (603.8)	516.3	373.5	244.3
4-nonylphenolmonoethoxycarboxylic acid (NP1EC)	3115-49-9	0.071	$\mu\text{g/L}$	278.4 (320.5)	320.5	249.3	235.3
4-nonylphenoldiethoxycarboxylic acid (NP2EC)	106807-78-7	0.069	$\mu\text{g/L}$	322.4 (364.5)	364.5	279.3	103.1
$d_{12}$ -ethylenediaminetetraacetic acid, SS, $d_{12}$ -EDTA	203806-08-0	14.150 <sup>a</sup>	—	304.2 (472.5)	472.6	236.3	150.2
4-n-NP2EC, SS, 4-n-NP2EC	104-40-5	6.036 <sup>a</sup>	—	322.4 (364.5)	364.5	145	103.1
1-phenylnonane, IS	1081-77-2	1.263 <sup>a</sup>	—	204.4 (—)	204.3	133.1	92.1

<sup>a</sup>Spiked concentration.

**Table 5.** Compound information for analyses of perfluorinated compounds (Boone and others, 2014) performed by the U.S. Environmental Protection Agency, Research and Development laboratories in the John C. Stennis Space Center in Mississippi, November 2012–August 2013.

[Values are in nanograms per liter. NWIS, National Water Information System; CAS, Chemical Abstracts Service Registry; LCMRL, lowest concentration minimal reporting level; DL, detection level; CR, Critical Level]

Compound	NWIS parameter code	CAS number	LCMRL	DL	CR
Perfluorobutanoic acid (PFBA)	67964	375-22-4	0.290	0.150	0.180
Perfluoropentanoic acid (PFPeA)	67965	2706-90-3	0.037	0.022	0.012
Perfluorohexanoic acid (PFHxA)	67966	307-24-4	0.035	0.011	0.014
Perfluoroheptanoic acid (PFHpA)	67967	375-85-9	0.038	0.025	0.016
Perfluorooctanoic acid (PFOA)	65227	335-67-1	0.600	0.042	0.270
Perfluorononanoic acid (PFNA)	67968	375-95-1	0.110	0.047	0.044
Perfluorodecanoic acid (PFDA)	67969	335-76-2	0.096	0.023	0.021
Perfluoroundecanoic acid PFUnDA)	52641	2058-94-8	0.058	0.025	0.022
Perfluorododecanoic acid (PFDoDA)	67970	307-55-1	0.032	0.015	0.011
Perfluorotridecanoic acid (PFTrDA)	67971	72629-94-8	0.035	0.008	0.005
Perfluorotetradecanoic acid (PFTeDA)	67972	376-06-7	0.170	0.031	0.032
Perfluorohexadecanoic acid (PFHxDA)	52642	67905-19-5	0.530	0.200	0.043
Perfluorooctadecanoic acid (PFOcDA)	52643	16517-11-6	0.180	0.070	0.037
Perfluorobutane sulfonate (PFBS)	67973	375-73-5	0.052	0.025	0.021
Perfluorohexane sulfonate (PFHxS)	67974	355-46-4	0.034	0.016	0.009
Perfluorooctanesulfonate (PFOS)	52644	1763-23-1	0.140	0.032	0.041
Perfluorodecane sulfonate (PFDS)	67975	335-77-3	0.060	0.023	0.012



## Ambient Purgeable Volatile Organic Compounds

Samples were analyzed for 100 ambient purgeable volatile organic compounds (VOCs) by using gas chromatography (table 3). In brief, helium was used to remove VOCs from water samples at ambient temperature. The purged VOCs were trapped in a tube, desorbed, and prepared for analysis by gas chromatograph/mass spectrometry (GC/MS) (Rose and others, 2016). Results are reported in micrograms per liter ( $\mu\text{g/L}$ ).

## Heat Purgeable Volatile Organic Compounds

Samples were analyzed for 38 (including surrogates) VOCs using the heat purgeable method outlined in Rose and others (2016). Similar to the ambient purgeable method, VOCs were removed from the sample and prepared for analysis. For the heat purgeable method, however, results were determined using electron impact ionization/mass spectrometry, with full scan/selected ion monitoring. Results are reported in micrograms per liter (table 3).

## Steroid Hormones and Related Compounds

Unfiltered samples were analyzed for 20 hormone and related compounds, including natural and synthetic estrogen and androgen compounds, natural and synthetic progestin compounds, sterols and Bisphenol A (Foreman and others, 2012). Concentrations were determined using solid phase extraction (SPE) and gas chromatograph/tandem mass spectrometry (GC/MS–MS) (Foreman and others, 2012) (table 3). Results are reported in nanograms per liter (ng/L).

## Wastewater Indicator Compounds and Selected Pharmaceuticals

Unfiltered samples were analyzed for 69 wastewater compounds, including alkylphenol ethoxylate nonionic surfactants, food additives, fragrances, antioxidants, flame retardants, plasticizers, industrial solvents, disinfectants, fecal sterols, polycyclic aromatic hydrocarbons, and high-use domestic pesticides (Zaugg and others, 2006). Samples were prepared for extraction with the addition of 60 grams (g) of sodium chloride (NaCl) to increase ionic strength and extracted using a continuous liquid-liquid extractors and methylene chloride solvent and frozen to less than 10 °C until analyzed. Extracts were analyzed using capillary-column GC/MS (table 3). Results are reported in micrograms per liter.

In addition to the wastewater compounds listed above, a second analysis was performed on unfiltered samples to identify 48 pharmaceutical and other related organic compounds (Zaugg and others, 2014). Samples were extracted for this analysis using a continuous liquid-liquid extraction with dichloromethane. Concentrations were determined using capillary-column GC/MS (Zaugg and others, 2014) (table 3). Results are reported in micrograms per liter.

## Pesticides and Pesticide Metabolites

Two pesticide methods were analyzed on laboratory filtered samples at the USGS NWQL. The method described in Furlong and others (2001) was used to analyze for 60 polar pesticides and pesticide metabolites. Samples were extracted using a 0.5-gram graphitized carbon-based SPE cartridge. After two elution processes (first with methanol, followed by methylene chloride and methanol, acidified with trifluoroacetic acid anhydride), the resulting fractions were dried and combined for analysis. Concentrations were determined using high-performance liquid chromatography (HPLC), with electrospray ionization-mass spectrometry (Furlong and others, 2001) (table 3). Results are reported in micrograms per liter.

The methods described in Zaugg and others (1995), Lindley and others (1996), Madsen and others (2002), and Sandstrom and others (2001) were used to analyze a total of 83 low-level pesticide and pesticide metabolites. Forty-seven of these compounds are described in Zaugg and others (1995). In brief, samples were extracted using SPE containing porous silica bonded with an octadecyl (C-18) phase. After elution with hexane-isopropanol, and a final evaporation, the extracts were analyzed by capillary-column GC/MS operating in selected ion monitoring (SIM) mode (Zaugg and others, 1995). The method for acetochlor is described in Lindley and others (1996) and uses SPE, followed by an elution with hexane-isopropyl alcohol and further concentration of the extract. Concentrations are determined by capillary-column GC/MS. Fipronil and fipronil degradates were extracted using SPE and analyzed using capillary-column GC/MS using positive-ion electron-impact SIM. This method is described in Madsen and others (2002). The remaining compounds are described in Sandstrom and others (2001). Samples were extracted using SPE containing porous silica bonded with octadecyl (C-18) phase and dried, eluted using ethyl acetate, and analyzed using capillary-column GC/MS (table 3). Results are reported in nanograms per liter.

## Human-Use Pharmaceuticals, Pharmaceutical Metabolites, and Selected Polar Organic Compounds

Filtered samples were analyzed for 110 human-use pharmaceutical, pharmaceutical metabolites, and selected polar organic compounds. Samples were analyzed using direct injection HPLC/MS–MS, operated in positive ion mode (Furlong and others, 2014) (table 3). Results are reported in nanograms per liter.

## Selected Halogenated Organic Compounds

Unfiltered water samples, ranging from 403 to 551-milliliter (mL) volume, were fortified with 1 nanogram (ng) of surrogate compounds 4,4'-dibromooctafluorobiphenyl and PCB-202- $^{13}\text{C}_{12}$ , and 10 ng of 4,4'-DDT- $d_8$ . The samples were extracted by passing the water through Oasis® HLB SPE cartridges (Waters Corp., Milford, Massachusetts) at about

10 mL per minute flow rate by application of minimal head pressure using nitrogen gas. Each sample bottle was rinsed to remove residual sample water and settled sediment material with 35 mL of potassium phosphate monobasic/dibasic buffer solution (pH 7.0). This rinse was passed through the HLB cartridge. The uncapped sample bottle was then inverted to drain out the remaining water.

Residual water in the HLB cartridge sorbent was displaced with nitrogen (4 minutes at 13.8 kilopascals). Method compounds were then eluted from the HLB cartridge and passed through a 2-g Florisil® SPE cartridge (Biotage, LLC, Charlotte, N.C.) overlain with sodium sulfate by using 5 mL of hexane, followed by addition of a 10-mL rinse of the sample bottle with a mixture of dichloromethane (DCM) and diethyl ether (DEE) (80:20, volume to volume [v:v]), and finally by addition of another 30 mL of this DCM/DEE eluent directly to the HLB/Florisil solvent reservoir. The resultant collected eluent (extract) was concentrated using nitrogen to a final volume of 1 mL and transferred to a vial containing 2 ng of injection internal standard compounds polychlorinated biphenyls, PCB-141-<sup>13</sup>C<sub>12</sub> and PCB-209-<sup>13</sup>C<sub>12</sub>, in preparation for analysis.

Method compounds were determined by injection of 10 microliters (µL) of sample extract using a programmed-temperature vaporization (PTV) injector (from 50 to 320 °C) into a model 6890 gas chromatograph with model 5975 mass spectrometer operated in the electron-capture negative ionization mode (GC/ECNIMS; Agilent Technologies, Santa Clara, Calif.) by using ammonia reagent gas. Compounds were separated using a 30-meter by 0.25-mL internal diameter DB-35MS Ultra Inert capillary GC column having a 0.25-micrometer (µm) stationary phase film thickness (Agilent Technologies). Mass spectral data were obtained using simultaneous SIM and full-scan acquisition modes. Detection capability for analytes at low sample concentration or that had poor responses was improved by use of the SIM mode and by scanning for at least three unique analyte ions multiple times in SIM mode. Analyte identification was established by comparing both chromatographic retention times and the ratios of the quantification ion to the secondary qualification ions in environmental samples to the retention times and ion ratios obtained for the calibration standards. For those samples with high analyte concentrations, the SIM results were further confirmed by use of the full-scan spectral results. Full-scan results also helped deduce whether interferences might be present. The PTV GC inlet was used so that both thermally labile compounds and high molecular weight compounds could be analyzed in the same GC analysis. Method analytes were quantified using multiconcentration (typically nine levels) calibration standards with quantification relative to an injection internal standard compound. Response performance throughout the analyses was monitored by use of continuing calibration standards.

Analytes determined include 62 chemicals grouped into the following classes: selected polychlorinated biphenyl congeners, halogenated flame retardants, legacy and currently

used pesticides and selected pesticide degradates, and several industrial or personal care products and selected degradates. Detection levels were estimated using the spike-based method detection limit procedure of the EPA (2014) or by using laboratory blank-sample data for four blank-limited compounds (triclosan, PBDE-47, PBDE-99, and PBDE-100). Reporting levels were set at two or more times the detection level and range from 0.2 to 10 ng/L, with concentrations below the reporting and detection levels reported for identified analytes (Childress and others, 1999) (table 3).

Quality control (QC) samples prepared along with a set of typically 10 environmental samples included one laboratory blank and one laboratory spike sample prepared using reagent water matrix. Data for these QC samples were used to censor or qualify reported environmental sample results, if needed. Recoveries of surrogate compounds fortified into each sample provided an indication of sample preparation performance and also were used to qualify results, as needed.

### Selected Trace Elements, Cations, Anions, and Physical Properties

Unfiltered and filtered samples were analyzed for trace elements, cations, anions, and physical properties following methods described in Fishman and Friedman (1989), Hoffman and others (1996), Garbarino and Struzeski (1998), and Garbarino and others (2006) (table 3). Results of trace elements are reported in micrograms per liter. Results of cations, anions, acid neutralizing capacity (ANC), and total dissolved solids are reported in milligrams per liter (mg/L). Specific conductance is reported in microsiemens per centimeter (µS/cm) at 25 °C, pH in standard units, and turbidity in nephelometric turbidity units (NTU).

### Selected Nutrients and Total Organic Carbon

Unfiltered samples were analyzed for nitrogen, ammonia, phosphorus, and total organic carbon. Ammonia nitrogen concentrations were determined using EPA Method 350.1 using semiautomated colorimetry (EPA, 1993) (table 3). Concentrations of ammonium plus organic nitrogen were analyzed using Kjeldahl digestion, including an automated photometric finish and gas diffusion clean up (Patton and Truitt, 2000). Total nitrogen concentrations were determined using methods described in Patton and Kryskalla (2003). Total phosphorus was determined using EPA Method 365.1, using semiautomated colorimetry (O'Dell, 1993). Total organic carbon concentrations were determined using EPA method 5310b (Clesceri and others, 1998). All results were reported in milligrams per liter.

## Organic Geochemistry Research Laboratory

The USGS, Kansas Water Science Center, Organic Geochemistry Research Laboratory in Lawrence, Kansas, analyzed 38 filtered, surface-water samples for the following

suite of compounds: acetamide herbicides and degradation products, glyphosate, glufosinate, and aminomethylphosphonic acid, steroid hormones and phytoestrogens, triazine and phenylurea herbicides, and selected antibiotics.

### Acetamide Herbicides and Degradation Products

Samples for determination of acetamide herbicides and acetamide degradates were analyzed using an online SPE LC/MS with electrospray ionizations (ESI) and positive- and negative-ion switching modified from the USGS-approved method of Lee and Strahan (2003). Reporting limits, in micrograms per liter, and compound information are presented in table 3.

### Glyphosate, Glufosinate, and Aminomethylphosphonic Acid

Filtered samples were analyzed for glyphosate, glufosinate, and aminomethylphosphonic acid. Samples were extracted onto SPE cartridges, and the SPE cartridges were rinsed with 500  $\mu$ L of deionized water. All sample extracts were analyzed by LC/MS–MS with ESI in negative-ion mode using multiple reaction monitoring (MRM) (Meyer and others, 2009). Reporting limits, in micrograms per liter, and compound information are presented in table 3.

### Steroid Hormones and Phytoestrogens

Filtered samples were analyzed for 26 steroid hormones and phytoestrogens. The method is described in Yost and others (2014) and briefly described here. Samples were processed using SPE using 200 milligrams (mg) HLB cartridges (Waters Corp., Milford, Mass.), eluted with methanol and analyzed using ultra-pure LC/MS–MS. Reporting limits, in nanograms per liter, and compound information are presented in table 3.

### Triazine and Phenylurea Herbicides

Filtered samples were analyzed for 7 triazine and phenylurea herbicides and 12 degradation products. The method description can be found in Lee and others (2002). In brief, upon receipt at the laboratory, filtered samples were spiked with surrogate compounds, preconditioned in a graphitized carbon column, eluted (with methylene chloride, methanol, and ammonium hydroxide), spiked with internal standards, evaporated, and reconstituted. Detection and identification of sample components were determined using HPLC with a diode array detector and a mass spectrometry (MS) detector in SIM mode. Concentrations were determined after the concentrated samples were mixed with acetic acid and analyzed by LC/MS. Reporting limits, in micrograms per liter, and compound information are presented in table 3.

### Selected Antibiotics

Filtered samples were analyzed for antibiotic compounds, including chloramphenicol, lincomycin, ormetoprim, trimethoprim, five macrolides, six sulfonamide, six quinoline, and four tetracycline antibiotics, six antibiotic degradation products, and the pharmaceuticals carbamezpine and ibuprofen. Samples were analyzed using a method modified from Meyer and others (2009). Samples were extracted using SPE and concentrations determined using ultra-pressure LC/MS–MS with ESI using MRM. Except for chloramphenicol and ibuprofen, samples were analyzed in positive-ion mode (USGS Organic Geochemistry Research Laboratory, written commun., 2014). Reporting limits, in micrograms per liter, and compound information area presented in table 3.

### Pesticide Fate Research Laboratory

The USGS California Water Science Center, Pesticide Fate Research Group in Sacramento, Calif., analyzed 38 filtered, surface-water samples for the following suite of compounds: pesticide and pesticide degradates, herbicides, insecticides, and disinfection by-products.

### Pesticide and Pesticide Degradates

Filtered samples were analyzed for 60 pesticides and pesticide degradates. Samples were spiked as required, extracted using SPE, dried, and then eluted with ethyl acetate. Finally, the sample was reduced to 200  $\mu$ L, and an internal standard was added. Concentrations were determined using GC/MS (Hladik and others, 2008). Method detection levels, in nanograms per liter, and compound information are presented in table 3.

### Diuron and Diuron Degradates and Neonicotinoid Insecticides

Filtered samples were analyzed for the herbicide diuron and three degradates and six neonicotinoid insecticides. Samples were spiked with surrogate standards monuron (Chem Service, West Chester, Pennsylvania) and (or) imidacloprid- $d_4$  (Cambridge Isotope Laboratories, Andover, Massachusetts). The samples were then extracted using SPE, dried, and eluted with DCM and acetone. Finally, the elution was dried to less than 0.2 mL, and an internal standard ( $^{13}\text{C}_3$ -caffeine) was added. Extracted samples were analyzed using HPLC/MS–MS (Hladik and Calhoun, 2012). Method detection levels, in nanograms per liter, and compound information are presented in table 3.

### Disinfection By-Products

Filtered samples were analyzed for 29 disinfection by-products. Samples were spiked with surrogate standards



1-bromo-4-fluorobenzene and *d*<sub>4</sub>-1,2-dichlorobenzene, extracted using SPE within 48 hours. Extracts were then dried and eluted with 10 mL of methyl tert-butyl ether (MTBE) and reduced to 400 µL. Finally, 1-chlorooctane internal standard was added. Extracts were analyzed using GC/MS (Hladik and others, 2014). Method detection levels, in nanograms per liter, and compound information are reported in table 3.

## National Research Program

Five acidic organic compounds were measured in the formalin preserved (1 percent (v:v) added when samples were received) water samples, including the aminocarboxylic acid metal complexing agents nitrilotriacetic acid (NTA), ethylenediaminetetraacetic acid (EDTA), and diethylenetriaminepentaacetic acid (DTPA), and the nonionic surfactant degradation products 4-nonylphenolmonothoxycarboxylate (NP1EC) and 4-nonylphenoldiethoxycarboxylate (NP2EC), using methods described in Barber and others (2000) and Barber and others (2003). In brief, the method involves evaporating 100 mL of sample to dryness at 90 °C, adding 2 mL formic acid:distilled water (1:1 v:v), and using vacuum evaporation to bring the sample to dryness again. Acetyl chloride:propanol (1.5 mL, 1:10 v:v) is added to form propyl esters from the carboxylic acid functional groups, the samples are heated to 90 °C for 1 hour, and the propyl esters are extracted into chloroform. The extracts were evaporated to dryness under nitrogen, redissolved in 200 µL of toluene, and analyzed by GC/MS in full-scan and SIM modes. Table 4 lists the compounds determined, ions monitored, and method detection limits of the concentrated samples. Compound identification was based on matching of retention times ( $\pm 0.05$  minute) and ion ratios determined from analysis of standards, and concentrations were calculated based on SIM data using diagnostic ions for each compound. Multipoint external calibration-curve and internal-standard procedures were used for calculating concentrations.

## USGS National Fish Health Research Laboratory

Samples were collected and sent to the USGS NFHRL in Kearneysville, West Virginia, to be analyzed for Bioluminescent Yeast Estrogen Screen and Transgenic zebrafish embryo estrogen bioassays. Samples from five sites (East Branch Perkiomen Creek, Pennsylvania; Mill Creek, Ohio; South Fork Iowa River, Iowa; Sope Creek, Georgia; and West Clear Creek, Arizona) were ruined either in shipment or during analysis, and results are not reported. The methods used are described in Ciparis and others (2012) and are briefly described herein.

### Bioluminescent Yeast Estrogen Screen

Water samples were collected in chemically clean, 1-liter (L) amber Boston round bottles rated for semivolatiles. Samples were stored immediately on wet ice and shipped in

coolers overnight to the NFHRL in Kearneysville, W.V. Upon arrival at the laboratory, the water was pH adjusted to pH 3 via dropwise addition of 6N hydrochloric acid (if not complete prior to shipping) within 48 hours of collection. Samples were then filtered through a 0.7-µm glass-fiber filter (Whatman # 1825-047) and stored at 4 °C prior to extraction.

Oasis HLB glass cartridges (Waters # 186000683) were sequentially pre-conditioned with 5 mL each of ethyl acetate, 50:50 methanol:dichloromethane (DCM), methanol, and HPLC-grade water. All solvents were HPLC grade. A total volume of 800 mL of filtered water was extracted. Elution solvents consisted of 6 mL methanol and 6 mL of 50:50 methanol:DCM. Eluates were reduced to dryness and solubilized in 1 mL of 100 percent methanol. Reconstituted samples were stored at -20 °C and protected from light until screened.

Assays were performed blind to the sample location. The bioluminescent yeast estrogen screen (BLYES) was used to quantitatively assess estrogenic activity relative to 17β-estradiol (Sanseverino and others, 2005). Strain BLYES was purchased from 490 BioTech. The BLYES assay was run according to Ciparis and others (2012). In short, 5 µL of sample extract was added to 95 µL of yeast minimal media (YMM leu<sup>-</sup>, ura<sup>-</sup>) in triplicate wells of a black, clear-bottom 96-well plate (Costar). To this, 100 µL of a 48-hour culture of strain BLYES at approximately 0.75 (OD<sub>600</sub>) was added to each well. A 17β-estradiol (E2; Sigma-Aldrich Co.) standard curve ranging from 4 to 500 pictograms per milliliter (pg/mL) was included on each plate. The final concentration of methanol in sample and standard wells after the addition of strain BLYES was 2.5 percent. A media control was included on all plates to establish background luminescence. Plates were covered and incubated in the dark at 30 °C for 4 hours. Luminescence was quantified using a SpectraMax M4 microplate reader (Molecular Devices), in luminescence mode (1,000 millisecond integration time), and relative estrogenicity of each sample was interpolated using a 4-parameter curve-fit within SoftMax Pro 6.2.2 (Molecular Devices). Relative estrogenicity per liter of river water was then calculated on the basis of sample concentration. The detection limit for this assay was 0.2 ng/L.

### Transgenic Zebrafish Embryo Estrogen Bioassay

Methanol was evaporated and sample extracts were solubilized in dimethyl sulfoxide (DMSO). The embryo bioassay was run similar to that described by Gorelick and others (2014). In short, zebrafish embryos (1 day post fertilization) were exposed to sample extract adjusted to actual field concentrations and incubated in 24-well plates for 48 hours. Two embryos were exposed per treatment. Exposure occurred under static water conditions with no water changes during exposure. Embryos were incubated at 28 °C in the dark. Tissue-specific fluorescence was evaluated using a Zeiss Axiovert 25 inverted microscope fitted with a mercury-arc lamp and fluorescein filter block set. Observations of tissue-specific fluorescence were qualitatively recorded as present or absent relative to a solvent control.

## U.S. Environmental Protection Agency Laboratories

Analyses of perfluorinated compounds and selected bioassays were performed at the EPA Research and Development laboratories located in John C. Stennis Space Center, Mississippi, the Research Triangle Park (RTP), Durham, North Carolina, Duluth, Minnesota, and in Cincinnati, Ohio. These methods are described briefly below for reference.

### Perfluorinated Compounds

Analysis of 17 perfluorinated compounds was performed at the EPA Environmental Chemistry Branch laboratory at the John C. Stennis Space Center, Miss., using the EPA method outlined in Boone and others (2014). In brief, samples were extracted using SPE, concentrated, and spiked with 50  $\mu$ L of carbon-13 labeled injection standard and analyzed using LC/MS–MS. Detection levels, in nanograms per liter, and compound information are presented in table 5.

### Bioassays

*T47D-KBluc bioassay for estrogenic activity:* A human breast cancer cell line that naturally expresses estrogen receptors and has been engineered to stably express a luciferase reporter gene under control of a triplet estrogen response element promoter was used to screen extracts for total estrogenic activity based on relative luminescence (Wilson and others, 2004; Conley and others, 2016). Responses for the extract were compared with those for an estradiol standard curve, and activity was expressed as 17 $\beta$ -estradiol equivalents.

*MDA-kb2 bioassay for androgenic activity:* A human breast cancer cell line that naturally expresses both androgen and glucocorticoid receptors and has been engineered to stably express a luciferase reporter gene under control of a mouse mammary tumor virus promoter, which is about one hundred-fold more responsive to potent androgens than glucocorticoids, was used to characterize total androgenic activity of sample extracts as described by Wilson and others (2002). Responses for the extracts were compared with those for a dihydrotestosterone standard curve, and activity was expressed as dihydrotestosterone equivalents.

*Transduced CV-1 cell bioassay for glucocorticoid activity:* CV-1 cells (African green monkey kidney fibroblast) were transduced with human glucocorticoid receptor and a mouse mammary tumor virus-luciferase promoter-reporter construct using adenoviral vectors to evaluate total glucocorticoid receptor-mediated activity of the sample extracts. Responses for the extracts were compared with those of a dexamethasone standard curve, and activity was expressed as dexamethasone equivalents. This specific assay has not been previously described but is methodologically similar to Shih and others (1991) and Hartig and others (2008).

*Attogene™ cis and trans-factorial assays for nuclear hormone receptor-mediated activities:* Extracts of surface-water samples were analyzed with HepG2 cells transfected with libraries of cis- and trans-regulated transcription factor reporter constructs that allow for simultaneous assay of approximately 80 different transcription factor activities (Martin and others, 2010). Prior to extraction, whole water samples were filtered using glass fiber filters (0.7  $\mu$ m; Whatman, Maidstone, U.K.). Extracts were then prepared by solid phase extraction using Oasis HLB glass cartridges (200 mg; Waters Corp., Milford, Mass.). The cartridges were eluted sequentially with methanol and 1:1 methanol:dichloromethane, blown to dryness under a gentle stream of nitrogen gas and reconstituted in DMSO (one thousandfold enrichment relative to ambient water concentrations). Following dilution into the assay medium, cells were exposed to a sample representing a tenfold enrichment relative to ambient water concentration (assuming 100 percent extraction efficiencies) and five additional threefold dilutions (six dilutions per sample). Data were analyzed using the ToxCast pipeline for high throughput screening data (Filer and others, 2016).

*In vitro steroidogenesis with Pimephales promelas ovary subsections:* Water samples were tested directly for their ability to inhibit sex steroid (17 $\beta$ -estradiol and testosterone) synthesis by fathead minnow (*Pimephales promelas*) ovary tissue using methods described by Villeneuve and others (2007). Water samples were stored frozen at  $-20^{\circ}\text{C}$  until used for the assay. For use in the assay, medium 199 was prepared directly with field-collected water, adjusted to pH 7.3, and then passed through a 0.22- $\mu$ m syringe filter. Multiple dilutions were prepared using medium 199 prepared with ultrapure water to generate a dilution series of 100 percent, 33 percent, and 11 percent of the original water concentration. Medium 199 prepared with ultrapure water was used as a control. After media was prepared, 500  $\mu$ L of each dilution or control medium was added to 12 replicate glass test tubes. Ovary tissue was then collected from 10 reproductively mature adult female fathead minnows obtained from an onsite culture facility at the EPA Mid-Continent Ecology Division, Duluth, Minn. Five subsamples of ovary tissue (10–20 mg each) were cut from each individual ovary. One ovary subsample from each fish was placed into one replicate tube of each dilution, and the fifth subsample was immediately flash frozen in liquid nitrogen. Samples were then incubated at  $25^{\circ}\text{C}$  for 12 hours. Two additional replicates of each dilution were incubated without tissue to serve as assay blanks. Following incubation, the medium was removed and stored frozen at  $-20^{\circ}\text{C}$  until extracted and analyzed for 17 $\beta$ -estradiol and testosterone by radioimmunoassay (see Villeneuve and others, 2007, for details). Blanks were used to test and correct for any cross reactivity between constituents of the field-collected water sample or media and the antibodies used in the radioimmunoassays. Factorial ANOVA considering both dilution and fish as independent variables was then used to test for significant effects on steroid production.



*In vitro metabolomics:* Methods similar to those reported by Teng and others (2013) were used to evaluate the effect of site waters on the metabolome of several cell types exposed to cell culture medium prepared from site water. Following exposure, endogenous metabolites were extracted from the exposed cells and measured using proton nuclear magnetic resonance spectroscopy and high resolution mass spectrometry. To facilitate intersite comparisons, the overall effect from exposure to water from each site was determined by integrating all statistically significant metabolite changes into a single value for each site. A partial least-squares regression approach developed by Davis and others (2016) was used to identify covariance between measured contaminants and abundances of endogenous metabolites to support identification of contaminants with the greatest biological relevance.

*In vivo transcriptomics:* For selected samples, larval fathead minnows were exposed to field-collected water samples in the laboratory. Total ribonucleic acid (RNA) was extracted from whole bodies, and next generation RNA sequencing was used to provide a global view of changes in the whole body transcriptome compared to larval fathead minnows from the same cohort exposed to control water.

*Mutagenicity:* Samples were screened for mutagenic and genotoxic substances, using the Ames assay using methods similar to those described by Warren and others (2015).

*Microtox assay:* Finally, the Microtox assay was used as a rapid-screening tool, using bioluminescent bacteria to detect toxicants in water samples (The Environmental Technology Verification Program, 2003).

## Quality Assurance/Quality Control

Field and laboratory quality-assurance and quality-control (QA/QC) procedures were performed for all analyses and laboratories and are described below.

### Field

Field quality assurance consisted of field blanks and matrix spikes (table 6). Field blanks were completed at 15 sites and matrix spikes were completed at 13 sites. Between two and eight field blanks were analyzed for each USGS schedule and three to four matrix spikes were analyzed for every USGS schedule except nutrients and trace elements. A summary of field blank detections and field spike surrogate information can be found at Romanok and others, 2017.

### Laboratory

Laboratory QA/QC was performed by each participating laboratory, and available results are summarized herein.

## National Water Quality Laboratory

QA/QC procedures for pesticide, pharmaceutical, hormone, halogenated organic, volatile organic and perfluorinated compounds analyzed at the NWQL, in Denver, Colo., are summarized below.

### Pesticides and Pesticide Metabolites

Three surrogate compounds were analyzed in water-quality samples for polar pesticides and metabolites, including barban. Percentage recoveries ranged from 18.8 to 113 percent; 2,4,5-Trichlorophenoxyacetic acid, percentage recoveries ranged from 43.0 to 102 percent; and  $C^{13}$ -caffeine, percentage recoveries ranged from 45.6 to 143 percent. Additionally, two surrogate compounds were analyzed in water-quality samples, including diazinon- $d_{10}$ , percentage recoveries ranged from 61.5 to 110 percent; and  $\alpha$ -HCH- $d_6$ , percentage recoveries ranged from 74.1 to 112 percent.

### Human-Use Pharmaceuticals, Pharmaceutical Metabolites, and Selected Polar Organic Compounds

Percentage recovery ranges for 17 pharmaceutical surrogate compounds analyzed in water-quality samples were thiabendazole- $d_4$ , 90 to 133 percent; albuterol- $d_9$ , 58.0 to 188 percent; diltiazem- $d_3$ , 92.5 to 109 percent; trimethoprim- $d_9$ , 33.9 to 205 percent; acetaminophen- $d_3$ , 77.7 to 111 percent; norfluoxetine- $d_6$ , 78.3 to 135 percent; methadone- $d_9$ , 80.3 to 135 percent; oxycodone- $d_3$ , 88.9 to 139 percent; hydrocodone- $d_3$ , 75.3 to 112 percent; temazepam- $d_5$ , 90.8 to 128 percent; caffeine-(trimethyl- $^{13}C_3$ ), 84.3 to 185 percent; sulfamethoxazole-(pheynyl- $^{13}C_6$ ), 32.2 to 134 percent; cotinine- $d_3$ , 80.3 to 113 percent; amphetamine- $d_6$ , 73.9 to 140 percent; codeine- $d_9$ , 74.1 to 160 percent; pseudoephedrine- $d_3$ , 67.3 to 145 percent; diphenhydramine- $d_3$ , 33.7 to 127 percent; and fluoxetine- $d_6$ , from 80.2 to 108 percent.

### Steroid Hormones and Related Compounds

Percentage recovery ranges for 13 hormone surrogate compounds analyzed in water-quality samples were bisphe-nol-A- $d_{16}$ , 0 to 134 percent; cis-androsterone- $d_3$ , 53.0 to 103 percent; progesterone-2,3,4- $^{13}C_3$ , 2.68 to 77.0 percent; cholesterol- $d_{27}$ , 46.2 to 85.1 percent; ethynylestradiol- $d_4$ , 65.4 to 110 percent; trans-diethylstilbestrol- $d_8$ , 30.1 to 82.2 percent; mestranol- $d_4$ , 66.4 to 112 percent; estriol-2,4,16,17- $d_4$ , 59.4 to 109 percent; 16-epiestriol- $d_2$ , 51.2 to 136 percent; medroxy-progesterone- $d_3$ , 12.5 to 129 percent; nandrolone- $d_3$ , 42.2 to 96.0 percent; 17-b-estradiol- $^{13}C_6$ , 63.2 to 95.1 percent; and estrone- $^{13}C_6$ , 55.0 to 101 percent.

### Selected Halogenated Organic Compounds

Percentage recovery ranges for three halo-organic compounds in water-quality samples were PCB congener 202- $^{13}C_{12}$ , 31.8 to 92.3 percent; p,p'-DDT- $d_8$ , 43.6 to 126 percent; and 4,4'-dibromooctafluorobiphenyl, 31.5 to 121 percent.

**Table 6.** Summary of field quality-assurance samples collected from sites nationwide for the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.

[QA, quality assurance; NWQL, National Water Quality Laboratory; PFRG, Pesticide Fate Research Group; OGRL, Organic Geochemistry Research Laboratory; NRP, National Research Program; NFHRL, National Fish Health Research Laboratory]

Constituent group	Field blanks	Matrix spikes
NWQL		
Wastewater compounds	3	4
Pharmaceuticals	3	4
Halogenated organic compounds	3	4
Nutrients	3	0
Trace elements	3	0
Hormones	4	4
Ambient purgeable volatile organic compounds	3	4
Heat purgeable volatile organic compounds	3	4
Pesticides	2	4
Polar pesticide compounds	2	4
PFRG		
Pesticides (gas chromatography method)	4	4
Pesticides (liquid chromatography method)	3	4
Disinfectant by-products	3	4
OGRL		
Antibiotics	8	4
Triazine and phenylurea parents and degradation products	8	4
Glyphosate	8	4
Hormones	8	4
Acetaminde parents and degradation products	8	4
NRP		
Surfactants	2	3
NFHRL		
BioAssay	2	3

### Heat Purgeable Volatile Organic Compounds

Percentage recovery ranges for three volatile organic compounds, using the heat purgeable method, in water-quality samples were 1,2-dichloroethane- $d_4$ , 86.4 to 121 percent; toluene- $d_8$ , 93.4 to 107 percent; and 1-bromo-4-fluorobenzene, 80.9 to 108 percent.

### Ambient Purgeable Volatile Organic Compounds

Percentage recovery range for one ambient purgeable volatile organic compound was 1-bromo-3-chloropropane- $d_6$ , 71.1 to 95.0 percent.

### Organic Geochemistry Research Laboratory

Laboratory matrix spike analyses were performed for the OGRL-requested methods. The apparent spike recoveries for acetamide herbicides and degradation products ranged from 26 to 280 percent. For glyphosate, glufosinate, and amino-methylphosphonic acid, the recoveries ranged from 54 to 103 percent. For the triazine and phenylurea herbicides, recoveries ranged from 52 to 180 percent. Finally for the selected antibiotics, recoveries ranged from 64 to 170 percent.

## Pesticide Fate Research Group

Laboratory matrix spike and blank spikes were performed at the PFRG and are summarized here. For the pesticide and pesticide degradates method, matrix spike recoveries ranged from 48.7 to 123 percent. The laboratory blank spike recovery ranged from 74.2 to 106 percent. Surrogate recoveries for 13C-atrazine ranged from 75.4 to 118 percent. Matrix spike recoveries for diuron and diuron degradate and neonicotinoid insecticides ranged from 70.2 to 118 percent. Surrogate recoveries for monuron ranged from 72.8 to 112 percent and for imidacloprid from 71.2 to 118 percent. For the disinfectant by-product method, matrix spike recoveries ranged from 66.6 to 117 percent. Surrogate recoveries for 1-bromo-4-fluorobenzene ranged from 43.6 to 117 percent and for 1,2-dichlorobenzene-d4 from 70.2 to 116 percent.

## U.S. Environmental Protection Agency Laboratories

Primary and secondary samples analyzed for perfluorinated compounds were subject to performance surrogates, laboratory blanks and spikes, duplicate analysis, matrix spikes, calibration verification, and instrument blanks. Corrective action was done as necessary (table 7). Results that did not pass the quality-control requirements were not reported. For surface-water samples analyzed for this study, the percentage recovery of matrix spikes ranged from  $72.9 \pm 21.4$  to  $100 \pm 7.88$  percent per compound. Laboratory spike recoveries ranged from  $53.2 \pm 24.2$  to  $106 \pm 8.92$  percent per compound. Relative percent difference between primary and secondary

samples ranged from  $2.43 \pm 1.54$  to  $92.4 \pm 74.4$  percent per compound for samples with detections.

## National Research Program

In addition to routine laboratory quality control consisting of instrumental calibrations, distilled water blanks, and the field blanks and replicates, this dataset underwent extensive additional quality assurance. Every sample ( $n=29$ ) was spiked with surrogate standards before processing and analyzed in duplicate ( $n=58$ ). In addition, 10 of the field samples were analyzed as duplicate matrix spikes for a total of 68 analyses. Finally, only one target compound was detected in a single field blank replicate (EDTA,  $0.07 \mu\text{g/L}$ ). Laboratory surrogate standard percentage recoveries ranged from 14 to 278 percent for EDTA and 26 to 473 percent for NP2EC. Each sample received at the laboratory was run in duplicate, and in general, the average concentration for each pair of duplicate samples varied from no detection (nd) to  $3.16 \mu\text{g/L}$  for NTA,  $0.05$  to  $160.3 \mu\text{g/L}$  for EDTA, nd to  $69.0 \mu\text{g/L}$  for DTPA, nd to  $5.76 \mu\text{g/L}$  for NP1EC, and nd to  $11.1 \mu\text{g/L}$  for NP2EC. Matrix spike recoveries ranged from 20 to 223 percent for NTA, 8 to 139 percent for EDTA,  $-391$  to 394 percent for DTPA, 29 to 436 percent for NP1EC, and 36 to 386 percent for NP2EC.

## Geographic Information System Methods

A Geographic Information System (GIS) was used to create, interpret, and analyze spatial datasets representing potential sources of anthropogenic disturbance for watersheds located throughout the United States. Sampling site

**Table 7.** Quality-control procedures for perfluorinated compounds analyzed at the U.S. Environmental Protection Agency, Environmental Chemistry Branch at John C. Stennis Space Center in Mississippi, for surface-water samples collected as part of the U.S. Geological Survey Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–December 2013.

[QC, quality control;  $\leq$ , less than or equal to;  $>$ , greater than; LCMRL, lowest concentration minimal reporting level; RSD, relative standard deviation;  $\pm$ , plus or minus]

QC sample type	Frequency	Acceptance criteria*
Performance surrogate	Every sample	25–150 percent
Laboratory blank	One every batch analyzed	$\leq$ LCMRL**
Laboratory control spike	One every batch analyzed	70–130 percent
Sample duplicate	One at every facility sampled	RSD = $\pm 30$ percent
Sample matrix spike	One at every facility sampled	50–150 percent
Continuing calibration verification	1 every 10 injections	$\pm 20$ percent***
Instrument blank	1 every 10 injections	$\leq$ LCMRL**

\*Data will not be rejected due to the loss of acceptance criteria by individual analytes. The results will be subject to review and corrective actions.

\*\*Depending upon signal to noise ratio and matrix interference ( $>$ LCMRL or background).

\*\*\* $\pm 20$  percent for compounds with complementary surrogates and  $\pm 30$  percent for those without.

coordinates were obtained from the project manager and used to create a point shapefile of sampling site locations in ArcGIS. For sites that were previously sampled by the USGS National Water-Quality Assessment (NAWQA) Project, watershed boundary polygon shapefiles were downloaded from the NAWQA spatial data Web site (<https://internal.cida.usgs.gov/wiki/display/ADIAS/NAWQA+Spatial+Data;jsessionid=4D1025098492CEA2FF6F3659651AA5A7>, accessed December 21, 2016). For those sites not previously sampled, watersheds were generated on the basis of 12-digit hydrologic unit code (HUC) boundaries found in the Watershed Boundary Dataset (WBD) created as part of the National Hydrography Dataset (NHD) (<http://nhd.usgs.gov/>, accessed December 21, 2016). Final watershed boundaries were created in ArcGIS by editing the WBD linework on-screen in ArcGIS to match actual sampling point locations using digital raster graphics of USGS 7.5-minute topographic maps and NHD high resolution streamlines as background layers. The final watersheds ranged in area from 4.6 to 17,264 square miles.

Spatial datasets representing landscape metrics of watershed disturbance were created for each watershed from available national and regional datasets. Spatial datasets included land cover, hydrography, National Pollutant Discharge Elimination System (NPDES) and Toxic Release Inventory (TRI)

site locations, population density, county estimates of nitrogen and phosphorus from farm and non-farm fertilizer and manure use, nitrogen from atmospheric deposition, county-based farm animal counts, and wetland areas. These databases containing landscape metric data types and source information are listed in table 8.

Two types of landscape metric data—raster and vector—were processed differently to obtain summary statistics for each watershed. Landscape metric data in vector format were processed by intersecting the watershed polygons with the landscape datasets. Watersheds commonly crossed county boundaries; therefore, landscape metrics derived from county estimates (nitrogen and phosphorus from fertilizer and manure, and animal counts) were spatially weighted to better estimate their occurrence in an individual watershed. In brief, the area of each county within a particular watershed was calculated, and the percentage relative to the full county area was determined. County-based landscape metric values were multiplied by the percentage of the county area in a particular watershed to better approximate use in the watershed and to eliminate over reporting of applications for adjacent watersheds. Summary statistics for each watershed were then calculated. Landscape metric data in raster format were processed using the zonal statistics tool found in the ArcGIS, Spatial

**Table 8.** Landscape metric data types and source information.

[All websites were accessed on December 22, 2016. NPDES, National Pollutant Discharge Elimination System; TRI, Toxic Release Inventory; USGS, U.S. Geological Survey; EPA, U.S. Environmental Protection Agency; NAWQA, National Water-Quality Assessment; USDA, U.S. Department of Agriculture; USFWS, U.S. Fish and Wildlife Service]

Landscape metric	Data type	Data year	U.S. data source	Reference or source link
Land cover	Raster	2011 (2001 Puerto Rico)	National Land Cover Database	<a href="http://www.mrlc.gov/index.php">http://www.mrlc.gov/index.php</a>
Hydrography	Vector	2014	USGS National Hydrography Dataset V.2.20	<a href="http://nhd.usgs.gov/">http://nhd.usgs.gov/</a>
NPDES program sites	Vector	2014 (Downloaded June 9, 2015)	EPA	<a href="https://www.epa.gov/enviro/data-downloads">https://www.epa.gov/enviro/data-downloads</a>
TRI sites	Vector	2014 (Downloaded June 9, 2015)	EPA	<a href="https://www.epa.gov/enviro/data-downloads">https://www.epa.gov/enviro/data-downloads</a>
Population density	Vector	2010	U.S. Census Bureau 2010 Census	<a href="http://www.census.gov/geo/maps-data/data/tiger-data.html">http://www.census.gov/geo/maps-data/data/tiger-data.html</a>
Nitrogen and phosphorus from fertilizer	Vector	2006	USGS NAWQA Project	Gronberg and Spahr (2012)
Manure use	Vector	2002	USGS NAWQA Project	Mueller and Gronberg (2013)
Nitrogen atmospheric deposition	Raster	2011	National Atmospheric Deposition Program	<a href="http://nadp.sws.uiuc.edu/data/annualmaps.aspx">http://nadp.sws.uiuc.edu/data/annualmaps.aspx</a>
Agricultural pesticide use	Tabular	2009	USGS NAWQA Project	Stone (2013)
Animal counts	Vector	2012	USDA U.S. Census of Agriculture	<a href="http://www.agcensus.usda.gov/Publications/2012/">http://www.agcensus.usda.gov/Publications/2012/</a>
Wetland types	Vector	2014	USFWS National Wetlands Inventory	<a href="http://www.fws.gov/wetlands/">http://www.fws.gov/wetlands/</a>



Analyst extension. All work was conducted using ArcGIS, ArcMap 10.2 (Environmental Systems Research Institute, Redlands, Calif.) GIS software.

Two sites sampled during the study have upstream catchments that extend outside the United States (station names are Fishtrap Creek at IB near Lynden, Washington, and New River near Westmorland, California). The latest version of the WBD includes areas outside the United States for streams that flow into the United States; therefore, the watershed boundaries for Fishtrap Creek and the New River were derived from the WBD as well. Additionally, landscape metric data generally were not available for the portions of these two watersheds outside the United States with the exceptions of land cover, population, and hydrology. Land-cover data in vector format for Mexico were downloaded from DataBasin.org (<http://databasin.org/datasets/566d1770025a45829bf76cb3db1d2c57>, accessed December 21, 2016) and were used to estimate land-cover statistics for the portion of the New River watershed in Mexico. Land-cover data developed in the year 2000 in vector format for Canada were downloaded from GeoBase.ca (<http://open.canada.ca/data/en/dataset/97126362-5a85-4fe0-9dc2-915464cfdabb7>, accessed December 21, 2016) and were used to estimate land-cover statistics for the Fishtrap Creek watershed. Population data for Mexico and Canada were obtained from Instituto Nacional de Estadística y Geografía (<http://www.inegi.org.mx/default.aspx>, accessed December 21, 2016) and Statistics Canada (<http://www.statcan.gc.ca/>, accessed December 21, 2016), respectively. Hydrology data for the Mexican and Canadian portions of the watersheds mentioned above were obtained from the NHD V. 2.20.

Landscape metrics were also derived for two watersheds located in Puerto Rico. For these two watersheds, land-cover data for 2001 were used rather than 2011 as used for the continental U.S. watersheds because 2001 data were the most recent available. Additionally, county estimates of nitrogen and phosphorus from farm and non-farm fertilizer and manure use and data on nitrogen from atmospheric deposition were not available for Puerto Rico.

Information from these databases gathered for this study is available at Romanok and others, 2017.

## Results

Physical parameters and inorganic results for each site are reported in site-specific field sheets provided in appendix 2. The complete suite of results for inorganic and organic compounds analyzed for this study is available at U.S. Geological Survey (2017) and Romanok and others (2017).

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# Appendixes

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Appendix 1. Example of protocols provided to field personnel for the preparation, collection, processing, and shipping of samples collected for the Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.

Appendix 2. Site-specific pages containing site, sampling, and other basin information as well as physical parameter and inorganic chemical data collected at each site.

**Appendix 1. Example of protocols provided to field personnel for the preparation, collection, processing, and shipping of samples collected for the Toxic Substances Hydrology Program, Chemical Mixtures Pilot Study, November 2012–June 2014.**

## **Toxics Chemical Mixtures Study**

### **USGS Bottle List, Filling Instructions and General Instructions**

Station ID-Site Name

**Lead Sampler:**

Lead Sampler  
U.S. Geological Survey  
Address 1  
Address 2  
Contact phone number

**General Instructions:**

1. If you have any questions before, after or during sampling call *Project Lead*(XXX-XXX-XXXX) to discuss. If something isn't clear, please ask questions. Don't just "wing it".
2. Please wear nitrile gloves when handling bottles and observe all trace-level sampling procedures.
3. Please complete a USGS SW field form. No not leave blank fields. If something is not applicable, please write in "N/A".
4. Please take photographs of the sampling location from a point of reference (ex. bridge or road crossing), upstream of the sampling location, downstream of the sampling locations, any contaminant sources or unusual conditions and "action shots" of sampling and sample processing. Please provide word file with a brief description of each picture or compile the pictures into a power point with a brief description of each picture.
5. Please measure field parameters (pH, SC, air and water temperature, DO, turbidity). Please record these measurements and all calibration information (including standard lot numbers) on the field form.
6. Please scan the field form and copies of completed ASRs.
7. Please copy all off the above files into our ftp site under the folder for your site. *ftp site url*
8. Please ship the samples on the same day they are collected. Many of the samples have short hold times.

### **Analytical Schedules:**

NWQL Schedule 4433 - Wastewater compounds  
    NWQL Lab Code 8244 - GC Pharmaceuticals add-on to NWQL Schedule 1433  
    NWQL Lab Code 2753 - TIC analysis  
NWQL Schedule 9017 - DAI LC/MS/MS pharmaceuticals  
    NWQL Lab Code 4200 - Sample filtration, lab, organic analysis  
NWQL Schedule 4434 - Hormones in filtered water  
NWQL Lab Code 8099 - Halogenated organics (PCBs and PBDEs)  
NWQL Schedule 2521 - Whole water nutrients and TOC  
NWQL Schedule 2524 - Whole water major ions, trace metals, turbidity and ANC  
    NWQL Lab Code 4205 - sample filtration, lab, inorganic analysis  
NWQL Lab Code 8170 - MRDP Forensic analysis via LC Q-TOF  
NWQL Schedule 4440 – 100 VOCs +TIC analysis via ambient purge  
NWQL Schedule 4437 – 38 VOCs via heated purge and trap  
    NWQL Lab Code 8252 – TIC analysis  
NWQL Schedule 2033 – GC/MS pesticides  
    NWQL Lab Code 4200- Sample filtration, lab, organics analysis  
NWQL Schedule 2060 – HPLC/MS-SPE, polar pesticides and metabolites  
    NWQL Lab Code 4200- Sample filtration, lab, organics analysis  
PFRG - GC pesticides and GC/MS forensics  
PFRG – LC pesticides  
PFRG - Disinfection byproducts (DBP)  
OGR L CAB - Antibiotics  
OGR L CEA - Triazine pesticides and degradates  
OGR L CGY - Glyphosate, degradate, and glufosinate  
OGR L CHM - Hormones  
OGR L CPD - Acetanilide pesticides and degradates  
L. Barber – Surfactants (ENV and BLANK)  
L. Iwanowicz – BLYES and Zebrafish E2 reporter bioassays (ENV and BLANK)  
S. Boone - Polyfluorinated compounds

### **USGS Bottle Count:**

7 40 mL amber glass vials (1 PHARM, 6 GCV)  
1 125 mL amber glass bottles (1 TOC)  
13 1 L amber glass bottles (13 GCC)  
1 125 mL clear polyethylene bottle (1 WCA)  
1 250 mL acid rinsed, clear polyethylene bottle for metals analysis (1 RA)  
2 250 mL natural polyethylene bottle (1 RU, 1 FU)  
5 500 mL clear polyethylene bottle (1 TBY, 4 HUN)  
2 1 L brown nalgene wide-mouth bottle (2 PFC)



## **Sampling and Bottle Filling Instructions:**

### **1. Fill pre-cleaned churn with ~6L organic blank water**

HUN – 1 500mL clear polyethylene bottle, LEAVE HEADSPACE FOR FREEZER EXPANSION, NO RINSE OR TREATMENT, NOTE BOTTLE LOT # ON LABEL; CHILL (NWQL LC 8170)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL (PFRG Forensic blank)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL (OGRL Forensic blank)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL (Barber - Surfactants) - BLANK

GCC – 1 L amber glass, NO RINSE, NO TREATMENT, CHILL (L. Iwanowicz - BioAssays) - BLANK

### **2. Collect the following from the stream (grab samples)**

EPA Cincinnati bottle #4 (see instructions within cooler for details)

EPA Cincinnati sample collection bottle, fill to line, then decant into bottle #10 (see instructions within cooler for details)

TOC – 1 125mL amber glass bottle, NO RINSE, FILL BOTTLE TO SHOULDER ONLY (NWQL SCH 2521)

PFC – 2 1L brown, Nalgene, wide-mouth bottle, NO CONTACT WITH TEFLON, NO RINSE, FILL BOTTLE TO SHOULDER ONLY (S. Boone - PFCs)

### **3. Collect 6 40mL samples using VOC sampler**

GCV – 3 40mL amber glass vials, NO RINSE, FILL COMPLETELY, ACIDIFY TO pH<2 WITH 1:1 HCl:H<sub>2</sub>O SOLUTION, CHILL (NWQL SCH 4440)

GCV – 3 40mL amber glass vials, NO RINSE, FILL COMPLETELY, ACIDIFY TO pH<2 WITH 1:1 HCl:H<sub>2</sub>O SOLUTION, CHILL (NWQL SCH 4437+LC8252)

**4. Collect unfiltered samples fill directly from churn**

**Collect the 4 1L amber glass bottles within the EPA Duluth, MN cooler  
(see instructions within cooler for details)**

**Collect the 8 1L amber glass bottles within the EPA RTP, NC cooler,  
NO HEADSPACE, (see instructions within cooler for details)**

**Collect bottles #1-3, 5-9 within the EPA Cincinnati, OH cooler  
(see instructions within cooler for details)**

**PHARM – 1 40mL amber glass vial, NO RINSE OR TREATMENT, CHILL  
(NWQL LC 9017 + LC 4200)**

**WCA – 125 mL clear polyethylene bottle, RINSE WITH SAMPLE WA-  
TER, FILL BOTTLE, ACIDIFY TO pH<2 W/ 1 mL 4.5N H<sub>2</sub>SO<sub>4</sub>.  
CHILL. (NWQL SCH 2521)**

**RA – 250mL acid rinsed, clear polyethylene bottle, RINSE WITH SAM-  
PLE WATER, FILL BOTTLE, ACIDIFY TO pH<2 W/2 mL 7.5N  
ULTREX HNO<sub>3</sub> (NWQL SCH 2524)**

**FU – 250mL clear polyethylene bottle, RINSE WITH SAMPLE WATER,  
FILL BOTTLE, NO TREATMENT (NWQL SCH 2524+ LC 4205)  
LAB WILL FILTER**

**RU – 250mL clear polyethylene bottle, RINSE WITH SAMPLE WATER,  
FILL BOTTLE, NO TREATMENT (NWQL SCH 2524)**

**TBY – 500mL clear polyethylene bottle, RINSE WITH SAMPLE WATER,  
FILL BOTTLE, NO TREATMENT (NWQL SCH 2524)**

**HUN – 1 500mL clear polyethylene bottle, LEAVE HEADSPACE FOR  
FREEZER EXPANSION, NO RINSE OR TREATMENT, CHILL  
(NWQL SCH 4434)**

**HUN – 1 500mL clear polyethylene bottle, LEAVE HEADSPACE FOR  
FREEZER EXPANSION, NO RINSE OR TREATMENT, NOTE  
BOTTLE LOT # ON LABEL, CHILL (NWQL LC 8170)**

**HUN – 1 500mL clear polyethylene bottle, LEAVE HEADSPACE FOR  
FREEZER EXPANSION, NO RINSE OR TREATMENT, NOTE  
BOTTLE LOT # ON LABEL, CHILL (NWQL LC 8170)**

**GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL (NWQL  
SCH 4433+ LC 8244+ 2753)**

**GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL (NWQL  
LC 8099)**

**GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(NWQL SCH 2033+LC 4200)**

**Collect unfiltered samples fill directly from churn --CONTINUED**

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(NWQL SCH 2060+LC 4200)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(PFRG GC Pesticides + GC Forensics)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(PFRG LC Pesticides)

GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(PFRG DBPs)

GCC – 1 L amber glass bottle, NO RINSE OR TREATMENT, CHILL  
(OGRL – LC Pesticides, antibiotics and hormones)

GCC – 1 L amber glass bottle, NO RINSE OR TREATMENT, CHILL  
(OGRL – LC Pesticides, antibiotics and hormones)

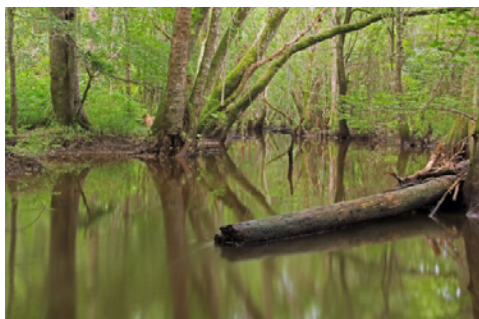
GCC – 1 L amber glass, NO RINSE OR TREATMENT, CHILL  
(Barber - Surfactants)

GCC – 1 L amber glass, NO RINSE, NO TREATMENT, CHILL  
(L. Iwanowicz - BioAssays)



## **Appendix 2. Site-specific pages containing site, sampling, and other basin information as well as physical parameter and inorganic chemical data collected at each site.**

[All data summaries in these sheets are available at Romanok and others, 2017; <https://doi.org/10.5066/F7GF0RPH>, accessed March 22, 2017. Maps provided were obtained from <http://ngmdb.usgs.gov/maps/Topoview>, accessed December 21, 2016. Photographs were taken by various field personnel listed in the Acknowledgments section. E, estimated; <, less than; NA, not available; mi<sup>2</sup>, square miles; %, percent; kg, kilogram; NTU, nephelometric turbidity units; mg/L, milligrams per liter; µg/L, micrograms per liter]



## Abrams Creek near Oakfield, GA

### Site Information

**Station ID:** 02350524

**Site Type:** Routine

**Sampling Date:** July 23, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 80.2 mi<sup>2</sup>

**Urban Land Use:** 1.05%

**Agricultural Land Use:** 30.2%

### Human and Agricultural Census Data

**Population:** 12.4

**Cattle and Calves:** 1,264

**Hogs:** 6.68

**Poultry:** 45.9

**Sheep:** 0

**Goats:** 58.5

**Horses:** 63.0

**Turkeys:** 1.7

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 417,200

**P applied as fertilizer to farms:** 142,173

**N applied as fertilizer to non-farms:** 2.51

**P applied as fertilizer to non-farms:** 0.318

**N produced from manure in CAFOs:** 27,636

**P produced from manure in CAFOs:** 6,089

**N produced from manure by unconfined animals:** 69,012

**P produced from manure by unconfined animals:** 18,307

**Atmospheric deposition of total organic N:** 51,594

### Pesticide Usage Summary (kg)

**Total:** 49,033

**Fungicides:** 5,792

**Fumigants:** 18,377

**Herbicides:** 15,136

**Insecticides:** 5,359

**Others:** 4,368

### Physical Parameters and Inorganic Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 11.2	6.18	24.3	61.1	5.67	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
5.36	2.03	1.68	11.3	2.54



***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.057	7.60	0.088	1.07

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
17.5	0.895	0.046

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.18	1.09	<0.016	0.7440	0.908
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
1.00	0.768	0.205	<0.06	0.157



## Blue River at Kansas City, MO

### Site Information

Station ID: 06893500

Site Type: Routine

Sampling Date: February 11, 2014

Gaging Station: Yes

### Basin Characteristics

Drainage Area: 188 mi<sup>2</sup> Urban Land Use: 41.4%

Agricultural Land Use: 29.0%

### Human and Agricultural Census Data

Population: 1,696

Cattle and Calves: 4,538

Hogs: 456

Poultry: 693

Sheep: 215

Goats: 155

Horses: 661

Turkeys: 8.22

### Industrial Dischargers

NPDES Major Dischargers: 2

EPA TRI Facilities: 12

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 698,236

P applied as fertilizer to farms: 92,128

N applied as fertilizer to non-farms: 1,124,874

P applied as fertilizer to non-farms: 156,452

N produced from manure in CAFOs: 95,513

P produced from manure in CAFOs: 23,993

N produced from manure by unconfined animals: 201,474

P produced from manure by unconfined animals: 62,454

Atmospheric deposition of total organic N: 288,445

### Pesticide Usage Summary (kg)

Total: 12,723

Fungicides: 139

Fumigants: 0

Herbicides: 11,979

Insecticides: 605

Others: 0.268

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
E 3.45	7.90	0.16	1,826	13.9	35.7

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
81.5	20.4	12.8	7.16	250

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.1936	379	0.548	155

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
7.56	9.32	1.31

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.308	1.14	0.055	0.786	2.14
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.241	2.03	0.653	<0.06	0.651



## C-111 Canal 100 feet above S-177 near Homestead, FL

### Site Information

**Station ID:** 252414080333200

**Site Type:** Routine

**Sampling Date:** April 9, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 51.0 mi<sup>2</sup>

**Urban Land Use:** 4.87%

**Agricultural Land Use:** 65.4%

### Human and Agricultural Census Data

**Population:** 298

**Cattle and Calves:** 103

**Hogs:** 6.55

**Poultry:** 211

**Sheep:** 40.1

**Goats:** 39.0

**Horses:** 61.8

**Turkeys:** 16.1

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 3,499,642

**P applied as fertilizer to farms:** 556,267

**N applied as fertilizer to non-farms:** 274,305

**P applied as fertilizer to non-farms:** 26,049

**N produced from manure in CAFOs:** 2,265

**P produced from manure in CAFOs:** 345

**N produced from manure by unconfined animals:** 26,555

**P produced from manure by unconfined animals:** 7,247

**Atmospheric deposition of total organic N:** 45,081

### Pesticide Usage Summary (kg)

**Total:** 274,686

**Fungicides:** 38,271

**Fumigants:** 196,432

**Herbicides:** 8,518

**Insecticides:** 30,628

**Others:** 838

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
<2.00	8.10	25.0	533	7.30	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
79.5	15.2	3.07	6.11	58.8

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
NA	NA	0.21	3.84

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
18.3	1.02	0.009

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.211	0.970	<0.016	<0.300	1.16
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.240	1.95	0.211	<0.060	1.11





## Chicago Sanitary and Ship Canal at Lockport, IL

### Site Information

Station ID: 05537000

Site Type: Routine

Sampling Date: February 11, 2014 Gaging Station: No

### Basin Characteristics

Drainage Area: 740 mi<sup>2</sup>

Urban Land Use: 77.3%

Agricultural Land Use: 1.86%

### Human and Agricultural Census Data

Population: 5,773

Cattle and Calves: 644

Hogs: 311

Poultry: 812

Sheep: 132

Goats: 123

Horses: 1,544

Turkeys: 12.3

### Industrial Dischargers

NPDES Major Dischargers: 23

EPA TRI Facilities: 901

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 427,497

P applied as fertilizer to farms: 62,552

N applied as fertilizer to non-farms: 2,806,700

P applied as fertilizer to non-farms: 334,452

N produced from manure in CAFOs: 16,379

P produced from manure in CAFOs: 3,546

N produced from manure by unconfined animals: 9,363

P produced from manure by unconfined animals: 2,302

Atmospheric deposition of total organic N: 1,558,529

### Pesticide Usage Summary (kg)

Total: 5,838

Fungicides: 372

Fumigants: 22

Herbicides: 5,124

Insecticides: 319

Others: 0.978

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
E 3.53	7.52	8.04	1,878	7.71	1,750

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
65.9	22.6	8.05	5.11	275

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.180	420	0.820	117

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
6.49	7.92	0.705

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.416	1.13	0.056	1.27	2.90
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.694	4.65	0.776	<0.06	0.509



## Chisholm Creek near Edmond, OK

### Site Information

**Station ID:** 07159735

**Site Type:** Routine

**Sampling Date:** November 28, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 38.8 mi<sup>2</sup>

**Urban Land Use:** 50.4%

**Agricultural Land Use:** 5.38%

### Human and Agricultural Census Data

**Population:** 2,289

**Cattle and Calves:** 800

**Hogs:** 33.5

**Poultry:** 127

**Sheep:** 50.0

**Goats:** 125

**Horses:** 182

**Turkeys:** 5.83

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 7

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 74,483

**P applied as fertilizer to farms:** 8,175

**N applied as fertilizer to non-farms:** 58,887

**P applied as fertilizer to non-farms:** 4,394

**N produced from manure in CAFOs:** 1,931

**P produced from manure in CAFOs:** 343

**N produced from manure by unconfined animals:** 62,298

**P produced from manure by unconfined animals:** 18,577

**Atmospheric deposition of total organic N:** 40,321

### Pesticide Usage Summary (kg)

**Total:** 443

**Fungicides:** 18.2

**Fumigants:** 0

**Herbicides:** 325

**Insecticides:** 73.2

**Others:** 27.0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 7.14	7.10	9.41	1,028	7.08	4.00

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
46.6	25.1	12.8	7.73	116

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.2164	133	0.462	135

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
5.26	12.3	2.12

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.419	1.66	0.037	0.732	2.52
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.323	1.21	0.735	<0.06	0.745



## Deep Creek at Highway 224, OR

### Site Information

Station ID: 452340122251000

Site Type: Routine

Sampling Date: January 13, 2014

Gaging Station: No

### Basin Characteristics

Drainage Area: 49.0 mi<sup>2</sup>

Urban Land Use: 7.91%

Agricultural Land Use: 40.4%

### Human and Agricultural Census Data

Population: 392

Cattle and Calves: 590

Hogs: 20.3

Poultry: 0

Sheep: 128

Goats: 67.1

Horses: 144

Turkeys: 25.8

### Industrial Dischargers

NPDES Major Dischargers: 0

EPA TRI Facilities: 3

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 490,131

P applied as fertilizer to farms: 75,643

N applied as fertilizer to non-farms: 19,306

P applied as fertilizer to non-farms: 1,394

N produced from manure in CAFOs: 96,942

P produced from manure in CAFOs: 27,242

N produced from manure by unconfined animals: 74,212

P produced from manure by unconfined animals: 19,377

Atmospheric deposition of total organic N: 26,518

### Pesticide Usage Summary (kg)

Total: 6,077

Fungicides: 1,010

Fumigants: 2,398

Herbicides: 1,750

Insecticides: 914

Others: 4.69

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
E 11.0	7.20	8.00	61.0	12.0	310

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
29.1	9.53	1.12	22.6	50.5

*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.030	3.53	0.020	1.58

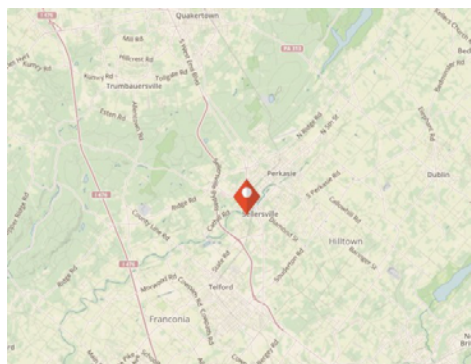
*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.44	2.32	0.057

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.331	0.746	<0.016	0.594	4.12
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.114	9.06	0.173	<0.060	0.017





## East Branch Perkiomen Creek near Derstines, PA

### Site Information

**Station ID:** 01472705

**Sampling Date:** January 14, 2014

**Site Type:** Routine

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 29.4 mi<sup>2</sup>

**Urban Land Use:** 13.5%

**Agricultural Land Use:** 43.7%

### Human and Agricultural Census Data

**Population:** 929

**Cattle and Calves:** 329

**Hogs:** 25.0

**Poultry:** 540

**Sheep:** 90.0

**Goats:** 35.4

**Horses:** 152

**Turkeys:** 40.7

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 7

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 94,947

**P applied as fertilizer to farms:** 17,774

**N applied as fertilizer to non-farms:** 25,826

**P applied as fertilizer to non-farms:** 3,565

**N produced from manure in CAFOs:** 24,751

**P produced from manure in CAFOs:** 4,778

**N produced from manure by unconfined animals:** 16,600

**P produced from manure by unconfined animals:** 4,243

**Atmospheric deposition of total organic N:** 47,133

### Pesticide Usage Summary (kg)

**Total:** 3,688

**Fungicides:** 544

**Fumigants:** 0

**Herbicides:** 2,644

**Insecticides:** 480

**Others:** 18.9

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 8.36	7.20	4.90	490	16.6	116

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
25.3	8.94	2.78	8.05	55.7

***Major Anions (mg/L, filtered):***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.035	108	0.074	17.7

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
4.21	1.99	0.086

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.693	0.035	0.720	2.43
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.713	0.919	0.191	<0.060	0.301



## Enoree River at Pelham, SC

### Site Information

**Station ID:** 02160326

**Sampling Date:** May 19, 2014

**Site Type:** Routine

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 84.2 mi<sup>2</sup>

**Agricultural Land Use:** 8.48%

**Urban Land Use:** 23.6%

### Human and Agricultural Census Data

**Population:** 1,110      **Cattle and Calves:** 717      **Hogs:** 53.6      **Poultry:** 366  
**Sheep:** 35.0      **Goats:** 222      **Horses:** 226      **Turkeys:** 12.0

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 29

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 122,275      **P applied as fertilizer to farms:** 19,703  
**N applied as fertilizer to non-farms:** 171,529      **P applied as fertilizer to non-farms:** 26,587  
**N produced from manure in CAFOs:** 5,455      **P produced from manure in CAFOs:** 1,030  
**N produced from manure by unconfined animals:** 57,994  
**P produced from manure by unconfined animals:** 17,245  
**Atmospheric deposition of total organic N:** 72,697

### Pesticide Usage Summary (kg)

**Total:** 1,611      **Fungicides:** 613      **Fumigants:** 244      **Herbicides:** 531  
**Insecticides:** 222      **Others:** 0.277

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 8.65	6.92	16.1	43.0	9.78	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
3.19	1.20	1.50	10.9	3.25

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.030	3.25	0.040	1.78

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.07	0.636	0.019

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	<0.280	<0.030	0.569	1.45
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.490	0.901	<0.100	<0.060	0.059



## Fall Creek near Ithaca, NY

### Site Information

Station ID: 04234000

Sampling Date: June 9, 2014

Site Type: Routine

Gaging Station: Yes

### Basin Characteristics

Drainage Area: 126 mi<sup>2</sup>

Agricultural Land Use: 45.3%

Urban Land Use: 1.26%

### Human and Agricultural Census Data

Population: 124

Cattle and Calves: 6,891

Hogs: 143

Poultry: 2,224

Sheep: 524

Goats: 148

Horses: 461

Turkeys: 11.4

### Industrial Dischargers

NPDES Major Dischargers: 0

EPA TRI Facilities: 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 288,781

P applied as fertilizer to farms: 47,267

N applied as fertilizer to non-farms: 21,760

P applied as fertilizer to non-farms: 2,898

N produced from manure in CAFOs: 316,361

P produced from manure in CAFOs: 58,936

N produced from manure by unconfined animals: 135,146

P produced from manure by unconfined animals: 30,001

Atmospheric deposition of total organic N: 209,708

### Pesticide Usage Summary (kg)

Total: 15,063

Fungicides: 3,311

Fumigants: 63.1

Herbicides: 10,339

Insecticides: 1,328

Others: 21.9

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
59.5	8.03	17.9	319	9.33	632

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
37.1	7.54	1.99	7.24	15.8

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.030	25.1	0.061	8.64

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
9.68	1.87	0.163

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	1.21	0.038	2.55	3.56
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
2.22	3.80	<0.100	<0.060	0.246





## Fishtrap Creek at 1B near Lyndon, WA

### Site Information

**Station ID:** 12211900

**Site Type:** Routine

**Sampling Date:** December 10, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 16.5 mi<sup>2</sup>

**Urban Land Use:** 50.4%

**Agricultural Land Use:** 36.3%

### Human and Agricultural Census Data

**Population:** NA

**Cattle and Calves:** NA

**Hogs:** NA

**Poultry:** NA

**Sheep:** NA

**Goats:** NA

**Horses:** NA

**Turkeys:** NA

### Industrial Dischargers

**NPDES Major Dischargers:** NA

**EPA TRI Facilities:** NA

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** NA

**P applied as fertilizer to farms:** NA

**N applied as fertilizer to non-farms:** NA

**P applied as fertilizer to non-farms:** NA

**N produced from manure in CAFOs:** NA

**P produced from manure in CAFOs:** NA

**N produced from manure by unconfined animals:** NA

**P produced from manure by unconfined animals:** NA

**Atmospheric deposition of total organic N:** NA

### Pesticide Usage Summary (kg)

**Total:** NA

**Fungicides:** NA

**Fumigants:** NA

**Herbicides:** NA

**Insecticides:** NA

**Others:** NA

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 3.44	6.67	4.32	230	9.70	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
23.6	6.57	2.87	14.7	9.04

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.042	10.7	0.033	27.8

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
3.27	3.03	0.032

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.754	<0.030	0.408	1.47
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.173	1.94	0.163	<0.060	0.057



## Fourmile Creek below Ankeny WWTP outfall, IA

### Site Information

**Station ID:** 05485605

**Site Type:** Routine

**Sampling Date:** November 19, 2012

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 60.9 mi<sup>2</sup>

**Urban Land Use:** 9.53%

**Agricultural Land Use:** 77.7%

### Human and Agricultural Census Data

**Population:** 393

**Cattle and Calves:** 971

**Hogs:** 1,836

**Poultry:** 190

**Sheep:** 103

**Goats:** 39.2

**Horses:** 127

**Turkeys:** 2,465

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 2

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 1,261,763

**P applied as fertilizer to farms:** 179,403

**N applied as fertilizer to non-farms:** 43,149

**P applied as fertilizer to non-farms:** 6,503

**N produced from manure in CAFOs:** 62,719

**P produced from manure in CAFOs:** 20,548

**N produced from manure by unconfined animals:** 55,102

**P produced from manure by unconfined animals:** 19,193

**Atmospheric deposition of total organic N:** 94,760

### Pesticide Usage Summary (kg)

**Total:** 21,168

**Fungicides:** 358

**Fumigants:** 0

**Herbicides:** 19,857

**Insecticides:** 953

**Others:** 0.005

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 5.37	7.23	16.8	856	6.70	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
55.3	18.3	11.9	12.0	79.7

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.144	92.8	<0.040	75.9

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
8.59	12.6	4.59

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.364	1.09	0.054	0.432	4.61
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.300	1.71	0.450	<0.060	0.496



## Hawksbill Creek at Rt. 648 at Springfield, VA

### Site Information

**Station ID:** 01630540

**Site Type:** Routine

**Sampling Date:** April 15, 2014

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 68.2 mi<sup>2</sup>

**Urban Land Use:** 4.22%

**Agricultural Land Use:** 31.6%

### Human and Agricultural Census Data

**Population:** 140

**Cattle and Calves:** 5,066

**Hogs:** 285

**Poultry:** 45,009

**Sheep:** 171

**Goats:** 104

**Horses:** 92.1

**Turkeys:** 160,123

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 1

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 62,395

**P applied as fertilizer to farms:** 15,418

**N applied as fertilizer to non-farms:** 8,915

**P applied as fertilizer to non-farms:** 1,684

**N produced from manure in CAFOs:** 1,029,666

**P produced from manure in CAFOs:** 321,200

**N produced from manure by unconfined animals:** 181,065

**P produced from manure by unconfined animals:** 55,600

**Atmospheric deposition of total organic N:** 68,565

### Pesticide Usage Summary (kg)

**Total:** 2,982

**Fungicides:** 355

**Fumigants:** 0

**Herbicides:** 2,395

**Insecticides:** 230

**Others:** 1.56

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 2.92	7.50	14.8	181	8.98	106

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
20.1	5.84	1.73	7.90	4.24

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.030	6.14	0.069	8.40

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.20	1.33	0.055

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	<0.280	<0.030	0.387	<0.800
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.151	<0.200	<0.100	<0.060	0.044





## Hillsboro Canal at S-6 near Shawano, FL

### Site Information

**Station ID:** 02281200

**Site Type:** Routine

**Sampling Date:** March 12, 2013

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 311 mi<sup>2</sup>

**Urban Land Use:** 1.58%

**Agricultural Land Use:** 93.4%

### Human and Agricultural Census Data

**Population:** 74.9

**Cattle and Calves:** 1,309

**Hogs:** 14.9

**Poultry:** 456

**Sheep:** 137

**Goats:** 209

**Horses:** 983

**Turkeys:** 4.77

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 8

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 6,330,637

**P applied as fertilizer to farms:** 1,006,253

**N applied as fertilizer to non-farms:** 67,905

**P applied as fertilizer to non-farms:** 6,448

**N produced from manure in CAFOs:** 5,378

**P produced from manure in CAFOs:** 784

**N produced from manure by unconfined animals:** 157,259

**P produced from manure by unconfined animals:** 41,011

**Atmospheric deposition of total organic N:** 214,914

### Pesticide Usage Summary (kg)

**Total:** 1,156,787

**Fungicides:** 124,760

**Fumigants:** 400,926

**Herbicides:** 369,102

**Insecticides:** 257,301

**Others:** 4,698

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
<2.00	7.80	21.4	1,220	5.10	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
87.6	28.4	7.72	15.3	117

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.581	190	0.828	52.9

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
34.7	2.21	0.015

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	1.32	<0.016	<0.300	<0.700
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
<0.040	<0.190	0.200	<0.060	0.160



## Hite Creek at Sleepy Hollow Rd. near Prospect, KY

### Site Information

**Station ID:** 382054085332600

**Sampling Date:** May 20, 2014

**Site Type:** Routine

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 5.55 mi<sup>2</sup>

**Urban Land Use:** 51.9%

**Agricultural Land Use:** 10.3%

### Human and Agricultural Census Data

**Population:** 2,323

**Cattle and Calves:** 63.9

**Hogs:** 0.200

**Poultry:** 18.1

**Sheep:** 4.55

**Goats:** 3.03

**Horses:** 23.2

**Turkeys:** 0.366

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 5

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 10,285

**P applied as fertilizer to farms:** 2,172

**N applied as fertilizer to non-farms:** 18,887

**P applied as fertilizer to non-farms:** 2,826

**N produced from manure in CAFOs:** 759

**P produced from manure in CAFOs:** 178

**N produced from manure by unconfined animals:** 6,622

**P produced from manure by unconfined animals:** 1,853

**Atmospheric deposition of total organic N:** 10,519

### Pesticide Usage Summary (kg)

**Total:** 99.3

**Fungicides:** 9.29

**Fumigants:** 18.7

**Herbicides:** 62.9

**Insecticides:** 6.73

**Others:** 1.59

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 2.02	8.30	16.3	735	9.88	12.7

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
26.2	23.9	4.50	53.7	52.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.252	81.8	0.620	56.1

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
4.73	9.60	0.349

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	8.45	0.086	13.6	14.6
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
2.24	23.1	0.424	<0.060	1.65



## Hohokus Brook at mouth at Paramus, NJ

### Site Information

**Station ID:** 01391100

**Site Type:** Routine

**Sampling Date:** November 28, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 20.2 mi<sup>2</sup>

**Urban Land Use:** 19.9%      **Agricultural Land Use:** 0.141%

### Human and Agricultural Census Data

**Population:** 2,746

**Cattle and Calves:** 0

**Hogs:** 0

**Poultry:** 245

**Sheep:** 0

**Goats:** 0

**Horses:** 0

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 2

**EPA TRI Facilities:** 6

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 4,717

**P applied as fertilizer to farms:** 766

**N applied as fertilizer to non-farms:** 124,534

**P applied as fertilizer to non-farms:** 15,180

**N produced from manure in CAFOs:** 198

**P produced from manure in CAFOs:** 63.6

**N produced from manure by unconfined animals:** 177

**P produced from manure by unconfined animals:** 45.2

**Atmospheric deposition of total organic N:** 31,756

### Pesticide Usage Summary (kg)

**Total:** 104

**Fungicides:** 47.4

**Fumigants:** 5.81

**Herbicides:** 13.0

**Insecticides:** 34.7

**Others:** 3.00

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 2.50	7.90	9.60	1,655	14.2	32.1

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
64.1	19.6	8.20	11.2	213

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.122	392	0.111	31.6

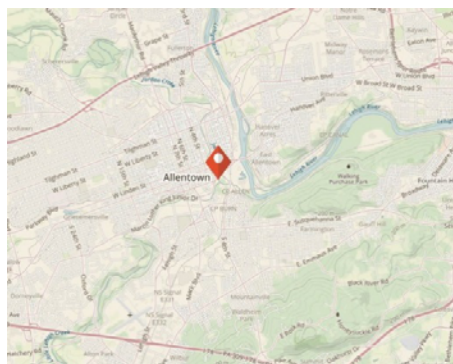
***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
4.79	11.0	1.97

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.208	0.835	0.037	<0.300	5.740
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.410	0.912	0.289	<0.060	0.387





## Jordan Creek at mouth at Allentown, PA

### Site Information

**Station ID:** 01452040

**Sampling Date:** June 17, 2013

**Site Type:** Routine

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 82.3 mi<sup>2</sup>

**Agricultural Land Use:** 48.5%

**Urban Land Use:** 13.8%

### Human and Agricultural Census Data

**Population:** 1,020

**Cattle and Calves:** 913

**Hogs:** 501

**Poultry:** 441

**Sheep:** 168

**Goats:** 154

**Horses:** 281

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 6

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 431,964

**P applied as fertilizer to farms:** 80,864

**N applied as fertilizer to non-farms:** 72,553

**P applied as fertilizer to non-farms:** 10,015

**N produced from manure in CAFOs:** 71,389

**P produced from manure in CAFOs:** 17,218

**N produced from manure by unconfined animals:** 9,501

**P produced from manure by unconfined animals:** 1,682

**Atmospheric deposition of total organic N:** 137,320

### Pesticide Usage Summary (kg)

**Total:** 21,289

**Fungicides:** 3,521

**Fumigants:** 59.3

**Herbicides:** 15,635

**Insecticides:** 1,930

**Others:** 143

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
NA	7.54	18.1	264	9.35	218

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
24.5	7.70	1.37	7.55	13.3

***Major Anions (mg/L, filtered)***

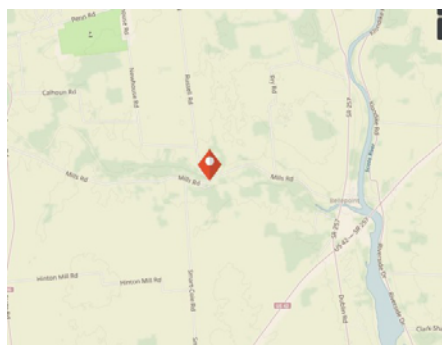
<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.036	24.3	0.048	23.0

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
1.75	4.10	0.064

***Selected Trace Metals (µg/L, unfiltered):***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.298	0.017	<0.300	0.968
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.901	0.423	0.190	<0.060	0.095



## Mill Creek near Bellepoint, OH

### Site Information

**Station ID:** 03220000

**Sampling Date:** June 4, 2013

**Site Type:** Routine

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 178 mi<sup>2</sup>

**Agricultural Land Use:** 75.5%

**Urban Land Use:** 5.40%

### Human and Agricultural Census Data

**Population:** 167

**Cattle and Calves:** 3,462

**Hogs:** 10,566

**Poultry:** 224

**Sheep:** 568

**Goats:** 325

**Horses:** 497

**Turkeys:** 22.7

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 9

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 2,101,407

**P applied as fertilizer to farms:** 353,176

**N applied as fertilizer to non-farms:** 26,629

**P applied as fertilizer to non-farms:** 2,961

**N produced from manure in CAFOs:** 283,656

**P produced from manure in CAFOs:** 86,242

**N produced from manure by unconfined animals:** 90,164

**P produced from manure by unconfined animals:** 31,030

**Atmospheric deposition of total organic N:** 354,362

### Pesticide Usage Summary (kg)

**Total:** 58,097

**Fungicides:** 1,060

**Fumigants:** 0

**Herbicides:** 55,920

**Insecticides:** 1,114

**Others:** 2.72

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 16.6	NA	NA	NA	NA	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
79.1	33.7	4.68	5.54	33.6

*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.044	50.4	0.496	108

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Carbon (inorganic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
6.20	NA	3.62	0.168

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.214	2.08	0.016	0.522	1.47
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.363	5.80	0.217	<0.060	2.76



## New River near Westmorland, CA

### Site Information

**Station ID:** 10255550

**Site Type:** Routine

**Sampling Date:** March 5, 2013

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 1,471 mi<sup>2</sup>

**Urban Land Use:** 4.92%

**Agricultural Land Use:** 27.3%

### Human and Agricultural Census Data

**Population:** 52.2

**Cattle and Calves:** 46,984

**Hogs:** 0

**Poultry:** 24.4

**Sheep:** 7,078

**Goats:** 37.9

**Horses:** 19.5

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 3

**EPA TRI Facilities:** 2

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 9,975,905

**P applied as fertilizer to farms:** 1,825,435

**N applied as fertilizer to non-farms:** 22,966

**P applied as fertilizer to non-farms:** 3,250

**N produced from manure in CAFOs:** 2,895,680

**P produced from manure in CAFOs:** 734,037

**N produced from manure by unconfined animals:** 1,362,448

**P produced from manure by unconfined animals:** 312,317

**Atmospheric deposition of total organic N:** 57,174

### Pesticide Usage Summary (kg)

**Total:** 0

**Fungicides:** 0

**Fumigants:** 0

**Herbicides:** 0

**Insecticides:** 0

**Others:** 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
85.5	7.80	16.0	3,790	12.7	617

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
171	80.9	12.5	20.5	508

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.959	748	0.494	691

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
14.0	12.8	1.38

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.36	6.19	0.122	7.30	6.00
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
3.10	6.92	2.45	<0.120	8.79



## North Dry Creek 2.0 mi southwest of Bridge South of Kearny, NE

### Site Information

**Station ID:** 06770195

**Sampling Date:** June 10, 2013

**Site Type:** Routine

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 77.7 mi<sup>2</sup>

**Urban Land Use:** 0.240%

**Agricultural Land Use:** 88.3%

### Human and Agricultural Census Data

**Population:** 3.47

**Cattle and Calves:** 23,317

**Hogs:** 47.5

**Poultry:** 53.4

**Sheep:** 140

**Goats:** 25.6

**Horses:** 38.5

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 1,975,047

**P applied as fertilizer to farms:** 298,242

**N applied as fertilizer to non-farms:** 0

**P applied as fertilizer to non-farms:** 0

**N produced from manure in CAFOs:** 704,698

**P produced from manure in CAFOs:** 180,162

**N produced from manure by unconfined animals:** 229,628

**P produced from manure by unconfined animals:** 57,733

**Atmospheric deposition of total organic N:** 136,782

### Pesticide Usage Summary (kg)

**Total:** 71,082

**Fungicides:** 4,211

**Fumigants:** 14,584

**Herbicides:** 51,557

**Insecticides:** 728

**Others:** 1.108

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 3.17	8.50	20.6	1,200	15.2	0.687

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
118	27.0	24.0	10.0	76.5



*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.211	36.4	0.293	402

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
7.71	2.64	<0.004

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.479	7.35	0.034	<0.300	1.09
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.140	2.05	12.5	<0.060	55.5



## North Sylamore Creek near Fifty Six, AR

### Site Information

**Station ID:** 07060710

**Site Type:** Routine

**Sampling Date:** January 7, 2014

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 58.1 mi<sup>2</sup>

**Urban Land Use:** 0.207%

**Agricultural Land Use:** 1.51%

### Human and Agricultural Census Data

**Population:** 1.36

**Cattle and Calves:** 2,225

**Hogs:** 4.55

**Poultry:** 89.9

**Sheep:** 17.6

**Goats:** 69.5

**Horses:** 83.39

**Turkeys:** 6,766

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 11,099

**P applied as fertilizer to farms:** 1,360

**N applied as fertilizer to non-farms:** 16.9

**P applied as fertilizer to non-farms:** 3.61

**N produced from manure in CAFOs:** 17,641

**P produced from manure in CAFOs:** 5,223

**N produced from manure by unconfined animals:** 14,088

**P produced from manure by unconfined animals:** 4,448

**Atmospheric deposition of total organic N:** 103,860

### Pesticide Usage Summary (kg)

**Total:** 62.1

**Fungicides:** 0.020

**Fumigants:** 0

**Herbicides:** 62.0

**Insecticides:** 0.101

**Others:** 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
NA	7.71	0.540	259	15.2	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
46.3	6.12	0.613	6.26	1.16

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.012	1.60	0.034	4.45

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
1.11	0.143	<0.004

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	<0.280	<0.016	<0.300	<0.7
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
<0.040	<0.190	0.061	<0.060	0.328



## Penn Swamp Branch near Batsto, NJ

### Site Information

**Station ID:** 01409480    **Site Type:** Routine

**Sampling Date:** December 10, 2012    **Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 4.80 mi<sup>2</sup>    **Urban Land Use:** 0.174%

**Agricultural Land Use:** 0%

### Human and Agricultural Census Data

**Population:** 0    **Cattle and Calves:** 7.67    **Hogs:** 0    **Poultry:** 19.9  
**Sheep:** 4.64    **Goats:** 3.06    **Horses:** 16.9    **Turkeys:** 0.562

### Industrial Dischargers

**NPDES Major Dischargers:** 0    **EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 47.6    **P applied as fertilizer to farms:** 7.74  
**N applied as fertilizer to non-farms:** 25.8    **P applied as fertilizer to non-farms:** 3.13  
**N produced from manure in CAFOs:** 6.83    **P produced from manure in CAFOs:** 1.32  
**N produced from manure by unconfined animals:** 3.45  
**P produced from manure by unconfined animals:** 0.793  
**Atmospheric deposition of total organic N:** 6,377

### Pesticide Usage Summary (kg)

**Total:** 0    **Fungicides:** 0    **Fumigants:** 0    **Herbicides:** 0    **Insecticides:** 0    **Others:** 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 3.01	4.01	10.4	53.0	7.80	2.76

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
0.354	0.345	0.204	4.84	1.93

*Major Anions (mg/L, filtered)*

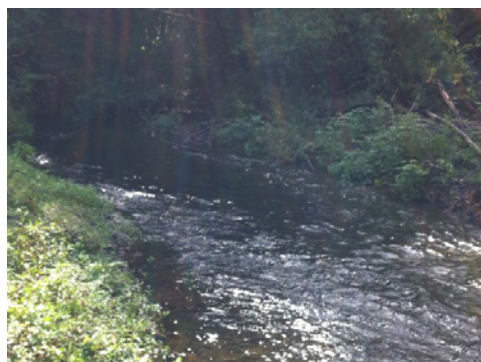
<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
E 0.026	3.75	<0.040	6.46

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
15.9	0.346	<0.004

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.369	0.055	<0.300	<0.700
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
1.28	0.625	0.092	<0.060	0.056



## Rio Bairoa at mouth, PR

### Site Information

**Station ID:** 50055410

**Site Type:** Routine

**Sampling Date:** March 19, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 7.70 mi<sup>2</sup>

**Urban Land Use:** 27.2%

**Agricultural Land Use:** 1.05%

### Human and Agricultural Census Data

**Population:** 2,733

**Cattle and Calves:** 375

**Hogs:** 97.2

**Poultry:** 11,487

**Sheep:** 38.9

**Goats:** 10.7

**Horses:** 11.3

**Turkeys:** NA

### Industrial Dischargers

**NPDES Major Dischargers:** 3

**EPA TRI Facilities:** 3

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** NA

**P applied as fertilizer to farms:** NA

**N applied as fertilizer to non-farms:** NA

**P applied as fertilizer to non-farms:** NA

**N produced from manure in CAFOs:** NA

**P produced from manure in CAFOs:** NA

**N produced from manure by unconfined animals:** NA

**P produced from manure by unconfined animals:** NA

**Atmospheric deposition of total organic N:** NA

### Pesticide Usage Summary (kg)

**Total:** NA

**Fungicides:** NA

**Fumigants:** NA

**Herbicides:** NA

**Insecticides:** NA

**Others:** NA

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 2.23	7.78	27.4	541	8.40	25.5

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
11.0	4.75	7.14	22.5	28.0

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.239	56.7	NA	39.3

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
5.17	4.90	0.310

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.466	<0.016	<0.300	1.29
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.141	0.432	0.130	<0.060	<0.014





## Rio Fajardo below Fajardo WWTP Outfall, PR

### Site Information

Station ID: 181847065391301  
Sampling Date: March 25, 2013

Site Type: Routine  
Gaging Station: No

### Basin Characteristics

Drainage Area: 20.4 mi<sup>2</sup>      Urban Land Use: 4.20%  
Agricultural Land Use: 2.36%

### Human and Agricultural Census Data

Population: 300      Cattle and Calves: 1,672      Hogs: 0.330      Poultry: 205  
Sheep: 43.8      Goats: 9.78      Horses: 44.9      Turkeys: NA

### Industrial Dischargers

NPDES Major Dischargers: NA      EPA TRI Facilities: NA

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: NA      P applied as fertilizer to farms: NA  
N applied as fertilizer to non-farms: NA      P applied as fertilizer to non-farms: NA  
N produced from manure in CAFOs: NA      P produced from manure in CAFOs: NA  
N produced from manure by unconfined animals: NA  
P produced from manure by unconfined animals: NA  
Atmospheric deposition of total organic N: NA

### Pesticide Usage Summary (kg)

Total: NA      Fungicides: NA      Fumigants: NA      Herbicides: NA  
Insecticides: NA      Others: NA

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
<2.00	8.40	29.6	269	9.10	15.6

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
63.6	8.67	3.37	5.35	35.8

*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.115	34.4	0.120	11.0

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.09	1.69	0.390

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.927	<0.016	<0.300	<0.700
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
<0.040	<0.190	0.067	<0.060	0.091



## Rush Creek at Highway 303 near Arlington, TX

### Site Information

**Station ID:** 324305097101900

**Site Type:** Routine

**Sampling Date:** May 13, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 28.9 mi<sup>2</sup>

**Urban Land Use:** 57.1%

**Agricultural Land Use:** 2.27%

### Human and Agricultural Census Data

**Population:** 2,956

**Cattle and Calves:** 497

**Hogs:** 3.15

**Poultry:** 102

**Sheep:** 25.5

**Goats:** 51.6

**Horses:** 143

**Turkeys:** 2.32

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 3

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 19,117

**P applied as fertilizer to farms:** 2,372

**N applied as fertilizer to non-farms:** 212,147

**P applied as fertilizer to non-farms:** 31,331

**N produced from manure in CAFOs:** 2,572

**P produced from manure in CAFOs:** 567

**N produced from manure by unconfined animals:** 21,422

**P produced from manure by unconfined animals:** 6,408

**Atmospheric deposition of total organic N:** 25,748

### Pesticide Usage Summary (kg)

**Total:** 110

**Fungicides:** 2.08

**Fumigants:** 0

**Herbicides:** 99.6

**Insecticides:** 8.56

**Others:** 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 6.95	NA	NA	NA	NA	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
68.0	40.7	4.87	45.8	67.3

*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.283	60.1	0.438	163

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
6.91	0.704	0.040

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.495	7.04	0.449	21.9	15.0
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
7.29	26.8	0.862	0.123	4.26



## Sand Run Gulch at Highway 95 crossing near Parma, ID

### Site Information

**Station ID:** 13210360

**Site Type:** Routine

**Sampling Date:** June 18, 2013

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 79.5 mi<sup>2</sup>

**Urban Land Use:** 0.174%

**Agricultural Land Use:** 47.2%

### Human and Agricultural Census Data

**Population:** 28.6

**Cattle and Calves:** 13,889

**Hogs:** 85.6

**Poultry:** 1,386

**Sheep:** 168

**Goats:** 274

**Horses:** 446

**Turkeys:** 230

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 798,890

**P applied as fertilizer to farms:** 159,261

**N applied as fertilizer to non-farms:** 0

**P applied as fertilizer to non-farms:** 0

**N produced from manure in CAFOs:** 290,812

**P produced from manure in CAFOs:** 57,744

**N produced from manure by unconfined animals:** 386,039

**P produced from manure by unconfined animals:** 106,178

**Atmospheric deposition of total organic N:** 11,690

### Pesticide Usage Summary (kg)

**Total:** 73,870

**Fungicides:** 3,398

**Fumigants:** 50,012

**Herbicides:** 16,532

**Insecticides:** 3,836

**Others:** 91.7

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
59.2	NA	NA	NA	NA	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
41.8	12.3	4.23	43.9	41.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.076	14.4	0.470	41.8

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
4.95	3.67	0.519

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	8.39	0.0810	2.85	4.43
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
2.40	3.47	0.460	<0.060	7.32



## Santa Ana River below Prado Dam, CA

### Site Information

**Station ID:** 11074000

**Site Type:** Routine

**Sampling Date:** March 25, 2014

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 2,258 mi<sup>2</sup>

**Urban Land Use:** 23.5%

**Agricultural Land Use:** 4.60%

### Human and Agricultural Census Data

**Population:** 1,342

**Cattle and Calves:** 20,490

**Hogs:** 247

**Poultry:** 769,585

**Sheep:** 6,270

**Goats:** 967

**Horses:** 2,419

**Turkeys:** 62.6

### Industrial Dischargers

**NPDES Major Dischargers:** 18

**EPA TRI Facilities:** 376

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 8,687,982

**P applied as fertilizer to farms:** 1,589,765

**N applied as fertilizer to non-farms:** 1,790,181

**P applied as fertilizer to non-farms:** 253,351

**N produced from manure in CAFOs:** 10,306,629

**P produced from manure in CAFOs:** 2,208,577

**N produced from manure by unconfined animals:** 1,825,114

**P produced from manure by unconfined animals:** 391,227

**Atmospheric deposition of total organic N:** 505,675

### Pesticide Usage Summary (kg)

**Total:** 98,499

**Fungicides:** 36,213

**Fumigants:** 20,836

**Herbicides:** 28,054

**Insecticides:** 6,936

**Others:** 6,462

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
5.00	7.50	17.5	1,050	10.0	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
84.1	19.4	14.6	17.8	102



*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.218	136	0.393	99.1

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
11.8	2.32	1.60

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.438	3.56	0.072	0.835	2.22
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.598	2.56	0.300	<0.060	4.73



## Sope Creek near Marietta, GA

### Site Information

Station ID: 02335870      Site Type: Routine  
Sampling Date: April 16, 2013      Gaging Station: Yes

### Basin Characteristics

Drainage Area: 30.8 mi<sup>2</sup>  
Urban Land Use: 38.6%      Agricultural Land Use: 0.619%

### Human and Agricultural Census Data

Population: 2,340      Cattle and Calves: 34.3      Hogs: 1.07      Poultry: 103  
Sheep: 0      Goats: 45.6      Horses: 33.7      Turkeys: 0

### Industrial Dischargers

NPDES Major Dischargers: 0      EPA TRI Facilities: 12

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 0      P applied as fertilizer to farms: 0  
N applied as fertilizer to non-farms: 4,764      P applied as fertilizer to non-farms: 606  
N produced from manure in CAFOs: 0      P produced from manure in CAFOs: 0  
N produced from manure by unconfined animals: 0  
P produced from manure by unconfined animals: 0  
Atmospheric deposition of total organic N: 26,365

### Pesticide Usage Summary (kg)

Total: 27.1      Fungicides: 3.41      Fumigants: 0      Herbicides: 20.9  
Insecticides: 2.75      Others: 0.005

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
E 3.31	6.50	16.0	98.4	9.10	34.0

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
8.66	2.45	1.85	12.4	5.51

***Major Anions (mg/L, filtered)***

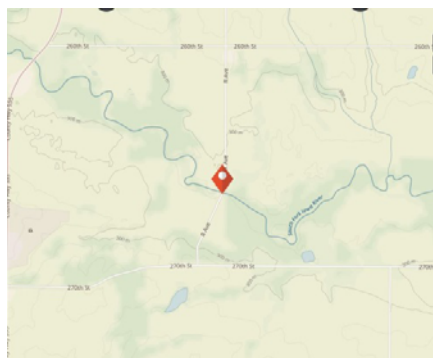
<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.053	7.25	E 0.051	4.70

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.29	0.552	0.012

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	<0.280	<0.016	<0.300	1.16
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.200	0.442	0.088	<0.060	0.017



## South Fork Iowa River near New Providence, IA

### Site Information

**Station ID:** 05451210 **Site Type:** Routine

**Sampling Date:** May 20, 2013 **Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 266 mi<sup>2</sup> **Urban Land Use:** 1.29%

**Agricultural Land Use:** 88.4%

### Human and Agricultural Census Data

**Population:** 7.96 **Cattle and Calves:** 7,233 **Hogs:** 316,939 **Poultry:** 58,941  
**Sheep:** 519 **Goats:** 66.5 **Horses:** 244 **Turkeys:** 183,129

### Industrial Dischargers

**NPDES Major Dischargers:** 0 **EPA TRI Facilities:** 1

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 4,746,645 **P applied as fertilizer to farms:** 674,899  
**N applied as fertilizer to non-farms:** 2,676 **P applied as fertilizer to non-farms:** 403  
**N produced from manure in CAFOs:** 3,368,229 **P produced from manure in CAFOs:** 1,429,830  
**N produced from manure by unconfined animals:** 322,785  
**P produced from manure by unconfined animals:** 140,771  
**Atmospheric deposition of total organic N:** 357,074

### Pesticide Usage Summary (kg)

**Total:** 121,120 **Fungicides:** 1,838 **Fumigants:** 0 **Herbicides:** 114,392  
**Insecticides:** 4,890 **Others:** 0.103

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
< 2.00	8.11	18.5	665	9.41	335

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
86.7	27.0	2.23	8.89	6.25

*Major Anions (mg/L, filtered)*

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.027	26.5	0.309	24.5

*Nutrients (mg/L, unfiltered)*

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
11.3	17.9	0.279

*Selected Trace Metals (µg/L, unfiltered)*

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.692	2.22	0.040	1.31	2.70
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
1.23	2.89	2.96	<0.060	3.69



## South Fork Zumbro River below WWTP nr Rochester MN

### Site Information

**Station ID:** 05373005

**Site Type:** Routine

**Sampling Date:** December 5, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 312 mi<sup>2</sup>

**Urban Land Use:** 8.52%

**Agricultural Land Use:** 62.3%

### Human and Agricultural Census Data

**Population:** 317

**Cattle and Calves:** 18,742

**Hogs:** 47,894

**Poultry:** 840

**Sheep:** 441

**Goats:** 147

**Horses:** 581

**Turkeys:** 51,649

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 15

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 3,943,059

**P applied as fertilizer to farms:** 741,267

**N applied as fertilizer to non-farms:** 98,439

**P applied as fertilizer to non-farms:** 14,010

**N produced from manure in CAFOs:** 993,688

**P produced from manure in CAFOs:** 285,425

**N produced from manure by unconfined animals:** 405,228

**P produced from manure by unconfined animals:** 130,969

**Atmospheric deposition of total organic N:** 396,885

### Pesticide Usage Summary (kg)

**Total:** 88,250

**Fungicides:** 3,280

**Fumigants:** 16.3

**Herbicides:** 81,043

**Insecticides:** 3,910

**Others:** 0.965

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
E 3.65	NA	NA	NA	NA	31.3

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
87.6	24.2	6.84	9.79	86.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.141	140	0.437	41.2

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
3.96	10.4	0.351

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.606	0.683	0.045	<0.300	4.34
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.160	1.14	0.367	<0.060	0.678





## South Platte River near Commerce, CO

### Site Information

**Station ID:** 394847104570700

**Site Type:** Routine

**Sampling Date:** December 11, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 4,082 mi<sup>2</sup>

**Urban Land Use:** 7.46%

**Agricultural Land Use:** 0.575%

### Human and Agricultural Census Data

**Population:** 436

**Cattle and Calves:** 20,729

**Hogs:** 268

**Poultry:** 6,824

**Sheep:** 897

**Goats:** 1,450

**Horses:** 9,286

**Turkeys:** 292

### Industrial Dischargers

**NPDES Major Dischargers:** 21

**EPA TRI Facilities:** 193

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 323,369

**P applied as fertilizer to farms:** 44,688

**N applied as fertilizer to non-farms:** 355,531

**P applied as fertilizer to non-farms:** 73,598

**N produced from manure in CAFOs:** 102,298

**P produced from manure in CAFOs:** 24,774

**N produced from manure by unconfined animals:** 1,103,361

**P produced from manure by unconfined animals:** 308,674

**Atmospheric deposition of total organic N:** 2,058,164

### Pesticide Usage Summary (kg)

**Total:** 8,852

**Fungicides:** 33.2

**Fumigants:** 19.9

**Herbicides:** 7,799

**Insecticides:** 999

**Others:** 0.698

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 4.05	7.00	18.3	858	NA	186

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
4.33	2.07	14.5	14.0	3.73

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
NA	93.6	0.902	116

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
14.9	17.7	1.62

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	<0.280	<0.030	0.620	1.61
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.322	0.532	<0.100	<0.060	0.022



## Sunrise River Tributary near Lindstrom, MN

### Site Information

**Station ID:** 05340041

**Site Type:** Routine

**Sampling Date:** December 4, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 6.60 mi<sup>2</sup>

**Urban Land Use:** 36.5%

**Agricultural Land Use:** 22.4%

### Human and Agricultural Census Data

**Population:** 1,194

**Cattle and Calves:** 151

**Hogs:** 8.39

**Poultry:** 24.8

**Sheep:** 6.84

**Goats:** 3.34

**Horses:** 16.0

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 1

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 27,587

**P applied as fertilizer to farms:** 5,186

**N applied as fertilizer to non-farms:** 5,588

**P applied as fertilizer to non-farms:** 795

**N produced from manure in CAFOs:** 5,197

**P produced from manure in CAFOs:** 1,154

**N produced from manure by unconfined animals:** 3,904

**P produced from manure by unconfined animals:** 1,221

**Atmospheric deposition of total organic N:** 7,988

### Pesticide Usage Summary (kg)

**Total:** 405

**Fungicides:** 15.2

**Fumigants:** 10.6

**Herbicides:** 352

**Insecticides:** 27.6

**Others:** 0.060

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 2.53	NA	NA	NA	NA	1.05

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
48.5	15.6	15.0	16.1	127

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.101	220	1.04	25.5

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
8.15	4.44	0.313

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.275	0.613	0.035	<0.300	2.81
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.160	4.01	0.099	<0.060	0.101



## Swiftcurrent Creek above Swiftcurrent Lake at Many Glacier, MT

### Site Information

Station ID: 05014300

Sampling Date: August 28, 2013

Site Type: Routine

Gaging Station: Yes

### Basin Characteristics

Drainage Area: 15.8 mi<sup>2</sup>

Agricultural Land Use: 0.070%

Urban Land Use: 0%

### Human and Agricultural Census Data

Population: 0

Cattle and Calves: 241

Hogs: 111

Poultry: 591

Sheep: 7.74

Goats: 0.188

Horses: 27.3

Turkeys: 2.60

### Industrial Dischargers

NPDES Major Dischargers: 0

EPA TRI Facilities: 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

N applied as fertilizer to farms: 0

P applied as fertilizer to farms: 0

N applied as fertilizer to non-farms: 0

P applied as fertilizer to non-farms: 0

N produced from manure in CAFOs: 0

P produced from manure in CAFOs: 0

N produced from manure by unconfined animals: 2,837

P produced from manure by unconfined animals: 958

Atmospheric deposition of total organic N: 14,451

### Pesticide Usage Summary (kg)

Total: 2.76

Fungicides: 0.010

Fumigants: 0

Herbicides: 2.68

Insecticides: 0.070

Others: 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

Turbidity (NTU)	pH (Standard Units)	Water temperature (degrees Celsius)	Specific conductance (microsiemens per centimeter)	Dissolved oxygen (milligrams per liter)	Discharge (cubic feet per second)
<2.00	NA	13.2	111	8.48	23.5

#### *Major Cations (mg/L, filtered)*

Calcium	Magnesium	Potassium	Silica	Sodium
14.3	4.42	0.118	1.61	0.421

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.010	<0.060	0.021	2.54

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
1.20	<0.050	<0.004

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	0.470	<0.016	<0.300	<0.700
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
<0.040	<0.190	<0.050	<0.060	0.142



## Sycamore Slough at County Line Rd. nr Dunnigan, CA

### Site Information

**Station ID:** 385531121532001

**Sampling Date:** June 18, 2013

**Site Type:** Routine

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 64.3 mi<sup>2</sup>

**Urban Land Use:** 2.64%

**Agricultural Land Use:** 95.0%

### Human and Agricultural Census Data

**Population:** 7.22

**Cattle and Calves:** 820

**Hogs:** 32.6

**Poultry:** 13.9

**Sheep:** 105

**Goats:** 37.0

**Horses:** 27.8

**Turkeys:** 0

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 1

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 1,967,398

**P applied as fertilizer to farms:** 360,003

**N applied as fertilizer to non-farms:** 145

**P applied as fertilizer to non-farms:** 20.5

**N produced from manure in CAFOs:** 16,633

**P produced from manure in CAFOs:** 3,195

**N produced from manure by unconfined animals:** 63,666

**P produced from manure by unconfined animals:** 15,684

**Atmospheric deposition of total organic N:** 25,977

### Pesticide Usage Summary (kg)

**Total:** 103,584

**Fungicides:** 36,067

**Fumigants:** 5,410

**Herbicides:** 56,044

**Insecticides:** 5,701

**Others:** 362

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
128	7.60	20.9	525	4.99	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
49.5	10.2	1.50	7.98	94.8

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.054	24.0	0.267	52.8

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
7.80	1.42	0.390

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.328	<0.280	0.287	0.555	14.3
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.492	2.44	0.859	<0.060	0.768





## Tembladero Slough at Castroville, CA

### Site Information

**Station ID:** 364554121453401  
**Sampling Date:** March 3, 2014

**Site Type:** Routine  
**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 154 mi<sup>2</sup>      **Urban Land Use:** 13.7%  
**Agricultural Land Use:** 26.7%

### Human and Agricultural Census Data

**Population:** 977      **Cattle and Calves:** 2,772      **Hogs:** 3.05      **Poultry:** 154  
**Sheep:** 145      **Goats:** 32.6      **Horses:** 94.8      **Turkeys:** 20.5

### Industrial Dischargers

**NPDES Major Dischargers:** 1      **EPA TRI Facilities:** 5

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 7,938,013      **P applied as fertilizer to farms:** 1,452,532  
**N applied as fertilizer to non-farms:** 52,834      **P applied as fertilizer to non-farms:** 7,477  
**N produced from manure in CAFOs:** 56,335      **P produced from manure in CAFOs:** 12,365  
**N produced from manure by unconfined animals:** 145,736  
**P produced from manure by unconfined animals:** 43,783  
**Atmospheric deposition of total organic N:** 18,156

### Pesticide Usage Summary (kg)

**Total:** 367,794      **Fungicides:** 96,293      **Fumigants:** 191,760      **Herbicides:** 20,664  
**Insecticides:** 57,440      **Others:** 1,636

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
311	6.3	13.5	970	7.10	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
50.8	23.1	7.84	7.52	63.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.750	123	0.290	115

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
NA	22.0	1.46

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.264	0.613	0.030	0.426	1.37
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.164	7.98	0.216	<0.060	0.270



## Trinity River below Dallas, TX

### Site Information

**Station ID:** 08057410

**Site Type:** Routine

**Sampling Date:** December 4, 2012

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 6,265 mi<sup>2</sup>

**Urban Land Use:** 15.4%

**Agricultural Land Use:** 14.1%

### Human and Agricultural Census Data

**Population:** 696

**Cattle and Calves:** 302,252

**Hogs:** 2,517

**Poultry:** 31,419

**Sheep:** 11,954

**Goats:** 25,992

**Horses:** 39,412

**Turkeys:** 1,087

### Industrial Dischargers

**NPDES Major Dischargers:** 30

**EPA TRI Facilities:** 654

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 17,516,024

**P applied as fertilizer to farms:** 2,172,975

**N applied as fertilizer to non-farms:** 8,199,129

**P applied as fertilizer to non-farms:** 1,210,889

**N produced from manure in CAFOs:** 3,509,017

**P produced from manure in CAFOs:** 850,390

**N produced from manure by unconfined animals:** 17,074,765

**P produced from manure by unconfined animals:** 4,925,944

**Atmospheric deposition of total organic N:** 5,738,168

### Pesticide Usage Summary (kg)

**Total:** 119,202

**Fungicides:** 4,373

**Fumigants:** 0

**Herbicides:** 92,370

**Insecticides:** 21,914

**Others:** 545

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 12.4	7.26	21.7	747	8.70	NA

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
54.4	7.27	14.2	7.35	81.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
E 0.221	91.0	0.698	89.5

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
8.32	17.0	1.92

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
0.464	2.11	0.046	0.713	3.57
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.935	5.39	0.189	<0.060	0.215



## West Branch Delaware River at South Kortright, NY

### Site Information

**Station ID:** 01421640

**Site Type:** Routine

**Sampling Date:** November 19, 2012

**Gaging Station:** No

### Basin Characteristics

**Drainage Area:** 48.2 mi<sup>2</sup>

**Urban Land Use:** 0.672%

**Agricultural Land Use:** 34.8%

### Human and Agricultural Census Data

**Population:** 63.1

**Cattle and Calves:** 782

**Hogs:** 37.2

**Poultry:** 221

**Sheep:** 43.3

**Goats:** 31.1

**Horses:** 35.0

**Turkeys:** 11.8

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 45,826

**P applied as fertilizer to farms:** 7,500

**N applied as fertilizer to non-farms:** 2,956

**P applied as fertilizer to non-farms:** 394

**N produced from manure in CAFOs:** 84,260

**P produced from manure in CAFOs:** 15,620

**N produced from manure by unconfined animals:** 41,322

**P produced from manure by unconfined animals:** 9,974

**Atmospheric deposition of total organic N:** 59,837

### Pesticide Usage Summary (kg)

**Total:** 1,198

**Fungicides:** 98.6

**Fumigants:** 27.1

**Herbicides:** 907

**Insecticides:** 158

**Others:** 6.40

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
< 2.00	7.58	2.72	129	14.1	42.1

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
10.1	1.91	1.22	3.52	9.45

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.0175	15.0	<0.040	7.78

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
2.40	0.930	0.017

***Selected Trace Metals (µg/L, unfiltered):***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
< 0.180	0.423	< 0.016	< 0.300	0.917
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.115	0.216	0.051	< 0.060	0.035



## West Clear Creek near Camp Verde, AZ

### Site Information

**Station ID:** 09505800

**Site Type:** Routine

**Sampling Date:** January 15, 2014

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 241 mi<sup>2</sup>

**Watershed Stream Length:** 449 mi

**Urban Land Use:** 0.008%

**Agricultural Land Use:** 0%

### Human and Agricultural Census Data

**Population:** 0.07

**Cattle and Calves:** 713

**Hogs:** 2.69

**Poultry:** 41.5

**Sheep:** 537

**Goats:** 142

**Horses:** 130

**Turkeys:** 3.79

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 0

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 0

**P applied as fertilizer to farms:** 0

**N applied as fertilizer to non-farms:** 6.40

**P applied as fertilizer to non-farms:** 0.980

**N produced from manure in CAFOs:** 0

**P produced from manure in CAFOs:** 0

**N produced from manure by unconfined animals:** 1,948

**P produced from manure by unconfined animals:** 548

**Atmospheric deposition of total organic N:** 164,070

### Pesticide Usage Summary (kg)

**Total:** 0

**Fungicides:** 0

**Fumigants:** 0

**Herbicides:** 0

**Insecticides:** 0

**Others:** 0

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
< 2.00	8.2	6.55	381	10.8	21

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
42.5	24.0	1.10	15.1	5.92

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
<0.030	3.36	0.089	2.29

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
1.24	0.058	0.006

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	2.17	<0.030	1.40	<0.800
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
<0.040	<0.200	0.199	<0.060	0.625





## Zollner Creek near Mt. Angel, OR

### Site Information

**Station ID:** 14201300

**Site Type:** Routine

**Sampling Date:** May 19, 2014

**Gaging Station:** Yes

### Basin Characteristics

**Drainage Area:** 15.0 mi<sup>2</sup>

**Urban Land Use:** 5.05%

**Agricultural Land Use:** 88.0%

### Human and Agricultural Census Data

**Population:** 72.0

**Cattle and Calves:** 506

**Hogs:** 14.6

**Poultry:** 0

**Sheep:** 109

**Goats:** 25.2

**Horses:** 37.1

**Turkeys:** 11.5

### Industrial Dischargers

**NPDES Major Dischargers:** 0

**EPA TRI Facilities:** 1

### Nitrogen (N) and Phosphorus (P) Sources (kg)

**N applied as fertilizer to farms:** 776,812

**P applied as fertilizer to farms:** 119,888

**N applied as fertilizer to non-farms:** 229

**P applied as fertilizer to non-farms:** 16.5

**N produced from manure in CAFOs:** 41,610

**P produced from manure in CAFOs:** 9,052

**N produced from manure by unconfined animals:** 31,073

**P produced from manure by unconfined animals:** 6,611

**Atmospheric deposition of total organic N:** 4,834

### Pesticide Usage Summary (kg)

**Total:** 14,625

**Fungicides:** 1,221

**Fumigants:** 9,229

**Herbicides:** 2,858

**Insecticides:** 1,309

**Others:** 7.01

### Physical Parameters and Chemical Constituents

#### *Physical Parameters (in units listed)*

<b>Turbidity (NTU)</b>	<b>pH (Standard Units)</b>	<b>Water temperature (degrees Celsius)</b>	<b>Specific conductance (microsiemens per centimeter)</b>	<b>Dissolved oxygen (milligrams per liter)</b>	<b>Discharge (cubic feet per second)</b>
E 12.8	7.16	15.6	306	8.04	20

#### *Major Cations (mg/L, filtered)*

<b>Calcium</b>	<b>Magnesium</b>	<b>Potassium</b>	<b>Silica</b>	<b>Sodium</b>
30.0	10.2	3.73	18.7	12.2

***Major Anions (mg/L, filtered)***

<b>Bromide</b>	<b>Chloride</b>	<b>Fluoride</b>	<b>Sulfate</b>
0.053	27.8	0.107	16.5

***Nutrients (mg/L, unfiltered)***

<b>Carbon (organic)</b>	<b>Nitrogen (total)</b>	<b>Phosphorus (total)</b>
5.30	6.54	0.243

***Selected Trace Metals (µg/L, unfiltered)***

<b>Antimony</b>	<b>Arsenic</b>	<b>Cadmium</b>	<b>Chromium</b>	<b>Copper</b>
<0.180	1.11	<0.030	0.869	2.74
<b>Lead</b>	<b>Nickel</b>	<b>Selenium</b>	<b>Thallium</b>	<b>Uranium</b>
0.366	1.86	<0.100	<0.060	0.081



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