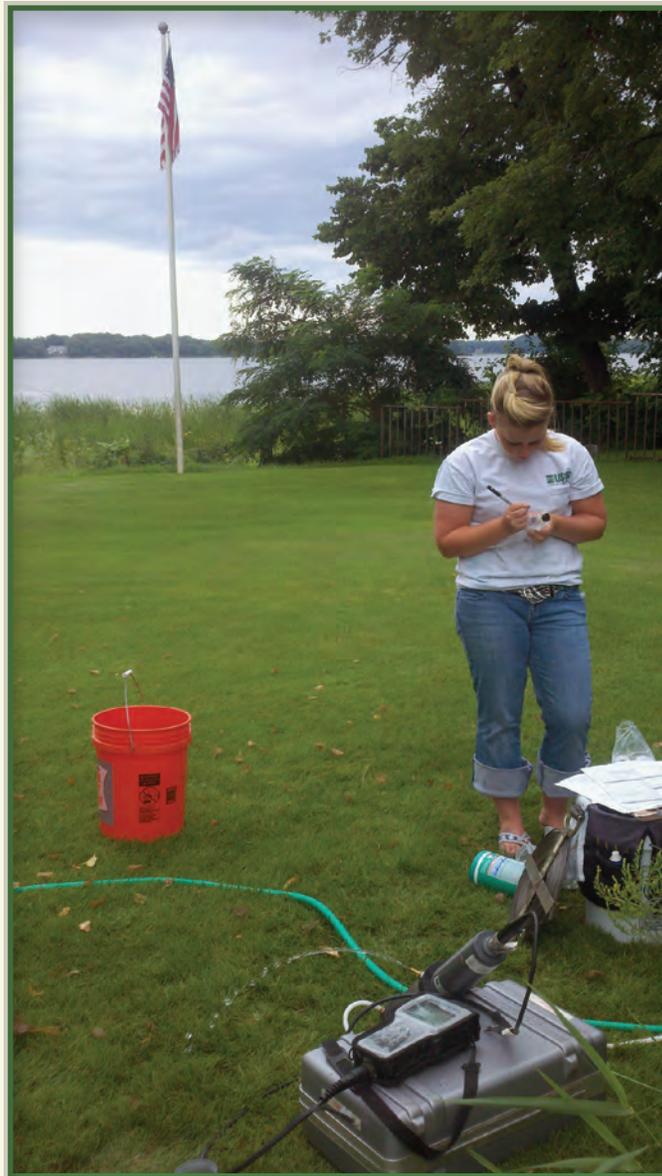


Prepared in cooperation with the Minnesota Pollution Control Agency

# Contaminants of Emerging Concern in Ambient Groundwater in Urbanized Areas of Minnesota, 2009–12



Scientific Investigations Report 2014–5096  
Version 1.2, September 2014

**Cover photograph.** Water-level and water quality measurement in a domestic well, White Bear Lake, Minnesota, 2011. Photograph by Perry M. Jones, U.S. Geological Survey.

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By Melinda L. Erickson, Susan K. Langer, Jason L. Roth, and Sharon E. Kroening

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Version 1.2, September 2014

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

**U.S. Department of the Interior**  
SALLY JEWELL, Secretary

**U.S. Geological Survey**  
Suzette M. Kimball, Acting Director

U.S. Geological Survey, Reston, Virginia: 2014  
First release: 2014  
Revised: September 2014 (ver. 1.2)

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Suggested citation:

Erickson, M.L., Langer, S.K., Roth, J.L., and Kroening, S.E., 2014, Contaminants of emerging concern in ambient groundwater in urbanized areas of Minnesota, 2009–12 (ver. 1.2, September 2014): U.S. Geological Survey Scientific Investigations Report 2014–5096, 38 p., with appendix, <http://dx.doi.org/10.3133/sir20145096>.

ISSN 2328-0328 (online)

## Acknowledgments

Minnesota Pollution Control Agency (MPCA) hydrologists David Duffey, Sharon Kroening, and Gerald Flom, and MPCA student workers Mark Lunda, Meaghan McGinn, and Sophia Vaughan collected the groundwater samples for this study. In November 2008, Minnesota voters approved a three-eighths of 1 percent increase in the State sales tax rate under the Clean Water, Land and Legacy Amendment. Approximately 33 percent of the funds were dedicated to a Clean Water Fund to protect, enhance, and restore water quality in lakes, rivers, streams, and groundwater. Funding for this study was provided by the State of Minnesota's Clean Water Fund and the U.S. Geological Survey's Cooperative Water Program.



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## Conversion Factors

Inch/Pound to SI

<b>Multiply</b>	<b>By</b>	<b>To obtain</b>
	Length	
foot (ft)	0.3048	meter (m)

SI to Inch/Pound (used for analytical methods)

<b>Multiply</b>	<b>By</b>	<b>To obtain</b>
	Length	
centimeter (cm)	0.3937	inch (in.)
	Volume	
liter (L)	1.057	quart (qt)
liter (L)	0.2642	gallon (gal)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
milligram (mg)	0.00003527	ounce, avoirdupois (oz)

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

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

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

Concentrations of chemical constituents in water are given in milligrams per liter (mg/L), micrograms per liter (µg/L), or nanograms per liter (ng/L).

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius (µS/cm at 25 °C).

## Abbreviations

CEC	contaminant of emerging concern
DEET	<i>N,N</i> -diethyl-meta-toluamide
DO	dissolved oxygen
E	estimated
EAC	endocrine active chemical
EPA	U.S. Environmental Protection Agency
FYROL CEF	Tris(2-chloroethyl) phosphate
GIS	geographic information system
HBSL	Health-Based Screening Levels
HBV	Health-Based Value (Minnesota Department of Health)
HHCB	hexahydrohexamethylcyclopentabenzopyran
HRL	Health Risk Limit (Minnesota Department of Health)
IDS	isotope dilution standard
MCL	Maximum Contaminant Level (U.S. Environmental Protection Agency)
MDH	Minnesota Department of Health
MPCA	Minnesota Pollution Control Agency
NFM	National Field Manual for the Collection of Water-Quality Data (U.S. Geological Survey)
NLCD	National Land Cover Dataset
NWQL	National Water Quality Laboratory (U.S. Geological Survey)
OGRL	Organic Chemistry Research Laboratory (U.S. Geological Survey)
PCFF	Personal Computer Field Forms (U.S. Geological Survey)
PhAC	pharmaceutically active compound
®	registered trademark
SSTS	subsurface sewage treatment systems for wastewater disposal
TCMA	Twin Cities Metropolitan area
USGS	U.S. Geological Survey



# Contaminants of Emerging Concern in Ambient Groundwater in Urbanized Areas of Minnesota, 2009–12

By Melinda L. Erickson,<sup>1</sup> Susan K. Langer,<sup>1</sup> Jason L. Roth,<sup>1</sup> and Sharon E. Kroening<sup>2</sup>

## Abstract

A study of contaminants of emerging concern (CECs) in ambient groundwater in urbanized areas of Minnesota was completed by the U.S. Geological Survey in cooperation with the Minnesota Pollution Control Agency. For this study, water samples were collected from November 2009 through June 2012 from 118 wells located in different land-use settings. The sampled wells primarily were screened in vulnerable sand and gravel aquifers (surficial and buried glacial aquifers) or vulnerable bedrock aquifers such as the Prairie du Chien-Jordan aquifer. Sampled well depths ranged from 9 to 285 feet below land surface. Water samples were collected by Minnesota Pollution Control Agency staff. The water samples were analyzed at U.S. Geological Survey laboratories for steroidal hormones, human-use pharmaceutical compounds, human- and animal-use antibiotics, and a broad suite of organic chemicals associated with wastewater. Reported detections were censored and not counted as detections in the data analyses if the chemical was detected in a laboratory or field blank at a similar concentration.

During this study, 38 out of 127 CECs analyzed were detected among all water samples collected. Three of the detected CECs, however, were analyzed using two different analytical methods, so 35 distinct chemicals were detected. The number of detections of CECs in individual water samples ranged from 0 to 10. The three wells in proximity to landfills had the most CEC detections. One or more CECs were detected in a total of 43 samples (35 percent); no CECs were detected in 80 samples.

Of the 127 CECs included for analysis in this study, 28 have established enforceable or non-enforceable health-based water-quality standards or benchmarks. Fourteen of the 35 chemicals detected in this study have established water-quality standards, whereas 21 of the chemicals detected have no established standard or benchmark. All detections in this study were less than established health-based water-quality standards, although *p*-cresol was detected at a concentration nearing a health-based water quality standard. Four of the six

most frequently detected chemicals—azithromycin, diphenhydramine, tributyl phosphate, and lincomycin—have no health-based water-quality standards or benchmarks.

The antibiotic sulfamethoxazole was the most frequently detected CEC, detected in a total of 14 of 123 samples (11.4 percent) by one or both analytical methods that include sulfamethoxazole as an analyte. Most (11 of 14, or 79 percent) of the detections of sulfamethoxazole were in samples from domestic wells or monitoring wells located in areas where septic systems or potentially leaking centralized sewers are prevalent. The chemical *N,N*-Diethyl-*meta*-toluamide (DEET) was detected at the highest concentration of any CEC, at 7.9 micrograms per liter. Bisphenol A was detected second most frequently of all chemicals. DEET and Bisphenol A were detected most frequently in wells in proximity to closed landfills. Samples from bedrock wells, most of which are drinking water wells that are deeper than glacial wells, had a higher percentage of wells with CEC detections compared to samples from wells completed in glacial aquifers. The higher dissolved oxygen concentrations and lower specific conductance for the bedrock wells sampled indicate shorter duration flow paths from the land surface to these wells than for wells completed in glacial aquifers.

## Introduction

Approximately 78 percent of the population of Minnesota uses groundwater as drinking water (Kenny and others, 2009). Groundwater also provides critical base flow to streams and rivers, and groundwater supports lake and wetland water levels (Winter and others, 1998; Rosenberry and LaBaugh, 2008). Groundwater flowing into streams, rivers, lakes, and wetlands also can transport natural and anthropogenic chemicals present in the groundwater into these surface-water bodies. It is, therefore, important to understand the quality and vulnerability of groundwater in Minnesota to anthropogenic contamination.

Monitoring is essential to evaluate the status of groundwater quality and determine whether the State of Minnesota has enough clean groundwater to meet its citizens' needs. Monitoring the amount of chemicals in the State's groundwater helps policy-makers gage whether land-use practices

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

<sup>2</sup>Minnesota Pollution Control Agency.

degrade water quality and whether practices put in place to minimize groundwater contamination are working.

Contaminants of emerging concern (CECs; also called chemicals of emerging concern or emerging contaminants in the literature) are chemicals that are not commonly monitored or regulated in the environment. Examples of CEC chemical classes include prescription and over-the-counter pharmaceuticals, fire retardants, pesticides, personal-care products, hormones, and detergents. Some CECs are human-made, some are naturally occurring, and some also are endocrine active chemicals (EACs). In animals, the effects of exposure to EACs include reduced reproductive success, reduced survival, altered sex ratios, occurrence of intersex, and developmental abnormalities (Streets and others, 2008). Human exposure to EACs may result in diminished intelligence, altered behavior and development, and decreased immunity to disease. EACs are capable of disrupting any of the many parts or the endocrine (hormone) system. Many EACs and pharmaceutically active compounds (PhACs) are designed to benefit humans or animals when ingested, for example the antibiotic sulfamethoxazole or the hormone ethynyl estradiol. Some EACs and PhACs may, however, have an unwanted biological effect, such as the feminization of male fish, in the environment (Halling-Sorenson and others, 1998). Recent studies have documented the presence of CECs, including EACs, in Minnesota surface water and groundwater (Lee and others, 2004; Tornes and others, 2007; Lee and others, 2008; Erickson, 2012) and nationwide (Zogorski and others, 2006; DeSimone and others, 2009; Toccalino and Hopple, 2010).

This study was designed to assess the occurrence and distribution of CECs in ambient groundwater in urbanized areas that are vulnerable to contamination from non-agricultural chemicals and was completed by the U.S. Geological Survey (USGS) in cooperation with the Minnesota Pollution Control Agency (MPCA).

## Purpose and Scope

The purpose of this report is to present an assessment on the presence of CECs in ambient groundwater in urbanized areas in Minnesota. The assessment was based on analyses of 127 CECs in samples of groundwater collected from November 2009 through June 2012 from 118 wells completed in sand and gravel aquifers (surficial and buried glacial aquifers) or bedrock aquifers such as the Prairie du Chien-Jordan aquifer located in different land-use setting throughout the State of Minnesota.

## Study Design

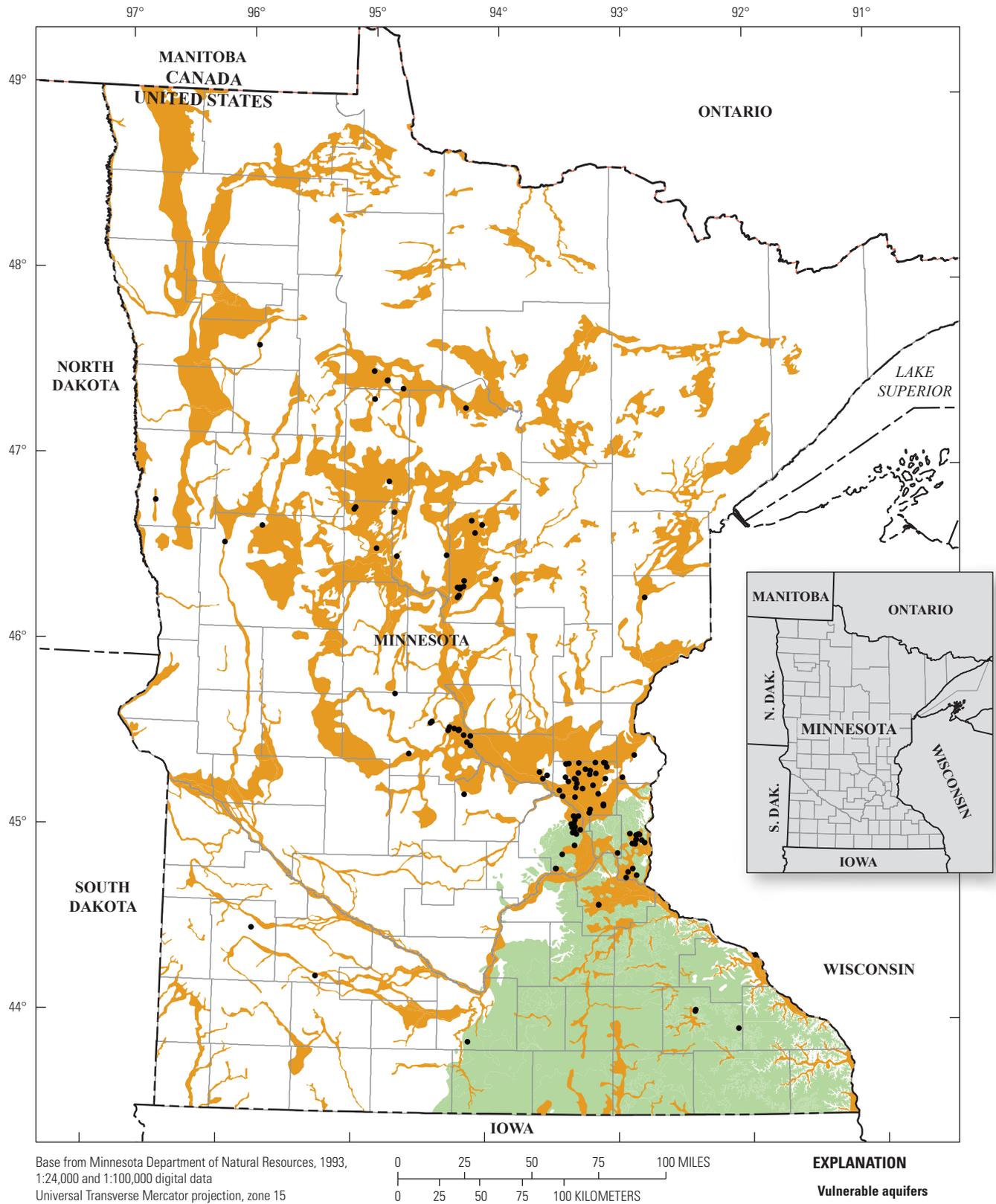
This study was designed to assess the occurrence and distribution of CECs in groundwater in urbanized areas that are vulnerable to contamination from non-agricultural chemicals. The study design was presented in Erickson (2012) and

Kroening (2012) and is presented again in this section of the report. The study used wells from the MPCA's ambient groundwater monitoring network to determine the occurrence of contamination from CECs in ambient groundwater in Minnesota. The broad suite of CECs analyzed in groundwater samples for this study included chemicals typically found in municipal wastewater: steroidal hormones, pharmaceuticals, antibiotics, and other organic chemicals.

The MPCA maintains an ambient groundwater monitoring network (hereinafter referred to as the "network") to determine if non-agricultural chemical contamination is present in the groundwater and to track any trends in contamination (Kroening, 2013). The MPCA monitoring network focuses on these specific chemical types to meet State and Federal authorities and requirements. The MPCA monitoring efforts complement its charge to minimize groundwater contamination from all other chemicals.

The MPCA network primarily is used to monitor groundwater expected to represent broad groundwater conditions and also is most likely to be contaminated with nonagricultural chemicals. The network consisted of about 200 wells as of 2013. Wells that are known to be or suspected to be affected by known point sources of contamination, such as leaking petroleum product storage tanks, Superfund sites, or landfills, are not part of the network (Kroening, 2012). Typically, shallow aquifers are sampled for this network because these aquifers tend to be the most vulnerable to contamination (Minnesota Department of Natural Resources, 2014). The network monitoring is targeted to wells completed in the vulnerable sand and gravel aquifers and vulnerable bedrock aquifers, such as the Prairie du Chien-Jordan aquifer (fig. 1), in areas that are sensitive to pollution, as described by the Minnesota Department of Natural Resources and others (Falteisek, 2013). The vulnerable parts of the aquifers contain water that infiltrated recently (that is, infiltration that fell as precipitation less than 10 years ago). Contamination caused by humans is more likely to quickly reach these vulnerable parts of the aquifers compared to the deeper, more protected aquifers, which generally are recharged by water that fell as precipitation several decades to centuries in the past (Minnesota Department of Natural Resources, 2014).

The wells in the MPCA network are used to discern the effect of urban land use on groundwater quality and constitute an "early warning system" of about 130 shallow wells placed near the water table in the sand and gravel aquifers. Most wells in the network contain water that was recharged into the groundwater within the past decade; the groundwater extracted from some of these wells is less than 1 year old (Kroening, 2013). The wells in the network are distributed among several different land-use settings to determine the effect these settings have on groundwater quality. The assessed land-use settings are (1) sewer residential, (2) septic residential, which are residential areas that use subsurface sewage treatment systems for wastewater disposal (SSTS; also known as septic systems), (3) commercial or industrial areas, and (4) undeveloped areas such as parkland or forests. The data collected from the wells



**Figure 1.** Areal extent of vulnerable aquifers targeted for groundwater sampling and wells sampled in the Minnesota Pollution Control Agency ambient groundwater monitoring network for this study, 2009–12.

in the undeveloped areas provide a baseline to assess the extent of any contamination from all other land-use settings.

Wells selected and sampled as part of the network were located within fairly homogeneous settings, in terms of land use/land cover. Existing wells installed to detect known or suspected contamination are not included in the network to eliminate any biases in the results from these locations. The selection of wells for inclusion in the network was accomplished using a two-level process. The first level was to use existing geographic information system (GIS) datasets and maps to define the characteristics surrounding the existing wells and determine suitable locations for newly installed monitoring wells. The second level of the process used field reconnaissance to verify the characteristics surrounding the well were consistent with the initial GIS assessment.

The land use/land cover contributing to groundwater-quality conditions at existing wells and any newly installed wells was defined using a 1,640-foot (ft; 500-meter) circular buffer. In many cases, neither the hydraulic characteristics nor the groundwater-flow directions in the aquifer in the immediate vicinity of the well were well known, and a 1,640-ft circular buffer has been reported to adequately define the characteristics surrounding monitoring wells in a wide range of hydrogeologic settings across the Nation (Koterba, 1998). Similar land use/land cover in 75 percent or more of the 1,640-ft circular buffer was used as a criterion to describe homogeneous settings.

Several sources of information for land use/land cover were used when evaluating existing wells for inclusion in the network and siting potential well locations. The 2001 National Land Cover Dataset (NLCD) (Homer and others, 2007) was used to delineate undeveloped and urbanized areas (Kroening, 2013). Urban land use is not designated as commercial/industrial, sewer, or as areas with septic systems in the NLCD data. The 2005 land-use data published by the Twin Cities Metropolitan Council were used to delineate commercial/industrial areas and urban areas on centralized sewerage systems maintained by the Metropolitan Council within the 7-county Twin Cities metropolitan area (TCMA) (Kroening, 2013). Maps published by individual municipalities were used to delineate urban land-use types outside of the TCMA, and residential areas on centralized sewerage systems not maintained by the Metropolitan Council. In areas outside of the TCMA, commercial/industrial areas were delineated in municipalities with populations greater than 10,000 people. Smaller localities likely did not contain a large enough commercial/industrial sector that could be contained within a 1,640-ft circular buffer.

MPCA staff, in consultation with USGS, selected a subset of about 40 ambient wells (generally less than 200-ft deep) per year primarily from the MPCA ambient groundwater monitoring network for sampling during the study. During 2009–12, 118 wells were sampled (figs. 1 and 2; table 1); 5 wells were sampled twice, so a total of 123 samples were collected.

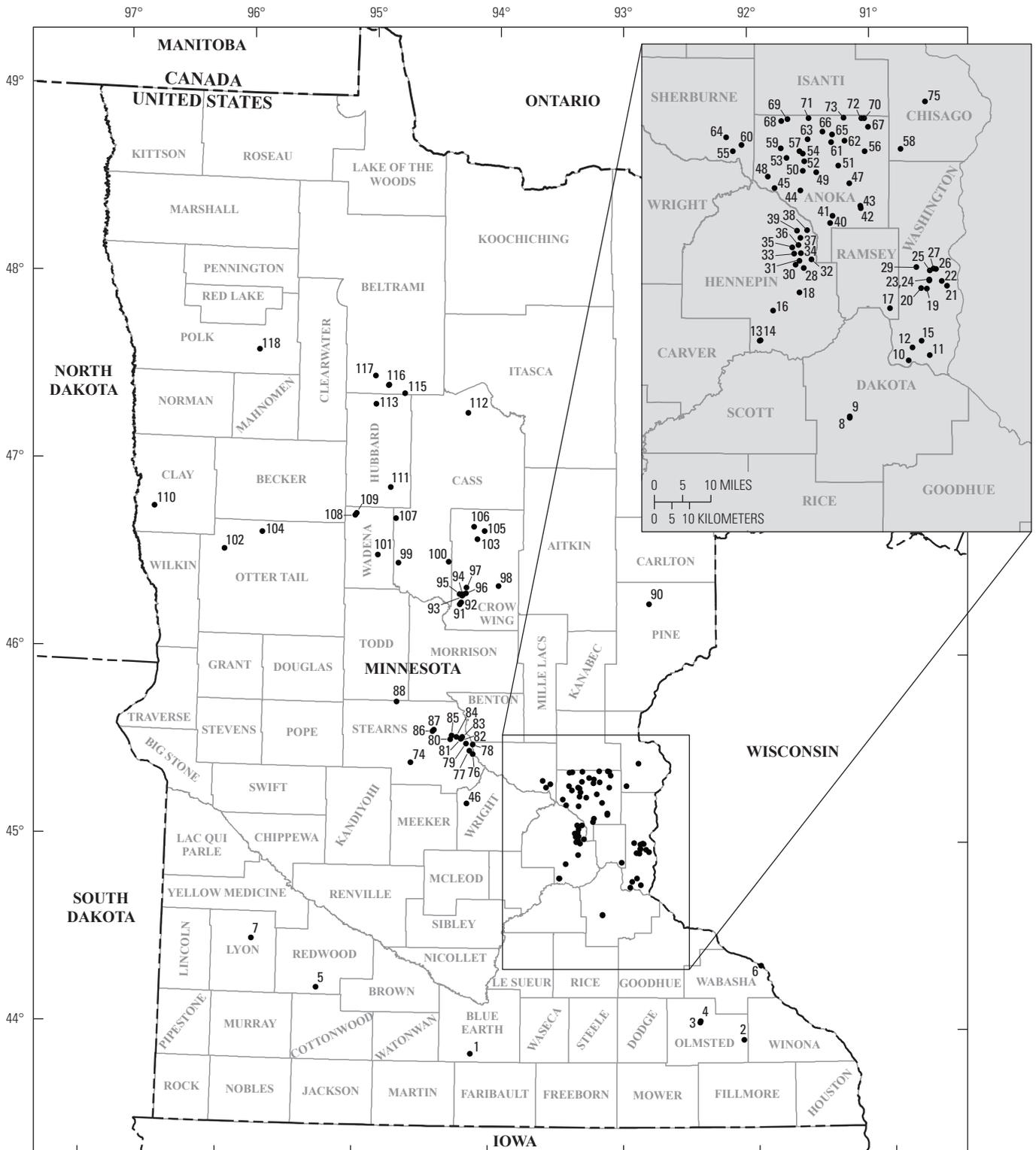
Three of the 118 wells sampled had been installed to monitor groundwater affected by landfill leachate (map

numbers 46, 54, and 65 on fig. 2, table 1) because landfill leachate has been identified as a source of CEC (Barnes and others, 2004; Andrews and others, 2011). All three landfills were unlined. None of the landfills were in operation at the time of sampling, and all of them ceased accepting waste 15–30 years before sample collection for this study. Two landfills had primarily accepted municipal waste, and the third landfill had primarily accepted demolition debris (Kroening, 2012).

Almost 70 percent of the wells sampled for this study were from the early warning network component of the MPCA network, which consisted mostly of wells completed in the surficial glacial aquifers. The remaining wells primarily were installed in deeper parts of the sand and gravel aquifers (buried glacial aquifers) or in vulnerable bedrock aquifers. The vulnerable bedrock aquifers from which samples were collected for this study (table 1), in descending order, are the St. Peter aquifer (contained in St. Peter Sandstone), Prairie du Chien-Jordan aquifer (contained in Prairie du Chien Group and Jordan Sandstone), and Franconia aquifer (contained in the Franconia Formation or the St. Lawrence and Franconia Formations). Information regarding these aquifers in Minnesota is provided by Adolphson and others (1981), Morey (1972), Runkel and others (2003), and Mossler (2008). Early warning network wells from all assessed land-use settings were included; however, the sampling was weighted towards wells located in unsewered (SSTS) and sewer residential areas.

Samples were collected from November 2009 through June 2012; most samples were collected during the months of April, May, and June. Sampled wells ranged from 9 to 285 ft deep. Bedrock wells (such as those completed in the Prairie du Chien-Jordan aquifer) generally were deeper (average of 188-ft deep) than wells screened in sand and gravel aquifers (glacial sediments; average of 30-ft deep). For wells screened in glacial sediments, monitoring wells were shallower (average of 23-ft deep) than domestic wells (average of 61-ft deep).

The water samples were analyzed for steroidal hormones, human-use pharmaceutical chemicals, human- and animal-use antibiotics, and a broad suite of organic chemicals associated with wastewater, as described in table 2, using published analytical methods (Foreman and others, 2012; Furlong and others, 2008; Meyer and others, 2007; and Zaugg and others, 2006). Samples were sent to the USGS National Water Quality Laboratory (NWQL) and the USGS Organic Geochemistry Research Laboratory (OGRL) (<http://ks.water.usgs.gov/research-lab>) for analysis. Analytical results for steroidal hormones in samples collected from October 2009 through June 2010 are presented in Erickson (2012) and are not reproduced in this report. These data were, however, included in the data analysis and assessment presented in this report.



Base from Minnesota Department of Natural Resources, 1993, 1:24,000 and 1:100,000 digital data  
 Universal Transverse Mercator projection, zone 15

0 25 50 75 100 MILES  
 0 25 50 75 100 KILOMETERS

**EXPLANATION**

● 2 Sampled well and number

Figure 2. Location of sampled wells, 2009–12.

**Table 1.** Selected information for wells sampled for this study.

[MUN, Minnesota unique well number; ft bgs, feet below ground surface; MPCA, Minnesota Pollution Control Agency; USGS, U.S. Geological Survey; MN040, Minnesota County Well Index]

Agency code	Station number	Map number (fig. 2)	MUN	Well type	Aquifer	Well depth (ft bgs)	MPCA-assigned land use or monitoring type
USGS	435328094080601	1	444696	Domestic	Prairie du Chien Group	128	Deep well
USGS	435809092063001	2	104953	Domestic	Prairie du Chien Group	268	Deep well
USGS	440349092255901	3	695881	Monitoring	St. Peter Sandstone	90	Deep well
USGS	440417092254501	4	220775	Domestic	Prairie du Chien Group	145	Deep well
MN040	441403095172101	5	189508	Domestic	Glacial buried sand and/or gravel	57	Deep well
USGS	442149091583501	6	474574	Domestic	Glacial surficial sand and/or gravel	58	Deep well
USGS	442913095465601	7	427800	Domestic	Glacial surficial sand and/or gravel	71	Deep well
USGS	443811093093301	8	639315	Monitoring	Glacial surficial sand and/or gravel	10	Sewered residential
USGS	443825093093401	9	639314	Monitoring	Glacial surficial sand and/or gravel	10	Sewered residential
MN040	444707092565201	10	131931	Domestic	Jordan Sandstone	270	Deep well
MN040	444753092521701	11	123530	Domestic	St. Lawrence-Franconia Formations	260	Deep well
USGS	444904092560401	12	778336	Monitoring	Glacial surficial sand and/or gravel	54	Sewered residential
MN040	445003093290501	13	194919	Domestic	Prairie du Chien Group	183	Deep well
MN040	445005093285401	14	417569	Domestic	Prairie du Chien Group	240	Deep well
USGS	445006092540301	15	778334	Monitoring	Glacial surficial sand and/or gravel	84	Sewered residential
MN040	445443093261401	16	204590	Domestic	Glacial surficial sand and/or gravel	112	Deep well
USGS	445508093005601	17	767633	Monitoring	St. Peter Sandstone	49	Deep well
USGS	445732093203201	18	639311	Monitoring	Glacial surficial sand and/or gravel	19	Sewered residential
USGS	445807092525301	19	778354	Monitoring	Glacial surficial sand and/or gravel	43	Septic residential
MN040	445815092541101	20	404244	Public water system	Prairie du Chien Group	133	Deep well
USGS	445832092483201	21	641236	Domestic	Jordan Sandstone	278	Deep well
MN040	445917092494301	22	479662	Domestic	Jordan Sandstone	285	Deep well
USGS	445925092522101	23	778353	Monitoring	Glacial surficial sand and/or gravel	23	Septic residential
USGS	445933092522601	24	778352	Monitoring	Glacial surficial sand and/or gravel	30	Sewered residential
USGS	450056092522001	25	783302	Monitoring	Glacial surficial sand and/or gravel	15	Septic residential
USGS	45011092505301	26	778375	Monitoring	Glacial surficial sand and/or gravel	32	Undeveloped
USGS	450116092512501	27	783301	Monitoring	Glacial surficial sand and/or gravel	75	Septic residential
USGS	450122093193801	28	560426	Monitoring	Glacial surficial sand and/or gravel	19	Sewered residential
USGS	450129092551101	29	778349	Monitoring	Glacial surficial sand and/or gravel	28	Septic residential
USGS	450148093212301	30	203583	Domestic	Glacial buried sand and/or gravel	72	Deep well

**Table 1.** Selected information for wells sampled for this study.—Continued

[MUN, Minnesota unique well number; ft bgs, feet below ground surface; MPCA, Minnesota Pollution Control Agency; USGS, U.S. Geological Survey; MN040, Minnesota County Well Index]

Agency code	Station number	Map number (fig. 2)	MUN	Well type	Aquifer	Well depth (ft bgs)	MPCA-assigned land use or monitoring type
USGS	450226093203901	31	560423	Monitoring	Glacial surficial sand and/or gravel	29	Sewered residential
USGS	450236093175801	32	560425	Monitoring	Glacial surficial sand and/or gravel	17	Sewered residential
USGS	450328093232001	33	560422	Monitoring	Glacial surficial sand and/or gravel	18	Sewered residential
USGS	450333093201701	34	560418	Monitoring	Glacial surficial sand and/or gravel	9	Sewered residential
USGS	450430093220801	35	560417	Monitoring	Glacial surficial sand and/or gravel	22.5	Sewered residential
USGS	450448093205301	36	560415	Monitoring	Glacial surficial sand and/or gravel	18	Sewered residential
USGS	450557093201801	37	560410	Monitoring	Glacial surficial sand and/or gravel	22.5	Sewered residential
USGS	450702093185101	38	560408	Monitoring	Glacial surficial sand and/or gravel	24	Sewered residential
USGS	450704093210401	39	560407	Monitoring	Glacial surficial sand and/or gravel	24.25	Sewered residential
USGS	450811093135601	40	785072	Monitoring	Glacial surficial sand and/or gravel	11	Commercial/industrial
USGS	450918093132301	41	689997	Monitoring	Glacial surficial sand and/or gravel	17.7	Commercial/industrial
USGS	451032093071901	42	785071	Monitoring	Glacial surficial sand and/or gravel	15	Commercial/industrial
USGS	451056093072205	43	243470	Monitoring	Glacial surficial sand and/or gravel	13.2	Deep well
USGS	451315093202501	44	560381	Monitoring	Glacial surficial sand and/or gravel	20.75	Sewered residential
USGS	451336093260401	45	785064	Monitoring	Glacial surficial sand and/or gravel	15	Commercial/industrial
USGS	451346094111901	46	757565	Monitoring	Glacial surficial sand and/or gravel	20	Landfill monitoring
USGS	451421093094601	47	785654	Monitoring	Glacial surficial sand and/or gravel	13	Septic residential
USGS	451520093273501	48	785065	Monitoring	Glacial surficial sand and/or gravel	35	Septic residential
USGS	451602093165801	49	785651	Monitoring	Glacial surficial sand and/or gravel	14	Septic residential
USGS	451615093195901	50	785069	Monitoring	Glacial surficial sand and/or gravel	17	Septic residential
USGS	451705093121001	51	785653	Monitoring	Glacial surficial sand and/or gravel	15	Septic residential
USGS	451745093193901	52	785068	Monitoring	Glacial surficial sand and/or gravel	29	Septic residential
USGS	451816093232801	53	785067	Monitoring	Glacial surficial sand and/or gravel	13	Septic residential
USGS	451855093195901	54	245653	Monitoring	Glacial surficial sand and/or gravel	27.6	Landfill monitoring
USGS	451903093351001	55	785097	Monitoring	Glacial surficial sand and/or gravel	16	Commercial/industrial
USGS	451917093062901	56	786975	Monitoring	Glacial surficial sand and/or gravel	18	Undeveloped
USGS	451917093204101	57	785100	Monitoring	Glacial surficial sand and/or gravel	21	Septic residential
USGS	451942092583901	58	637774	Domestic	Glacial buried sand and/or gravel	73	Deep well
USGS	451942093244301	59	785098	Monitoring	Glacial surficial sand and/or gravel	30	Septic residential
USGS	452013093332001	60	649956	Monitoring	Glacial surficial sand and/or gravel	48	Deep well

**Table 1.** Selected information for wells sampled for this study.—Continued

[MUN, Minnesota unique well number; ft bgs, feet below ground surface; MPCA, Minnesota Pollution Control Agency; USGS, U.S. Geological Survey; MN040, Minnesota County Well Index]

Agency code	Station number	Map number (fig. 2)	MUN	Well type	Aquifer	Well depth (ft bgs)	MPCA-assigned land use or monitoring type
MN040	452043093134801	61	148184	Domestic	Jordan Sandstone	109	Deep well
USGS	452055093105401	62	783316	Monitoring	Glacial surficial sand and/or gravel	14	Septic residential
USGS	452107093185601	63	785099	Monitoring	Glacial surficial sand and/or gravel	15	Septic residential
USGS	452120093364201	64	785664	Monitoring	Glacial surficial sand and/or gravel	53	Septic residential
USGS	452153093133501	65	W30009	Monitoring	Glacial surficial sand and/or gravel	18	Landfill monitoring
USGS	452219093153801	66	783315	Monitoring	Glacial surficial sand and/or gravel	14	Deep well
USGS	452304093054701	67	783317	Monitoring	Glacial surficial sand and/or gravel	28	Septic residential
USGS	452355093244101	68	785096	Monitoring	Glacial surficial sand and/or gravel	19	Septic residential
USGS	452412093231801	69	783312	Monitoring	Glacial surficial sand and/or gravel	16	Septic residential
USGS	452422093063301	70	639312	Monitoring	Glacial surficial sand and/or gravel	25	Septic residential
USGS	452423093184501	71	783313	Monitoring	Glacial surficial sand and/or gravel	19	Septic residential
USGS	452425093071001	72	639313	Monitoring	Glacial surficial sand and/or gravel	23.5	Septic residential
USGS	452426093110201	73	785070	Monitoring	Glacial surficial sand and/or gravel	15	Undeveloped
MN040	452647094370601	74	449015	Domestic	Glacial surficial sand and/or gravel	52	Deep well
USGS	452657092531801	75	500773	Domestic	Glacial surficial sand and/or gravel	87	Deep well
USGS	452938094085101	76	594117	Monitoring	Glacial surficial sand and/or gravel	24	Sewered residential
USGS	453042094102201	77	594125	Monitoring	Glacial surficial sand and/or gravel	23.5	Sewered residential
USGS	453247094085701	78	561099	Monitoring	Glacial surficial sand and/or gravel	25	Commercial/industrial
USGS	453304094114801	79	783233	Monitoring	Glacial surficial sand and/or gravel	22.7	Commercial/industrial
USGS	453431094190301	80	440156	Domestic	Glacial surficial sand and/or gravel	60	Deep well
USGS	453436094141901	81	588392	Monitoring	Glacial surficial sand and/or gravel	22	Sewered residential
USGS	453441094140702	82	594121	Monitoring	Glacial surficial sand and/or gravel	21.5	Deep well
USGS	453453094140501	83	507162	Domestic	Glacial surficial sand and/or gravel	58	Deep well
USGS	453509094162101	84	588391	Monitoring	Glacial surficial sand and/or gravel	12	Deep well
USGS	453537094182901	85	540955	Monitoring	Glacial surficial sand and/or gravel	31	Commercial/industrial
USGS	453646094272301	86	403748	Domestic	Glacial surficial sand and/or gravel	32	Sewered residential
USGS	453717094264101	87	415618	Domestic	Glacial surficial sand and/or gravel	31	Sewered residential
USGS	454610094440101	88	646144	Monitoring	Glacial surficial sand and/or gravel	19	Septic residential
USGS	461743094154301	89	649961	Monitoring	Glacial surficial sand and/or gravel	16	Septic residential
USGS	461809092481301	90	244281	Monitoring	Glacial surficial sand and/or gravel	28.6	Undeveloped

**Table 1.** Selected information for wells sampled for this study.—Continued

[MUN, Minnesota unique well number; ft bgs, feet below ground surface; MPCA, Minnesota Pollution Control Agency; USGS, U.S. Geological Survey; MN040, Minnesota County Well Index]

Agency code	Station number	Map number (fig. 2)	MUN	Well type	Aquifer	Well depth (ft bgs)	MPCA-assigned land use or monitoring type
USGS	461815094150701	91	778172	Monitoring	Glacial surficial sand and/or gravel	26	Septic residential
USGS	462024094144501	92	778193	Monitoring	Glacial surficial sand and/or gravel	19	Commercial/industrial
USGS	462044094141201	93	778194	Monitoring	Glacial surficial sand and/or gravel	18	Commercial/industrial
USGS	462055094144501	94	778173	Monitoring	Glacial surficial sand and/or gravel	22	Commercial/industrial
USGS	462057094154301	95	778195	Monitoring	Glacial surficial sand and/or gravel	22	Septic residential
USGS	462112094130301	96	778174	Monitoring	Glacial surficial sand and/or gravel	19	Commercial/industrial
USGS	462302094125001	97	785656	Monitoring	Glacial surficial sand and/or gravel	44	Septic residential
USGS	462337093575601	98	582082	Monitoring	Glacial surficial sand and/or gravel	23	Undeveloped
USGS	463042094443501	99	778327	Monitoring	Glacial surficial sand and/or gravel	14	Undeveloped
USGS	463114094211301	100	778171	Monitoring	Glacial surficial sand and/or gravel	15	Undeveloped
USGS	463309094541101	101	778175	Monitoring	Glacial surficial sand and/or gravel	14	Undeveloped
USGS	463342096052901	102	637252	Domestic	Glacial surficial sand and/or gravel	64	Deep well
USGS	463836094080301	103	775499	Monitoring	Glacial surficial sand and/or gravel	22	Undeveloped
MN040	463930095480201	104	137527	Domestic	Glacial surficial sand and/or gravel	74	Deep well
USGS	464120094043701	105	778360	Monitoring	Glacial surficial sand and/or gravel	26	Undeveloped
USGS	464240094093101	106	775500	Monitoring	Glacial surficial sand and/or gravel	26	Septic residential
USGS	464452094461001	107	778197	Monitoring	Glacial surficial sand and/or gravel	14	Undeveloped
USGS	464538095050101	108	438559	Domestic	Glacial surficial sand and/or gravel	40	Deep well
USGS	464616095043101	109	646145	Monitoring	Glacial surficial sand and/or gravel	28	Septic residential
USGS	464631096384101	110	437602	Domestic	Glacial surficial sand and/or gravel	72	Deep well
USGS	465448094485501	111	708370	Monitoring	Glacial surficial sand and/or gravel	31.4	Undeveloped
USGS	471909094130701	112	778361	Monitoring	Glacial surficial sand and/or gravel	15	Undeveloped
MN040	472129094562901	113	243852	Monitoring	Glacial surficial sand and/or gravel	21	Undeveloped
USGS	472459094430401	114	783235	Monitoring	Glacial surficial sand and/or gravel	32.5	Undeveloped
USGS	472728094505501	115	243264	Monitoring	Glacial surficial sand and/or gravel	22	Sewered residential
USGS	472741094504001	116	689995	Monitoring	Glacial surficial sand and/or gravel	30.3	Sewered residential
USGS	473031094570201	117	689996	Monitoring	Glacial surficial sand and/or gravel	15.3	Commercial/industrial
USGS	473750095521601	118	132701	Domestic	Glacial surficial sand and/or gravel	60	Deep well

**Table 2.** Analytical schedules, chemicals, and chemical categories of chemicals analyzed in groundwater samples.

[USGS, U.S. Geological Survey; CASRN, Chemical Abstracts Service Registry Number; µg/L, micrograms per liter; ng/L, nanograms per liter]

Analytical schedule	Chemical	USGS parameter code	Chemical category <sup>1</sup>	CASRN <sup>2</sup>	Laboratory reporting level	Unit
1433	1-Methylnaphthalene	62054	2–5 percent of gasoline, diesel fuel, or crude oil	90–12–0	0.022	µg/L
1433	1,4-Dichlorobenzene	34572	Moth repellant, fumigant, deodorant	106–46–7	0.04	µg/L
1433	2-Methylnaphthalene	62056	2–5 percent of gasoline, diesel fuel, or crude oil	91–57–6	0.036	µg/L
1433	2,6-Dimethylnaphthalene	62055	Present in diesel/kerosene (trace in gasoline)	581–42–0	0.06	µg/L
1433	3- <i>beta</i> -Coprostanol	62057	Carnivore fecal indicator	360–68–9	1.8	µg/L
1433	3-Methyl-1 <i>H</i> -indole	62058	Stench in feces and coal tar	83–34–1	0.036	µg/L
1433	3- <i>tert</i> -Butyl-4-hydroxyanisole	62059	Antioxidant, general preservative	121–00–6	0.6	µg/L
1433	4-Cumylphenol	62060	Nonionic detergent metabolite	599–64–4	0.06	µg/L
1433	4- <i>n</i> -Octylphenol	62061	Nonionic detergent metabolite	1806–26–4	0.06	µg/L
1433	4-Nonylphenol (all isomers)	62085	Nonionic detergent metabolite	104–40–5	2	µg/L
1433	4-Nonylphenol diethoxylate	62083	Detergent	20427–84–3	5	µg/L
1433	4- <i>tert</i> -Octylphenol	62062	Nonionic detergent metabolite	140–66–9	0.14	µg/L
1433	4- <i>tert</i> -Octylphenol diethoxylate	61705	Nonionic detergent metabolite	2315–61–9	1	µg/L
1433	4- <i>tert</i> -Octylphenolmonoethoxylate	61706	Nonionic detergent metabolite	2315–67–5	1	µg/L
1433	5-Methyl-1 <i>H</i> -benzotriazole	62063	Corrosion inhibitor used in antifreezes, brake fluids, dishwashing agents, deicing fluids and other applications	136–85–6	1.2	µg/L
1433	9,10-Anthraquinone	62066	Manufacturing dye/textiles, seed treatment, bird repellant	84–65–1	0.16	µg/L
1433	Acetophenone	62064	Fragrance in detergent and tobacco, flavor in beverages	98–86–2	0.4	µg/L
1433	Acetyl-hexamethyl-tetrahydro-naphthalene (AHTN)	62065	Musk, fragrance	21145–77–7	0.028	µg/L
1433	Anthracene	34221	Wood preservative, component of tar, diesel, or crude oil, combustion product	120–12–7	0.01	µg/L
1433	Benzo[ <i>a</i> ]pyrene	34248	Regulated polycyclic aromatic hydrocarbon, used in cancer research, combustion product	50–32–8	0.06	µg/L
1433	Benzophenone	62067	Fixative for perfumes and soaps	119–61–9	0.08	µg/L
1433	<i>beta</i> -Sitosterol	62068	Plant sterol	83–46–5	4	µg/L
1433	<i>beta</i> -Stigmastanol	62086	Plant sterol	19466–47–8	2.6	µg/L
1434	Bromacil	04029	Herbicide, non-cropland brush control	314–40–9	0.36	µg/L
1433	Caffeine	50305	Stimulant, diuretic, very mobile/biodegradable	58–08–2	0.06	µg/L
1433	Camphor	62070	Flavor, odorant, ointments	76–22–2	0.044	µg/L
1433	Carbaryl	82680	Insecticide, crop and garden uses, low persistence	63–25–2	0.16	µg/L
1433	Carbazole	62071	Insecticide, manufacturing dyes, explosives, and lubricants	86–74–8	0.03	µg/L

**Table 2.** Analytical schedules, chemicals, and chemical categories of chemicals analyzed in groundwater samples.—Continued

[USGS, U.S. Geological Survey; CASRN, Chemical Abstracts Service Registry Number; µg/L, micrograms per liter; ng/L, nanograms per liter]

Analytical schedule	Chemical	USGS parameter code	Chemical category <sup>1</sup>	CASRN <sup>2</sup>	Laboratory reporting level	Unit
1433	Chlorpyrifos	38933	Insecticide, domestic pest and termite control (domestic use restricted as of 2001)	2921-88-2	0.16	µg/L
1433	Cholesterol	62072	Often a fecal indicator, also a plant sterol	57-88-5	2	µg/L
1433	Cotinine	62005	Primary nicotine metabolite	486-56-6	0.8	µg/L
1433	Diazinon	39572	Insecticide, greater than 40 percent nonagricultural usage, ants, flies	333-41-5	0.16	µg/L
1433	D-Limonene	62073	Fragrance, solvent, cleaners	5989-27-5	0.08	µg/L
1433	Fluoranthene	34377	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), combustion product	206-44-0	0.024	µg/L
1433	Hexahydrohexamethylcyclopentabenzopyran (HHCB)	62075	Musk, fragrance	1222-05-5	0.052	µg/L
1433	Indole	62076	Pesticide inert ingredient, fragrance in coffee	120-72-9	0.08	µg/L
1433	Isoborneol	62077	Moth repellent, industrial manufacture of plastics, embalming fluids, explosives, cosmetics, and varnishes	124-76-5	0.08	µg/L
1433	Isophorone	34409	Solvent for lacquer, plastic, oil, silicon, resin	78-59-1	0.032	µg/L
1433	Isopropylbenzene	62078	Manufacturing phenol/acetone, fuels and paint thinner	98-82-8	0.3	µg/L
1433	Isoquinoline	62079	Flavors and fragrances	119-65-3	0.046	µg/L
1433	Menthol	62080	Cigarettes, cough drops, liniment, mouthwash	89-78-1	0.32	µg/L
1433	Metalaxyl	50359	Herbicide, fumigant, mildew, blight, pathogens, golf/turf	57837-19-1	0.12	µg/L
1433	Methyl salicylate	62081	Liniment, food, beverage, ultraviolet-absorbing lotion	119-36-8	0.044	µg/L
1433	Metolachlor	39415	Herbicide, indicator of agricultural drainage	51218-45-2	0.028	µg/L
1433	Naphthalene	34443	Fungicide, moth repellent, major component (about 10 percent) of gasoline	91-20-3	0.04	µg/L
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET)	62082	Insecticide, urban uses, mosquito repellent	134-62-3	0.06	µg/L
1433	<i>p</i> -Cresol (4-Methylphenol)	62084	Wood preservative	106-44-5	0.08	µg/L
1433	Phenanthrene	34462	Manufacturing explosives, component of tar, diesel fuel, or crude oil, combustion product	85-01-8	0.016	µg/L
1433	Phenol	34466	Disinfectant, manufacturing several products, leachate	108-95-2	0.16	µg/L
1434	Prometon	04037	Herbicide, non-crop annual and perennial broad-leaf weed, brush, and grass	1610-18-0	0.012	µg/L
1433	Pyrene	34470	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), combustion product	129-00-0	0.042	µg/L
1433	Tetrachloroethene	34476	Solvent, degreaser, veterinary anesthetic	127-18-4	0.12	µg/L
1433	Tribromomethane	34288	Wastewater ozonation byproduct, military/explosives	75-25-2	0.1	µg/L

**Table 2.** Analytical schedules, chemicals, and chemical categories of chemicals analyzed in groundwater samples.—Continued

[USGS, U.S. Geological Survey; CASRN, Chemical Abstracts Service Registry Number; µg/L, micrograms per liter; ng/L, nanograms per liter]

Analytical schedule	Chemical	USGS parameter code	Chemical category <sup>1</sup>	CASRN <sup>2</sup>	Laboratory reporting level	Unit
1433	Tributyl phosphate	62089	Anti-foaming agent, flame retardant	126–73–8	0.16	µg/L
1433	Triclosan	62090	Disinfectant, antimicrobial (concern for acquired microbial resistance)	3380–34–5	0.2	µg/L
1433	Triethyl citrate	62091	Cosmetics, pharmaceuticals	77–93–0	0.16	µg/L
1433	Triphenyl phosphate	62092	Plasticizer, resin, wax, finish, roofing paper	115–86–6	0.12	µg/L
1433	Tris(1,3-dichloroisopropyl) phosphate (FY-ROL FR 2)	62088	Flame retardant	13674–87–8	0.16	µg/L
1433	Tris(2-butoxyethyl) phosphate	62093	Flame retardant	78–51–3	0.8	µg/L
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	62087	Plasticizer, flame retardant	115–96–8	0.1	µg/L
2080	1,7-Dimethylxanthine	62030	Caffeine degradate	611–59–6	0.1	µg/L
2080	Acetaminophen	62000	Analgesic	103–90–2	0.12	µg/L
2080	Albuterol	62020	Bronchodilator	58–08–2	0.06	µg/L
2080	Caffeine	50305	Stimulant	58–08–2	0.06	µg/L
2080	Carbamazepine	62793	Anti-convulsant, attention-deficit hyperactivity disorder (ADHD)	298–46–4	0.06	µg/L
2080	Codeine	62003	Opiate agonist	76–57–3	0.046	µg/L
2080	Cotinine	62005	Primary nicotine metabolite	486–56–6	0.038	µg/L
2080	Dehydronifedipine	62004	Nifedipine degradate; nifedipine is an antihypertensive	67035–22–7	0.08	µg/L
2080	Diltiazem	62008	Antihypertensive	42399–41–7	0.06	µg/L
2080	Diphenhydramine	62796	Antihistamine	58–73–1	0.058	µg/L
2080	Sulfamethoxazole	62021	Antibiotic	723–46–6	0.091	µg/L
2080	Thiabendazole	62801	Anthelmintic, fungicide	148–79–8	0.06	µg/L
2080	Trimethoprim	62023	Antibiotic	738–70–5	0.034	µg/L
2080	Warfarin	62024	Anticoagulant, rodenticide	81–81–2	0.08	µg/L
2434	3- <i>beta</i> -Coprostanol	64512	Carnivore fecal indicator; useful sewage tracer	360–68–9	200	ng/L
2434	4-Androstene-3,17-dione	64513	Testosterone precursor; illicit steroid	63–05–8	0.8	ng/L
2434	11-Ketotestosterone	64507	Testosterone metabolite	564–35–2	2	ng/L
2434	17- <i>alpha</i> -Estradiol	64508	Low occurrence in humans, common in other species	57–91–0	0.8	ng/L
2434	17- <i>alpha</i> -Ethinyl estradiol	64509	Used in oral contraceptives	57–63–6	0.8	ng/L
2434	17- <i>beta</i> -Estradiol	64510	Principal estrogen in humans	50–28–2	0.8	ng/L
2434	Bisphenol A	67304	Used to make polycarbonate plastics and epoxy resins	80–05–7	100	ng/L
2434	Cholesterol	64514	Ubiquitous sterol; produced by animals and plants	57–88–5	200	ng/L

**Table 2.** Analytical schedules, chemicals, and chemical categories of chemicals analyzed in groundwater samples.—Continued

[USGS, U.S. Geological Survey; CASRN, Chemical Abstracts Service Registry Number; µg/L, micrograms per liter; ng/L, nanograms per liter]

Analytical schedule	Chemical	USGS parameter code	Chemical category <sup>1</sup>	CASRN <sup>2</sup>	Laboratory reporting level	Unit
2434	<i>cis</i> -Androsterone	64515	Testosterone metabolite; used in deer repellent	53-41-8	0.8	ng/L
2434	Dihydrotestosterone	64524	Testosterone metabolite	521-18-6	4	ng/L
2434	Epitestosterone	64517	Testosterone isomer; human androgen	481-30-1	2	ng/L
2434	Equilenin	64518	Equine estrogen; used in hormone replacement therapy	517-09-9	2	ng/L
2434	Equilin	64519	Equine estrogen; used in hormone replacement therapy	474-86-2	8	ng/L
2434	Estriol	64520	Metabolite of 17- <i>beta</i> -estradiol	50-27-1	2	ng/L
2434	Estrone	64521	Metabolite of 17- <i>beta</i> -estradiol	53-16-7	0.8	ng/L
2434	Mestranol	64522	Used in oral contraceptives; metabolized to ethynylestradiol prior to excretion	72-33-3	0.8	ng/L
2434	Norethindrone	64511	Used in oral contraceptives	68-22-4	0.8	ng/L
2434	Progesterone	64523	Principal human progestational hormone	57-83-0	8	ng/L
2434	Testosterone	64525	Principal human androgen	58-22-0	1.6	ng/L
2434	<i>trans</i> -Diethylstilbestrol	64516	Pharmaceutical	56-53-1	0.8	ng/L
LCAB	4-Epichlorotetracycline	63731	Chlorotetracycline degradate	14297-93-9	0.01	µg/L
LCAB	4-Epioxytetracycline	63729	Oxytetracycline degradate	14206-58-7	0.01	µg/L
LCAB	4-Epitetracycline	63727	Tetracycline degradate	23313-80-6	0.01	µg/L
LCAB	Anhydroerthromycin	63674	Macrolide antibiotic used in erythromycin degradate	114-07-8	0.008	µg/L
LCAB	Azithromycin	62792	Macrolide antibiotic used in humans	83905-01-5	0.005	µg/L
LCAB	Carbamazepine	62793	Antiepileptic	298-46-4	0.005	µg/L
LCAB	Chloramphenicol	65194	Macrolide antibiotic used in humans	56-75-7	0.1	µg/L
LCAB	Chlorotetracycline	61744	Tetracycline antibiotic used in swine, poultry, cattle, sheep, ducks	57-62-5	0.01	µg/L
LCAB	Ciprofloxacin	62898	Quinolone antibiotic used in humans, swine, chickens	85721-33-1	0.005	µg/L
LCAB	Doxycycline	62694	Tetracycline antibiotic used in humans, dog	564-25-0	0.01	µg/L
LCAB	Enrofloxacin	66495	Quinolone antibiotic used in cattle, swine, poultry, dogs, cats	93106-60-6	0.005	µg/L
LCAB	Erythromycin	62797	Macrolide antibiotic used in humans, poultry, swine	114-07-8	0.008	µg/L
LCAB	Ibuprofen	62014	Nonsteroidal anti-inflammatory drug for pain relief, fever reduction, and swelling reduction	15687-27-1	0.05	µg/L
LCAB	Isochlorotetracycline	64175	Chlorotetracycline degradate	514-53-4	0.01	µg/L
LCAB	Isoepichlorotetracycline	64047	Chlorotetracycline degradate	not available	0.01	µg/L
LCAB	Lincomycin	62894	Antibiotic used in poultry, swine	154-21-2	0.005	µg/L
LCAB	Lomefloxacin	62900	Quinolone antibiotic used in humans	98079-51-7	0.005	µg/L

**Table 2.** Analytical schedules, chemicals, and chemical categories of chemicals analyzed in groundwater samples.—Continued

[USGS, U.S. Geological Survey; CASRN, Chemical Abstracts Service Registry Number; µg/L, micrograms per liter; ng/L, nanograms per liter]

Analytical schedule	Chemical	USGS parameter code	Chemical category <sup>1</sup>	CASRN <sup>2</sup>	Laboratory reporting level	Unit
LCAB	Norfloxacin	62757	Quinolone antibiotic used in humans, poultry	70458–96–7	0.005	µg/L
LCAB	Ofloxacin	62899	Quinolone antibiotic used in poultry, humans	82419–36–1	0.005	µg/L
LCAB	Ormetoprim	62962	Antibiotic used in fish, poultry	6981–18–6	0.005	µg/L
LCAB	Oxytetracycline	61759	Tetracycline antibiotic used in poultry, fish, swine, cattle, sheep, bees, lobsters	79–57–2	0.01	µg/L
LCAB	Roxithromycin	62895	Macrolide antibiotic used in humans	80214–83–1	0.005	µg/L
LCAB	Sarafloxacin	62771	Quinolone antibiotic used in poultry, fish	98105–99–8	0.005	µg/L
LCAB	Sulfachloropyridazine	62774	Sulfonamide antibiotic used in swine, calves, dogs	80–32–0	0.005	µg/L
LCAB	Sulfadiazine	62963	Sulfonamide antibiotic used in horses, humans	68–35–9	0.005	µg/L
LCAB	Sulfadimethoxine	62776	Sulfonamide antibiotic used in fish, poultry	122–11–2	0.005	µg/L
LCAB	Sulfamethazine	61762	Sulfonamide antibiotic used in swine, cattle	57–68–1	0.005	µg/L
LCAB	Sulfamethoxazole	62775	Sulfonamide antibiotic used in humans	723–46–6	0.005	µg/L
LCAB	Sulfathiazole	62778	Sulfonamide antibiotic used in swine	72–14–0	0.005	µg/L
LCAB	Tetracycline	62781	Tetracycline antibiotic used in humans, dogs, cattle	60–54–8	0.01	µg/L
LCAB	Trimethoprim	62023	Antibiotic used in humans, dogs, horses	738–70–5	0.005	µg/L
LCAB	Tylosin	62896	Macrolide antibiotic used in chickens, swine, cattle	1401–69–0	0.01	µg/L
LCAB	Virginiamycin	62897	Macrolide antibiotic used in poultry, swine, cattle	21411–53–0	0.005	µg/L

<sup>1</sup> From Foreman and others (2012), Furlong and others (2008), Meyer and others (2007), and Zaugg and others (2006).<sup>2</sup> This report contains Chemical Abstracts Service Registry Numbers (CASRN)<sup>®</sup>, which is a registered trademark of the American Chemical Society. The CASRN online database provides the latest registry number information: <http://www.cas.org/>. Chemical Abstracts Service recommends the verification of the CASRNs through CAS Client Services<sup>SM</sup>.

## Methods

This section of the report describes field procedures used to collect the groundwater samples, laboratory analytical methods, laboratory quality-assurance and quality-control samples and analyses, field quality-assurance and quality-control samples and analyses, and data censoring criteria.

### Groundwater-Sample Collection

Methods for collection of groundwater samples were presented in Erickson (2012) and are briefly described in this section. Staff from the USGS provided training to MPCA hydrologic technicians on USGS sampling protocols and on the use of the USGS Personal Computer Field Forms (PCFF) computer program, which was used to record field data. An experienced USGS hydrologic technician accompanied MPCA staff during the first week of sampling to fully train and assist MPCA staff. Staff from the MPCA collected 123 environmental water samples from 118 wells during November 2009 through June 2012, with most samples collected during April, May, and June of each year. Associated field quality-assurance samples, such as replicate samples and blank samples, were also collected. Water samples were collected by MPCA staff according to the USGS National Field Manual for the Collection of Water-Quality Data (NFM) (U.S. Geological Survey, variously dated). Staff from the USGS verified sample completeness and labeling, shipped all samples to the USGS laboratories, and entered necessary site and sample information into USGS databases.

Before collecting samples at each site, the well was purged using the submersible pump already installed in the well (domestic or public water system wells) or a submersible or peristaltic pump outfitted with polytetrafluoroethylene tubing (monitoring wells). Field properties of water, such as dissolved oxygen, pH, specific conductance, and temperature were measured with a multiparameter sonde and recorded using the PCFF computer program as specified in the NFM. Wells were purged until field properties were stable. Samples for analysis of organic contaminants were filtered and collected in baked glass amber bottles, according to USGS protocols (Wilde and others, 2004), except that the samples for analysis using schedule 2434 were contained in new 0.5-liter (L) high-density polyethylene bottles, as specified for that method (Foreman and others, 2012). Samples for analysis by schedule 2434 that were not processed within 3 days of receipt by the NWQL were stored in a freezer at -5 degrees Celsius (°C) or less, until the day preceding extraction when the samples were allowed to thaw at room temperature.

Sampling equipment was cleaned between sampling sites using, in sequence, Liqui-Nox® and tap water solution, tap water, deionized water, methanol, and organic-free blank water as specified in the NFM (U.S. Geological Survey, variously dated). Sampling personnel generally refrained from using personal-care products (for example, mosquito repellent

containing *N,N*-diethyl-*meta*-toluamide [DEET]) to avoid contamination of the samples during collection.

Field quality-assurance samples collected for this study included replicate, spike, and blank samples. Field-replicate samples were collected at 12 wells during the sampling to quantify variability between samples. Matrix spike/matrix spike duplicate samples were collected at 6 of the 118 wells to quantify potential matrix interferences in laboratory methods. Field-equipment blank samples were collected at 16 of the 118 wells to characterize any contamination potentially introduced during field activities.

### Laboratory Analytical Methods

Wastewater chemicals were analyzed using NWQL schedule 1433, which consists of solid-phase extraction and gas chromatography/mass spectrometry (Zaugg and others, 2006). The method focuses on the determination of chemicals that are indicators of municipal and domestic wastewater, such as alkylphenol ethoxylate nonionic surfactants and their degradates, food additives, fragrances, antioxidants, flame retardants, plasticizers, industrial solvents, disinfectants, fecal sterols, polycyclic aromatic hydrocarbons, and high-use domestic pesticides. Briefly, water samples are filtered and then are extracted by vacuum through disposable solid-phase cartridges that contain polystyrene-divinylbenzene resin. The cartridges are then dried with nitrogen gas, and the sorbed chemicals are eluted with dichloromethane-diethyl ether. The concentrations of the wastewater chemicals are measured by capillary-column gas chromatography/mass spectrometry (Zaugg and others, 2006).

Human-health pharmaceuticals were analyzed using NWQL schedule 2080, by solid phase extraction and high-performance liquid chromatography/mass spectrometry (Furlong and others, 2008). The method quantifies 14 commonly used human pharmaceuticals in filtered-water samples. Briefly, the method uses a chemically modified styrene-divinylbenzene resin-based solid-phase extraction cartridge for analyte isolation and concentration. For analyte detection and quantitation, this method uses a high-performance liquid chromatography/mass spectrometry system to separate the pharmaceuticals of interest from each other and from co-extracted material. Immediately following separation, the pharmaceuticals are ionized by electrospray ionization operated in the positive mode, and the positive ions produced are detected, identified, and quantified using a quadrupole mass spectrometer (Furlong and others, 2008).

Steroidal hormones and related chemicals were analyzed using NWQL schedule 2434, which consists of solid-phase extraction, derivatization, and gas chromatography with tandem mass spectrometry (Foreman and others, 2012). Chemicals analyzed include 16 steroidal hormones and 4 other related compounds (*trans*-diethylstilbestrol, bisphenol A, cholesterol, and 3-*beta*-coprostanol). Briefly, a technique referred to as isotope-dilution quantification is used for this analytical

method to improve quantitative accuracy by accounting for sample-specific procedural losses in the determined analyte concentration (Foreman and others, 2012). Deuterium- or carbon-13-labeled isotope-dilution standards (IDSs), all of which are direct or chemically similar isotopic analogs of the method analytes, are added to all environmental and quality-control and quality-assurance samples before extraction. Method analytes and IDS compounds are isolated from filtered water by solid-phase extraction onto an octadecylsilyl disk, and eluted with methanol. The extract is evaporated to dryness, reconstituted in solvent, passed through a Florisil solid-phase extraction column to remove polar organic interferences, again evaporated to dryness, and reacted to ether derivatives that are more amenable to gas chromatographic separation than the original compounds. The analysis is carried out by gas chromatography with tandem mass spectrometry using calibration standards that are derivatized concurrently with the sample extracts (Foreman and others, 2012). Chemical concentrations are quantified relative to specific IDS compounds in the sample, which directly compensate for incomplete recovery in the determined and reported analyte concentrations. Thus, reported concentrations or recoveries of the steroidal hormones and related chemicals are corrected based on recovery of the corresponding IDS compound during the quantification process (Foreman and others, 2012).

Antibiotics and other pharmaceuticals were analyzed using the LCAB method of the USGS OGRL. The LCAB method is an evolving method that was modified from a liquid chromatography/tandem mass spectrometry version of the online solid-phase extraction liquid chromatography/mass spectrometry method in Meyer and others (2007). Briefly, water samples were analyzed for chloramphenicol, lincomycin, ormetoprim, trimethoprim, macrolide antibiotics, sulfonamide antibiotics, quinolone antibiotics, and tetracycline antibiotics, antibiotic degradation products, and the pharmaceuticals carbamazepine and ibuprofen using an online solid-phase extraction and ultra-pressure liquid chromatography/tandem mass spectrometry with electrospray ionization using multiple reaction monitoring. Samples were analyzed in positive-ion mode except for chloramphenicol and ibuprofen, which were analyzed in negative-ion mode (Meyer and others, 2007).

## Quality-Assurance Analyses

Quality-assurance plans were established to evaluate laboratory and field sampling techniques, assess possible sources of contamination, and assure representative samples. All field personnel were familiar with study design and sampling protocols before field sampling or data processing to assure sample integrity.

## Laboratory Quality Assurance

Results of analyses completed by the NWQL for this study are reported as estimated values, or “E” coded values, for concentrations measured above the long-term method detection level but below the laboratory reporting level (Childress and others, 1999). Nondetections are censored at the laboratory reporting level (table 2).

Laboratory quality-control samples were used to validate and interpret the environmental data. Laboratory quality-control samples included laboratory blanks, reagent spikes, and surrogates. Details of USGS quality-control specifications are described in Maloney (2005). Because the NWQL reports estimated values, analytical results from laboratory and field quality-control samples must be carefully scrutinized and compared to environmental sample results to ensure that reported environmental detections are unlikely to be false positives.

Recoveries for chemicals spiked into laboratory reagent water, and surrogate compounds spiked into environmental samples, indicate the general proficiency of the laboratory methods. For laboratory reagent-water spike samples related to the sample sets for this study (table 3), most chemicals had satisfactory spike recoveries, between 50 and 120 percent (Sando and others, 2006). The chemicals on schedule 1433 with low average recoveries (less than 50 percent) were 3-*tert*-butyl-4-hydroxyanisole, carbaryl, cotinine, D-limonene, isopropylbenzene, and tetrachloroethene; from the schedule 2080 list, the chemicals diltiazem and sulfamethoxazole had low average recoveries. No chemicals had average recoveries greater than 120 percent. All average recoveries were within the normal recovery ranges for the analyte and method; however the recovery of the chemical isoquinoline (laboratory analytical schedule 1433) was highly variable.

Surrogates are compounds that have similar properties to the chemicals of interest, but do not interfere with quantitation of the chemicals of interest. Each analytical method had surrogate compounds added to environmental and field quality-control samples before extraction to monitor method performance, as described in the references provided previously in the “Laboratory Analytical Methods” section. The compound decafluorobiphenyl (schedule 1433) was the only compound with an average surrogate spike recovery below 50 percent (table 4), and this low average recovery is within the normal recovery range for this compound (Zaugg and others, 2006).

Laboratory reagent-water blank samples were used to assess potential sample contamination attributed to laboratory processes. The laboratory-blank samples that were analyzed with the sample sets for this study were used in this analysis. A total of 31 chemicals on schedule 1433 had a detectable concentration in more than 1 laboratory-blank sample (table 5). The following chemicals that were detected in 10 or more of the 44 laboratory reagent-water blank samples were all from schedule 1433 and were, in decreasing order of detections: acetophenone, naphthalene, phenol, 4-nonylphenol (all isomers), 2-methylnaphthalene, benzophenone, *beta*-sitosterol,

**Table 3.** Summary of chemical recoveries for laboratory reagent-water spike samples related to sample sets for this study, 2009–12.

Analytical schedule	Chemical	Number of laboratory spikes analyzed	Average percent recovery	Standard deviation of percent recovery
1433	1-Methylnaphthalene	43	69	10
1433	1,4-Dichlorobenzene	43	59	10
1433	2-Methylnaphthalene	43	63	10
1433	2,6-Dimethylnaphthalene	43	59	11
1433	3- <i>beta</i> -Coprostanol	43	66	15
1433	3-Methyl-1 <i>H</i> -indole	43	80	15
1433	3- <i>tert</i> -Butyl-4-hydroxyanisole	43	38	20
1433	4-Cumylphenol	43	85	13
1433	4- <i>n</i> -Octylphenol	43	61	13
1433	4-Nonylphenol (all isomers)	43	71	11
1433	4-Nonylphenol diethoxylate	43	77	16
1433	4- <i>tert</i> -Octylphenol	43	79	13
1433	4- <i>tert</i> -Octylphenol diethoxylate	43	87	29
1433	4- <i>tert</i> -Octylphenolmonoethoxylate	43	74	14
1433	5-Methyl-1 <i>H</i> -benzotriazole	43	59	13
1433	9,10-Anthraquinone	43	92	13
1433	Acetophenone	43	101	10
1433	Acetyl-hexamethyl-tetrahydro-naphthalene (AHTN)	43	84	11
1433	Anthracene	43	78	10
1433	Benzo[ <i>a</i> ]pyrene	43	76	15
1433	Benzophenone	43	94	10
1433	<i>beta</i> -Sitosterol	43	61	20
1433	<i>beta</i> -Stigmastanol	43	61	19
1434	Bromacil	43	89	16
1433	Caffeine	43	91	10
1433	Camphor	43	87	9
1433	Carbaryl	43	44	14
1433	Carbazole	43	91	12
1433	Chlorpyrifos	43	69	12
1433	Cholesterol	43	64	16
1433	Cotinine	43	40	12
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET )	43	93	12
1433	Diazinon	43	87	12
1433	D-Limonene	43	36	15
1433	Fluoranthene	43	89	10
1433	Hexahydrohexamethylcyclopentabenzopyran (HHCB)	43	80	10
1433	Indole	43	62	19
1433	Isoborneol	43	81	13
1433	Isophorone	43	87	12
1433	Isopropylbenzene	43	49	12
1433	Isoquinoline	43	93	60
1433	Menthol	43	84	10

**Table 3.** Summary of chemical recoveries for laboratory reagent-water spike samples related to sample sets for this study, 2009–12.—Continued

Analytical schedule	Chemical	Number of laboratory spikes analyzed	Average percent recovery	Standard deviation of percent recovery
1433	Metalaxyl	43	95	11
1433	Methyl salicylate	43	90	9
1433	Metolachlor	43	90	12
1433	Naphthalene	43	73	9
1433	<i>p</i> -Cresol	43	86	12
1433	Phenanthrene	43	83	10
1433	Phenol	43	93	11
1434	Prometon	43	86	12
1433	Pyrene	43	90	10
1433	Tetrachloroethene	43	21	10
1433	Tribromomethane	43	59	9
1433	Tributyl phosphate	43	85	17
1433	Triclosan	43	78	11
1433	Triethyl citrate	43	85	18
1433	Triphenyl phosphate	43	74	14
1433	Tris(1,3-dichloroisopropyl) phosphate (FYROL FR 2)	43	89	11
1433	Tris(2-butoxyethyl) phosphate	43	83	13
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	43	87	9
2080	1,7-Dimethylxanthine	51	117	33
2080	Acetaminophen	51	88	23
2080	Albuterol	51	73	22
2080	Caffeine	51	116	22
2080	Carbamazepine	51	98	16
2080	Codeine	51	86	16
2080	Cotinine	51	93	12
2080	Dehydronifedipine	51	89	17
2080	Diltiazem	51	38	16
2080	Diphenhydramine	51	78	18
2080	Sulfamethoxazole	51	39	34
2080	Thiabendazole	51	82	19
2080	Trimethoprim	51	101	15
2080	Warfarin	51	55	22
2434	3- <i>beta</i> -Coprostanol	44	94	9
2434	4-Androstene-3,17-dione	44	95	11
2434	11-Ketotestosterone	44	89	27
2434	17- <i>alpha</i> -Estradiol	44	100	9
2434	17- <i>alpha</i> -Ethinyl estradiol	44	96	7
2434	17- <i>beta</i> -Estradiol	44	99	6
2434	Bisphenol A	44	89	14
2434	Cholesterol	44	94	11
2434	<i>cis</i> -Androsterone	44	107	20
2434	Dihydrotestosterone	44	99	20

**Table 3.** Summary of chemical recoveries for laboratory reagent-water spike samples related to sample sets for this study, 2009–12.—Continued

Analytical schedule	Chemical	Number of laboratory spikes analyzed	Average percent recovery	Standard deviation of percent recovery
2434	Epitestosterone	44	102	15
2434	Equilenin	44	82	10
2434	Equilin	44	80	14
2434	Estriol	44	88	23
2434	Estrone	44	99	7
2434	Mestranol	44	96	6
2434	Norethindrone	44	97	8
2434	Progesterone	44	94	27
2434	Testosterone	44	102	16
2434	<i>trans</i> -Diethylstilbestrol	44	88	7
LCAB	4-Epioxytetracycline	19	104	22
LCAB	4-Epitetracycline	19	103	22
LCAB	Anhydroerthromycin	19	100	10
LCAB	Azithromycin	19	100	27
LCAB	Carbamazepine	19	85	16
LCAB	Chloramphenicol	19	101	14
LCAB	Ciprofloxacin	19	99	14
LCAB	Doxycycline	19	103	23
LCAB	Enrofloxacin	19	90	21
LCAB	Erythromycin	19	120	16
LCAB	Ibuprofen	19	95	13
LCAB	Lincomycin	19	91	27
LCAB	Lomefloxacin	19	96	12
LCAB	Norfloxacin	19	94	24
LCAB	Ofloxacin	19	93	19
LCAB	Ormetoprim	19	94	19
LCAB	Oxytetracycline	19	107	23
LCAB	Roxithromycin	19	97	16
LCAB	Sarafloxacin	19	93	19
LCAB	Sulfachloropyridazine	19	94	14
LCAB	Sulfadiazine	19	85	12
LCAB	Sulfadimethoxine	19	68	9
LCAB	Sulfamethazine	19	104	12
LCAB	Sulfamethoxazole	19	93	11
LCAB	Sulfathiazole	19	91	26
LCAB	Tetracycline	19	101	22
LCAB	Total Chlorotetracycline	19	102	31
LCAB	Trimethoprim	19	89	24
LCAB	Tylosin	19	90	9
LCAB	Virginiamycin	19	91	14

**Table 4.** Summary of recoveries for surrogate compounds and isotope dilution standards analyzed in groundwater samples, 2009–12.[*d*, deuterium; <sup>13</sup>C, carbon-13]

Analytical schedule	Analyte	Number of surrogates analyzed	Average percent recovery	Standard deviation of percent recovery
1433	Caffeine- <sup>13</sup> C	160	87	12
1433	Decafluorobiphenyl	160	48	9
1433	Fluoranthene- <i>d</i> <sub>10</sub>	160	90	8
2080	Carbamazepine- <i>d</i> <sub>10</sub>	162	74	30
2080	Ethyl nicotinate- <i>d</i> <sub>4</sub>	162	78	18
2434	Bisphenol A- <i>d</i> <sub>16</sub>	163	80	16
2434	Progesterone-2,3,4- <sup>13</sup> C <sub>3</sub>	44	66	6
2434	Cholesterol- <i>d</i> <sub>7</sub>	163	69	11
2434	Ethinylestradiol- <i>d</i> <sub>4</sub>	163	81	12
2434	<i>trans</i> -Diethylstilbestrol- <i>d</i> <sub>8</sub>	163	65	13
2434	Mestranol- <i>d</i> <sub>4</sub>	163	79	10
2434	Estriol-2,4,16,17- <i>d</i> <sub>4</sub>	103	71	16
2434	16-Epiestriol- <i>d</i> <sub>2</sub>	161	65	22
2434	Medroxyprogesterone- <i>d</i> <sub>3</sub>	161	72	18
2434	Nandrolone- <i>d</i> <sub>3</sub>	161	80	16
2434	17- <i>beta</i> -Estradiol- <sup>13</sup> C <sub>6</sub>	161	84	19
2434	Estrone- <sup>13</sup> C <sub>6</sub>	161	83	17

and *p*-cresol. All of these frequent detections in laboratory-blank samples were estimated concentrations less than the laboratory reporting level. Four chemicals from schedule 2080, 1,7-dimethylxanthine, acetaminophen, caffeine, and dehydronifedipine, were detected in more than one laboratory reagent-water blank sample, but all concentrations were much less than the laboratory reporting level. Four compounds from schedule 2434, 17-*beta*-estradiol, cholesterol, estriol, and *trans*-diethylstilbestrol, were detected in more than one laboratory reagent-water blank sample; however, only the concentration of cholesterol was greater than the laboratory reporting level. No chemicals from schedule LCAB were detected in laboratory reagent-water blanks. Chemicals that were detected in one laboratory-blank sample are not included in table 5 because these chemicals were not used in the data censoring process, which is described in the “Data Censoring Criteria” section of this report.

## Field Quality Assurance

Quality-assurance samples were collected consistent with the USGS NFM (U.S. Geological Survey, variously dated). The collected field quality-assurance samples included blanks, replicates, and groundwater matrix spikes.

Potential contamination of water samples during sample collection, processing, and laboratory analysis was assessed with field equipment-blank samples. Field equipment-blank samples were prepared at selected sites before collecting the environmental sample. Field equipment-blank samples were prepared by processing high-performance liquid chromatography organic-free grade water (certified by the NWQL to be free of the chemicals of interest) through the same equipment used to collect and process field samples. Sixteen field equipment-blank samples were collected and analyzed to assess contamination introduced during sample collection, processing, and laboratory analysis for water samples. From schedule 1433, 22 chemicals were detected in one or more field equipment-blank samples (table 6), and detections in field equipment-blank samples were not censored because of detections in laboratory-blank samples. Most chemicals were detected in field equipment-blank samples at concentrations less than or slightly greater than the laboratory reporting level. The chemical DEET, which is an active ingredient in insect repellent, was most frequently detected (11 of 16 field equipment blank samples). DEET and phenol were the only compounds detected in more than one field equipment-blank sample at a concentration much greater than the laboratory reporting level (table 6). Both of the elevated DEET concentrations were in blank samples collected during 2010, the first full field season. Proper field protocols regarding the use of DEET during field

**Table 5.** Chemicals detected in more than one laboratory reagent-water blank sample related to sample sets for this study, 2009–12.

[LRL, laboratory reporting level; µg/L, micrograms per liter; E, estimated; ng/L, nanograms per liter]

Analytical schedule	Chemical	LRL	LRL units	Number of blanks	Number of detections	Censoring level <sup>1</sup>
1433	1-Methylnaphthalene	0.022	µg/L	44	6	E0.0031
1433	2-Methylnaphthalene	0.036	µg/L	44	14	E0.0068
1433	3- <i>beta</i> -Coprostanol	1.8	µg/L	44	6	E0.15
1433	4-Nonylphenol (all isomers)	2	µg/L	44	17	E0.1
1433	4-Nonylphenol diethoxylate	5	µg/L	44	6	E0.62
1433	4- <i>tert</i> -Octylphenol	0.14	µg/L	44	5	E0.004
1433	4- <i>tert</i> -Octylphenol diethoxylate	1	µg/L	44	2	E0.04
1433	4- <i>tert</i> -Octylphenolmonoethoxylate	1	µg/L	44	7	E0.07
1433	9,10-Anthraquinone	0.16	µg/L	44	5	E0.028
1433	Acetophenone	0.4	µg/L	44	37	E0.1
1433	Benzophenone	0.08	µg/L	44	12	E0.009
1433	<i>beta</i> -Sitosterol	4	µg/L	44	10	E0.4
1433	<i>beta</i> -Stigmastanol	2.6	µg/L	44	5	E0.13
1433	Caffeine	0.06	µg/L	44	6	E0.007
1433	Camphor	0.044	µg/L	44	3	E0.0057
1433	Cholesterol	2	µg/L	44	9	E0.3
1433	D-Limonene	0.08	µg/L	44	5	E0.019
1433	Fluoranthene	0.024	µg/L	44	2	E0.0009
1433	Hexahydrohexamethylcyclopentabenzopyran (HHCB)	0.052	µg/L	44	3	E0.003
1433	Isopropylbenzene	0.3	µg/L	44	3	E0.002
1433	Methyl salicylate	0.044	µg/L	44	9	E0.0063
1433	Naphthalene	0.04	µg/L	44	31	E0.0068
1433	<i>p</i> -Cresol	0.08	µg/L	44	10	E0.012
1433	Phenanthrene	0.032	µg/L	44	8	E0.0014
1433	Phenol	0.16	µg/L	44	30	E0.054
1433	Prometon	0.012	µg/L	44	4	0.018
1433	Pyrene	0.042	µg/L	44	7	E0.0012
1433	Triclosan	0.2	µg/L	44	6	E0.034
1433	Triphenyl phosphate	0.12	µg/L	44	2	E0.003
1433	Tris(2-butoxyethyl) phosphate	0.8	µg/L	44	3	E0.36
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	0.1	µg/L	44	3	E0.016
2080	1,7-Dimethylxanthine	0.1	µg/L	52	5	E0.013
2080	Acetaminophen	0.12	µg/L	52	3	E0.006
2080	Caffeine	0.06	µg/L	52	5	E0.011
2080	Dehydronifedipine	0.08	µg/L	52	3	E0.0005
2434	17- <i>beta</i> -Estradiol	0.8	ng/L	44	2	E0.059
2434	Cholesterol	200	ng/L	44	2	209
2434	Estriol	2	ng/L	44	3	E0.19
2434	<i>trans</i> -Diethylstilbestrol	0.8	ng/L	44	2	E0.14

<sup>1</sup> Second highest concentration detected in laboratory-blank samples.

**Table 6.** Concentrations of chemicals detected in field equipment-blank samples, 2009–12.

[E, estimated; LRL, laboratory reporting level; µg/L, micrograms per liter]

Analytical schedule	Chemical	LRL (µg/L)	Number of blanks	Number of detections	Maximum detection
1433	1-Methylnaphthalene	0.022	16	6	0.022
1433	2,6-Dimethylnaphthalene	0.06	16	1	E0.0083
1433	2-Methylnaphthalene	0.036	16	7	0.043
1433	3-Methyl-1 <i>H</i> -indole	0.036	16	1	E0.0039
1433	4- <i>tert</i> -Octylphenol	0.14	16	1	E0.0071
1433	9,10-Anthraquinone	0.16	16	1	E0.017
1433	Acetophenone	0.4	16	1	0.78
1433	Anthracene	0.01	16	2	E0.0064
1433	Benzophenone	0.08	16	1	E0.021
1433	Camphor	0.044	16	1	E0.02
1433	Carbazole	0.03	16	3	0.032
1433	Isophorone	0.032	16	6	E0.014
1433	Isoquinoline	0.046	16	1	0.15
1433	Menthol	0.32	16	4	E0.054
1433	Methyl salicylate	0.044	16	5	E0.014
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET), 2010 samples	0.06	6	4	3.8
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET), 2011–12 samples	0.06	10	7	0.19
1433	Naphthalene	0.04	16	9	0.19
1433	<i>p</i> -Cresol	0.08	16	2	E0.062
1433	Phenanthrene	0.016	16	7	0.026
1433	Phenol	0.16	16	7	1.1
1433	Tris(1,3-dichloroisopropyl) phosphate (FYROL FR 2)	0.16	16	2	E0.11
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	0.1	16	3	E0.063
2080	Acetaminophen	0.12	16	1	E0.0247
2080	Codeine	0.046	16	1	E0.0029
2080	Trimethoprim	0.034	16	1	E0.005
LCAB	Ormetoprim	0.005	16	1	0.012
LCAB	Trimethoprim	0.005	16	1	0.005

work was clarified before sampling began in 2011. Therefore, the elevated concentrations of DEET are only relevant to the samples collected in 2009–10, and not samples from subsequent years. Three chemicals from schedule 2080, acetaminophen, codeine, and trimethoprim, were each detected in one field equipment-blank sample at concentrations much less than the laboratory reporting level. Two chemicals from the LCAB schedule, ormetoprim and trimethoprim, were detected at concentrations near the laboratory reporting level.

Replicate samples were used to quantify the variability of detection and corresponding concentrations that result from sample processing (sample splitting, filtration, and transport) and laboratory techniques. Twelve replicate samples were collected for this study. The replicate sample pairs consisted of a primary environmental field sample and a sequential replicate sample collected immediately after the environmental sample;

the two samples should be nearly identical in composition. Reported concentrations, which were near or below laboratory reporting levels, were censored as described in the “Data Censoring Criteria” section of this report. No sample pairs had chemical detections in both samples that were not censored by detections in laboratory or field blanks. The concentration variability resulting from sample processing and analysis could not be evaluated.

Matrix interference was assessed by matrix spikes in groundwater samples. Groundwater matrix spike samples with associated duplicate samples were collected at six wells and shipped to the NWQL and OGRL for assessment of matrix interference. The samples were spiked at the laboratory. Most chemicals had recoveries in groundwater matrix spike samples (table 7) that were similar to recoveries for laboratory reagent-water spike samples (table 3). The calculated recoveries

**Table 7.** Summary of field matrix spike recoveries from groundwater samples collected in Minnesota, 2009–12.

Analytical schedule	Chemical	Number of matrix spikes analyzed	Average percent recovery	Standard deviation of percent recovery
1433	1,4-Dichlorobenzene	10	66	8
1433	1-Methylnaphthalene	10	72	8
1433	2-Methylnaphthalene	10	71	6
1433	2,6-Dimethylnaphthalene	10	66	9
1433	3- <i>beta</i> -Coprostanol	10	76	15
1433	3-Methyl-1 <i>H</i> -indole	10	82	19
1433	3- <i>tert</i> -Butyl-4-hydroxyanisole	9	36	25
1433	4-Cumylphenol	10	88	9
1433	4- <i>n</i> -Octylphenol	10	67	8
1433	4-Nonylphenol (all isomers)	10	74	10
1433	4-Nonylphenol diethoxylate	10	85	13
1433	4- <i>tert</i> -Octylphenol	10	85	12
1433	4- <i>tert</i> -Octylphenol diethoxylate	10	102	26
1433	4- <i>tert</i> -Octylphenolmonoethoxylate	10	82	9
1433	5-Methyl-1 <i>H</i> -benzotriazole	8	69	16
1433	9,10-Anthraquinone	10	100	9
1433	Acetophenone	10	100	9
1433	Acetyl-hexamethyl-tetrahydro-naphthalene (AHTN)	10	84	9
1433	Anthracene	10	84	5
1433	Benzo[ <i>a</i> ]pyrene	10	78	10
1433	Benzophenone	10	100	6
1433	<i>beta</i> -Sitosterol	10	69	16
1433	<i>beta</i> -Stigmastanol	9	71	16
1434	Bromacil	10	90	22
1433	Caffeine	10	93	8
1433	Camphor	10	90	8
1433	Carbaryl	10	64	23
1433	Carbazole	10	95	7
1433	Chlorpyrifos	10	66	15
1433	Cholesterol	10	74	16
1433	Cotinine	10	84	7
1433	Diazinon	10	87	9
1433	D-Limonene	10	40	10
1433	Fluoranthene	10	87	7
1433	Hexahydrohexamethylcyclopentabenzopyran (HHCB)	10	84	11
1433	Indole	8	73	17
1433	Isoborneol	10	86	6
1433	Isophorone	10	91	10
1433	Isopropylbenzene	10	64	14
1433	Isoquinoline	6	81	7
1433	Menthol	10	86	8
1433	Metalaxyl	10	97	4
1433	Methyl salicylate	10	95	7

**Table 7.** Summary of field matrix spike recoveries from groundwater samples collected in Minnesota, 2009–12.—Continued

Analytical schedule	Chemical	Number of matrix spikes analyzed	Average percent recovery	Standard deviation of percent recovery
1433	Metolachlor	10	89	7
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET )	18	92	3
1433	Naphthalene	10	85	17
1433	<i>p</i> -Cresol	10	81	21
1433	Phenanthrene	10	89	10
1433	Phenol	10	97	11
1434	Prometon	10	91	11
1433	Pyrene	10	86	5
1433	Tetrachloroethene	10	28	12
1433	Tribromomethane	10	65	12
1433	Tributyl phosphate	10	93	14
1433	Triclosan	10	77	8
1433	Triethyl citrate	10	92	30
1433	Triphenyl phosphate	10	69	16
1433	Tris(1,3-dichloroisopropyl) phosphate (FYROL FR 2)	10	90	6
1433	Tris(2-butoxyethyl) phosphate	10	85	8
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	10	88	8
2080	1,7-Dimethylxanthine	11	84	38
2080	Acetaminophen	11	87	59
2080	Albuterol	12	56	33
2080	Caffeine	11	89	39
2080	Carbamazepine	12	59	41
2080	Codeine	10	78	33
2080	Cotinine	12	62	30
2080	Dehydronifedipine	12	80	46
2080	Diltiazem	10	36	24
2080	Diphenhydramine	11	42	31
2080	Sulfamethoxazole	9	46	44
2080	Thiabendazole	8	59	29
2080	Trimethoprim	10	70	40
2080	Warfarin	11	67	40
2434	3- <i>beta</i> -Coprostanol	8	91	10
2434	4-Androstene-3,17-dione	8	92	4
2434	11-Ketotestosterone	8	93	12
2434	17- <i>alpha</i> -Estradiol	8	101	7
2434	17- <i>alpha</i> -Ethinyl estradiol	8	91	2
2434	17- <i>beta</i> -Estradiol	8	97	4
2434	Bisphenol A	16	84	10
2434	Cholesterol	8	93	8
2434	<i>cis</i> -Androsterone	8	103	9
2434	Dihydrotestosterone	8	90	10
2434	Epitestosterone	8	96	7
2434	Equilenin	8	81	10

**Table 7.** Summary of field matrix spike recoveries from groundwater samples collected in Minnesota, 2009–12.—Continued

Analytical schedule	Chemical	Number of matrix spikes analyzed	Average percent recovery	Standard deviation of percent recovery
2434	Equilin	8	93	28
2434	Estriol	8	93	5
2434	Estrone	8	96	6
2434	Mestranol	8	94	4
2434	Norethindrone	8	89	2
2434	Progesterone	8	94	16
2434	Testosterone	8	94	6
2434	<i>trans</i> -Diethylstilbestrol	8	85	7
LCAB	4-Epioxytetracycline	18	143	99
LCAB	Anhydroerthromycin	18	114	28
LCAB	Azithromycin	18	109	54
LCAB	Carbamazepine	18	92	31
LCAB	Chloramphenicol	18	117	50
LCAB	Chlorotetracycline	18	91	31
LCAB	Ciprofloxacin	18	109	47
LCAB	Doxycycline	18	89	76
LCAB	Enrofloxacin	18	63	31
LCAB	Erythromycin	18	140	43
LCAB	Ibuprofen	18	107	57
LCAB	Lincomycin	18	233	164
LCAB	Lomefloxacin	18	63	33
LCAB	Norfloxacin	18	65	33
LCAB	Ofloxacin	18	58	29
LCAB	Ormetoprim	18	103	32
LCAB	Oxytetracycline	18	133	70
LCAB	Roxithromycin	18	122	47
LCAB	Sarafloxacin	18	54	27
LCAB	Sulfachloropyridazine	18	109	31
LCAB	Sulfadiazine	18	91	34
LCAB	Sulfadimethoxine	18	83	33
LCAB	Sulfamethazine	18	120	35
LCAB	Sulfamethoxazole	18	112	34
LCAB	Sulfathiazole	18	83	23
LCAB	Trimethoprim	18	95	51
LCAB	Tylosin	18	117	38
LCAB	Virginiamycin	18	212	252

<sup>1</sup> Results from one site deleted from calculation because of high chemical concentration in associated environmental sample that greatly exceeded spiked concentration of this chemical.

in the groundwater matrix spike samples for the chemicals DEET (schedule 1433) and bisphenol A (schedule 2434) for the sample collected on June 6, 2012, from station number 451855093195901 (map number 54, table 1, fig. 2) were not included in the average matrix spike recovery calculations in table 7 because these two chemicals were detected in the associated environmental sample at concentrations much greater than the laboratory reporting level and the spiked concentration. These high concentrations in the environmental sample make spike recovery calculations for the associated groundwater spike sample highly uncertain. In contrast to the groundwater matrix spike results, the laboratory surrogate spikes in these environmental samples had normal recoveries, providing additional evidence that the recovery calculations for the groundwater matrix spike are erroneous for these samples with high environmental concentrations of DEET and bisphenol A. From schedule LCAB, the compounds 4-epioxytetracycline, lincomycin, and virginiamycin had average recoveries of more than 140 percent in the field matrix spike samples (table 7). The chemicals 4-epioxytetracycline and virginiamycin were not detected in any environmental samples, whereas lincomycin was detected in four environmental samples. Reported concentrations for lincomycin in environmental samples that are less than 10 times greater than the laboratory reporting level were flagged with an “E” to indicate that these reported concentrations are estimated (potential positive bias) based on the high matrix spike recoveries. In addition, the fluoroquinolones (ciprofloxacin, enrofloxacin, lomefloxacin, norfloxacin, ofloxacin, and sarafloxacin) were not recovered in 3 of the 18 groundwater matrix spike samples for the LCAB schedule, and in 1 of these 3 samples, the macrolides (azithromycin, erythromycin, erythromycin-H<sub>2</sub>O, roxithromycin, tylosin, and virginiamycin) and 3 other antibiotics (epi-chlorotetracycline, epi-iso-chlorotetracycline, and iso-chlorotetracycline) also were not recovered. These groundwater matrix spike results indicate that some of the groundwater matrices were rapidly sorbing, chelating, or degrading selected chemicals. All of these processes would result in a reduction of the measured chemical concentration in an environmental sample, if the compound had been present in the sample initially. These processes, therefore, introduce potential negative bias for detecting and reporting these chemicals.

## Data Censoring Criteria

The USGS Office of Water Quality issued technical guidance to the NWQL in 2011 for “flagging” environmental analytical results that may have been affected by laboratory contamination (U.S. Geological Survey Office of Water Quality, 2011). In this report, consistent with the technical guidance from the USGS Office of Water Quality, concentrations in environmental samples that are less than 10 times greater than the second highest concentration detected in laboratory-blank samples (table 5, Censoring level) or the maximum concentration in field-blank samples (table 6), whichever is

larger, were considered potentially affected by laboratory or field procedure contamination. Because of the large number of laboratory-blank samples, there is 90-percent confidence that 95 percent of detections will be less than the censoring level. Reported environmental concentrations that were less than 10 times greater than the criteria above were flagged with a “v” code and were not counted as environmental detections for data analyses in this report. The results for DEET were censored in two groups: 2010 data as one group compared to field blank results for only 2010, and 2011–12 results grouped together and compared to all field blank results for 2011–12. All other chemicals were censored together using all field blanks for 2010–12. All analytical results for the environmental samples are provided in the appendix tables (tables 1–1 through 1–5, Microsoft® Excel spreadsheet) as reported in the National Water Information System database (U.S. Geological Survey, 2013); however, sample results potentially affected by laboratory or field blank contamination (potential false positives) were flagged as such with a “v” code in the supplemental tables in this report and were not counted as detections in the data analyses.

The sensitivity of the various analytical methods used to analyze CECs for this study can affect the calculated detection frequencies of the chemicals. The CECs with low laboratory reporting levels likely would be detected more frequently than those with high laboratory reporting levels. Laboratory reporting levels for the CECs analyzed for this study range from 0.0008 µg/L (0.8 nanograms per liter, ng/L) to 5 µg/L (table 2). No additional data censoring was applied to account for the different laboratory reporting levels among the CECs.

## Presence of Chemicals of Emerging Concern in Ambient Groundwater

Physical properties and concentrations of CECs in samples of ambient groundwater collected from wells in urbanized areas of Minnesota during 2009–12 are presented in this section of the report. Concentrations of detected CECs are compared to health-based water-quality standards and benchmarks, and potential environmental implications are described.

## Physical Properties

Physical properties (dissolved oxygen, pH, specific conductance, and water temperature) were collected in the field using a water-quality multiparameter meter sonde at all wells before collecting water samples for analyses of CECs. These field measurements are summarized in table 8, and detailed results are provided in appendix table 1–1. Dissolved oxygen (DO) concentrations in drinking water wells screened in glacial aquifers averaged 1.77 milligrams per liter (mg/L), which is lower than the average DO concentration of 5.22 mg/L for monitoring wells screened in glacial aquifers. Wells completed

**Table 8.** Summary of well depths and physical properties in unfiltered groundwater, Minnesota, 2009–12.

[mg/L, milligrams per liter;  $\mu\text{S}/\text{cm}$ , microsiemens per centimeter at 25 degrees Celsius;  $^{\circ}\text{C}$ , degrees Celsius; ft BGS, feet below ground surface]

Well description	Statistic	Number of wells	Dissolved oxygen (mg/L)	pH (standard units)	Specific conductance ( $\mu\text{S}/\text{cm}$ )	Water temperature ( $^{\circ}\text{C}$ )	Well depth (ft BGS)
Glacial aquifers							
All glacial aquifer wells	Average	105	4.63	7.01	719	9.89	30
	Standard deviation	105	3.80	0.78	619	1.73	20
	Minimum	105	0	5.02	57	6.29	9
	Maximum	105	11.65	9.57	4,011	14.43	112
Drinking water wells <sup>1</sup>	Average	17	1.77	7.03	1,052	10.31	61
Monitoring wells	Average	88	5.22	7.00	650	9.80	23
Bedrock aquifers							
All bedrock aquifer wells	Average	13	4.42	7.20	659	10.92	188
	Standard deviation	13	4.22	0.24	228	0.73	83
	Minimum	13	0.00	6.86	413	9.89	49
	Maximum	13	11.33	7.70	1,249	12.37	285
Drinking water wells <sup>2</sup>	Average	11	4.36	7.25	671	10.86	209
Monitoring wells	Average	2	4.77	6.94	594	11.24	70
All wells combined							
All wells	Average	118	4.60	7.03	713	10.00	47
	Standard deviation	118	3.83	0.74	589	1.68	59
	Minimum	118	0	5.02	57	6.29	9
	Maximum	118	11.65	9.57	4,011	14.43	285

<sup>1</sup> All domestic wells.

<sup>2</sup> Ten domestic wells and one public water system well.

in bedrock aquifers also had a higher average DO concentration (4.42 mg/L) than drinking water wells screened in glacial aquifers. Most measured pH values were near neutral, with an average pH value of 7.03 standard units (standard deviation of 0.74 standard units) for all well types screened in either glacial or bedrock aquifers; pH values ranged from 5.02 to 9.57 standard units. Measured water temperature values ranged from 6.29 to 14.43  $^{\circ}\text{C}$ ; the average groundwater temperature was 10.00  $^{\circ}\text{C}$ , with a standard deviation of 1.68  $^{\circ}\text{C}$ . For wells screened in glacial aquifers, the specific conductance for drinking water wells averaged 1,052 microsiemens per centimeter at 25  $^{\circ}\text{C}$  ( $\mu\text{S}/\text{cm}$ ), compared to an average specific conductance of 650  $\mu\text{S}/\text{cm}$  for monitoring wells. For wells completed in bedrock aquifers, the average specific conductance was 659  $\mu\text{S}/\text{cm}$ . Measurements of groundwater physical properties for this study are similar to those for a statewide groundwater study completed by the MPCA in the 1990s (Minnesota Pollution Control Agency, 1998).

Specific conductance is a measure of the concentration of dissolved solids in water, and higher specific conductance measurements indicate that the groundwater being measured had a relatively longer contact time with the aquifer minerals allowing for more chemical changes to take place (Winter and

others, 1998). Similarly, lower DO concentrations indicate relatively longer residence time and isolation from atmospheric conditions. In this study, the specific conductance was higher and the DO concentration was lower for drinking water wells screened in the glacial aquifer than for monitoring wells screened in the glacial aquifer and for wells completed in bedrock aquifers, even though the sampled wells in the glacial aquifers generally were shallower than the sampled wells in the bedrock aquifers. These results indicate a relatively longer flow path of water to the glacial aquifer drinking water wells compared to other wells sampled in this study.

### Chemicals of Emerging Concern in Groundwater

During this study, 38 of 127 CECs analyzed were detected among all water samples collected (table 9). Three of the detected CECs (carbamazepine, cotinine, and sulfamethoxazole), however, were analyzed using two different analytical methods (2080 and 1433 or LCAB), so 35 distinct chemicals were detected. The number of detections of CECs in individual water samples ranged from 0 to 10 (table 10, tables 1–2 through 1–5; Erickson, 2012). The three wells in proximity

**Table 9.** Detected chemicals and maximum concentration of contaminants of emerging concern analyzed in groundwater samples, Minnesota, 2009–12.

[µg/L, micrograms per liter; E, estimated; ng/L, nanograms per liter]

Analytical schedule	Analyte	Number of detections	Maximum detection	Laboratory reporting level	Unit
1433	1,4-Dichlorobenzene	3	0.18	0.04	µg/L
1433	3-Methyl-1 <i>H</i> -indole	1	0.087	0.036	µg/L
1433	4-Cumylphenol	2	E0.14	0.06	µg/L
1433	4- <i>tert</i> -Octylphenol	2	0.19	0.14	µg/L
1433	5-Methyl-1 <i>H</i> -benzotriazole	1	0.16	1.2	µg/L
1434	Bromacil	2	0.50	0.36	µg/L
1433	Caffeine	1	0.10	0.06	µg/L
1433	Camphor	2	0.890	0.044	µg/L
1433	Carbazole	2	2.9	0.03	µg/L
1433	Cotinine	3	0.030	0.8	µg/L
1433	<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET), 2011-12	1	7.9	0.06	µg/L
1433	Fluoranthene	2	0.034	0.024	µg/L
1433	Tris(2-chloroethyl) phosphate (FYROL CEF)	2	1.4	0.1	µg/L
1433	HHCB	1	0.057	0.052	µg/L
1433	Indole	1	0.24	0.08	µg/L
1433	Isopropylbenzene	3	0.83	0.3	µg/L
1433	Metolachlor	3	E0.022	0.028	µg/L
1433	<i>p</i> -Cresol	1	2.9	0.08	µg/L
1433	Pyrene	3	0.044	0.042	µg/L
1433	Tetrachloroethene	1	0.18	0.12	µg/L
1433	Tributyl phosphate	5	0.98	0.16	µg/L
1433	Triethyl citrate	1	E0.01	0.16	µg/L
1433	Triphenyl phosphate	1	E0.037	0.12	µg/L
2080	Acetaminophen	1	0.75	0.12	µg/L
2080	Carbamazepine	2	0.122	0.06	µg/L
2080	Cotinine	3	0.030	0.038	µg/L
2080	Diphenhydramine	5	0.016	0.058	µg/L
2080	Sulfamethoxazole	<sup>1</sup> 7	E0.1126	0.091	µg/L
2434	3- <i>beta</i> -Coprostanol	1	249	200	ng/L
2434	Bisphenol A	6	4,411	100	ng/L
2434	<i>cis</i> -Androsterone	1	2.04	0.8	ng/L
2434	Mestranol	1	0.71	0.8	ng/L
LCAB	Azithromycin	5	0.023	0.005	µg/L
LCAB	Carbamazepine	2	0.122	0.005	µg/L
LCAB	Lincomycin	4	0.110	0.005	µg/L
LCAB	Sulfadiazine	2	0.021	0.005	µg/L
LCAB	Sulfamethazine	3	0.040	0.005	µg/L
LCAB	Sulfamethoxazole	<sup>1</sup> 13	0.171	0.005	µg/L

<sup>1</sup> Sulfamethoxazole detected by one or both analytical methods in 14 samples.

**Table 10.** Detection frequency of contaminants of emerging concern by use of water, aquifer type, and land use or monitoring type, Minnesota, 2009–12.

Well description	Number of wells	Number of samples	Number of samples with detections	Percentage of samples with detections	Number of detections	Maximum number of detections in one well
Use of water						
Domestic/public (drinking water) <sup>1</sup>	28	30	16	53	24	5
Monitoring	90	93	27	29	76	10
Aquifer type						
Glacial	105	110	35	32	88	10
Bedrock	13	13	8	62	12	5
Land use or monitoring type						
Commercial/industrial	13	13	4	31	8	3
Deep well	33	34	16	47	22	5
Landfill monitoring	3	5	5	100	34	10
Septic residential	30	31	7	23	16	4
Sewered residential	24	25	8	32	16	5
Undeveloped	15	15	3	20	4	2
All wells						
All wells	118	123	43	35	100	10

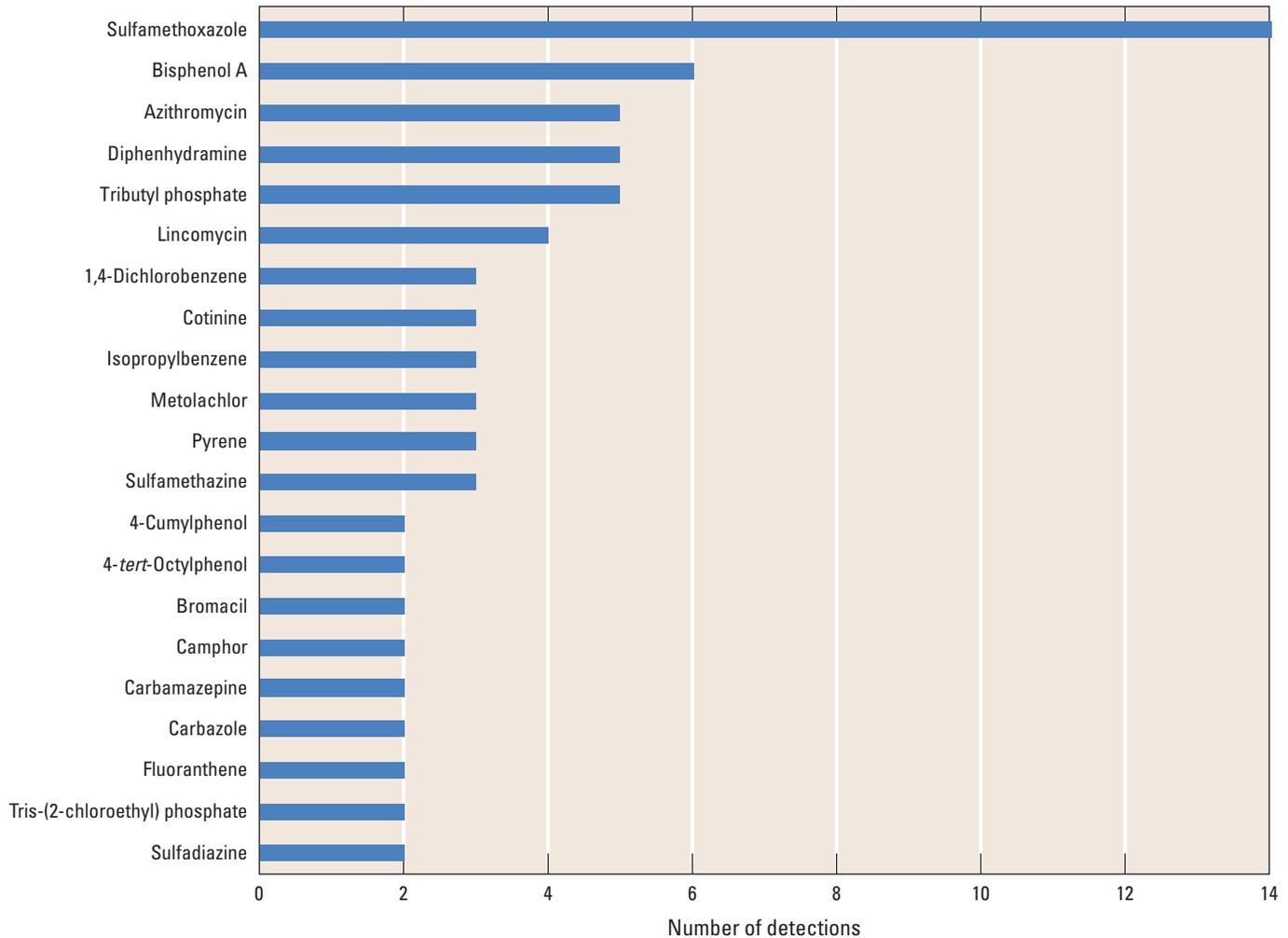
<sup>1</sup> One public system well and 27 domestic wells.

to landfills had the most CEC detections (table 10). One or more CECs were detected in a total of 43 samples (35 percent) (table 10); no CECs were detected in 80 samples.

The antibiotic sulfamethoxazole was the most frequently detected CEC, detected in a total of 14 of 123 samples (11.4 percent) by one or both analytical methods that include sulfamethoxazole as an analyte (table 9; table 1–3 and table 1–5). Other CECs detected in more than one sample were azithromycin, carbamazepine, diphenhydramine, lincomycin, sulfadiazine, and sulfamethazine (human and animal-use pharmaceuticals); camphor (flavor, fragrance); 4-cumylphenol and 4-*tert*-octylphenol (nonionic detergent metabolites); bromacil (herbicide active ingredient); carbazole and metolachlor (pesticide active ingredients); tris(2-chloroethyl) phosphate (plasticizer and flame retardant); cotinine (nicotine metabolite); bisphenol A (plastic and resin component); fluoranthene and pyrene (asphalt components, combustion products), 1,4-dichlorobenzene (moth repellant); tributyl phosphate (corrosion inhibitor); and isopropylbenzene (fuel and paint thinner component) (fig. 3; table 2; tables 1–2 through 1–5; Erickson, 2012). The hormone or hormone metabolites *cis*-androsterone and mestranol were each detected in one sample (table 1–4). Also detected in one sample each were a fecal indicator and a fecal stench component, 3-*beta*-coprostanol and 3-methyl-1*H*-indole, respectively. Chemicals detected in one sample and found in products used for ingestion or topical use were acetaminophen, caffeine, DEET, hexahydrohexamethylcyclopentabenzopyran (HHCb), and triethyl citrate. The chemicals indole, 5-methyl-1*H*-benzotriazole, *p*-cresol, tetrachloroethene, and triphenyl phosphate, which are components of

different types of industrial products, also were each detected in one sample. As noted previously in the “Data Censoring Criteria” section, concentration data were not censored to account for differences in the laboratory reporting levels for the CECs (table 2); thus the detection frequencies presented in this report are biased towards CECs with low laboratory reporting levels.

In a June 2012 sample, DEET was detected at the highest concentration of any CEC during the study at 7.9 micrograms per liter ( $\mu\text{g/L}$ ), at site 451855093195901 (map number 54, fig. 2, table 1), which is in proximity to a closed landfill. Bisphenol A was the second most frequently detected CEC, detected in 6 of 123 samples. Site 451855093195901 (map number 54, fig. 2, table 1), which is in proximity to a closed landfill, had the most CECs detected (10 chemicals) in one sample. The five samples from the three wells in proximity to closed landfills (denoted as “Landfill monitoring” in table 1) had 34 of the 100 total CEC detections, and these samples had the highest frequency of CEC detection (table 10; tables 1–2 through 1–5; Erickson, 2012). Deep wells and wells located in sewered residential land use areas had higher percentages of samples with CEC detections than wells located in undeveloped and septic residential land uses (table 10). Wells classified as deep were primarily bedrock drinking water wells. Samples from wells used as drinking water sources had a higher percentage of CEC detections than did samples from monitoring wells; however, only 24 percent of the wells sampled were drinking water wells. The spatial distribution of CEC detections in sampled wells is shown in figure 4.



**Figure 3.** Number of detections of selected contaminants of emerging concern in groundwater samples from urbanized areas of Minnesota, 2009–12.

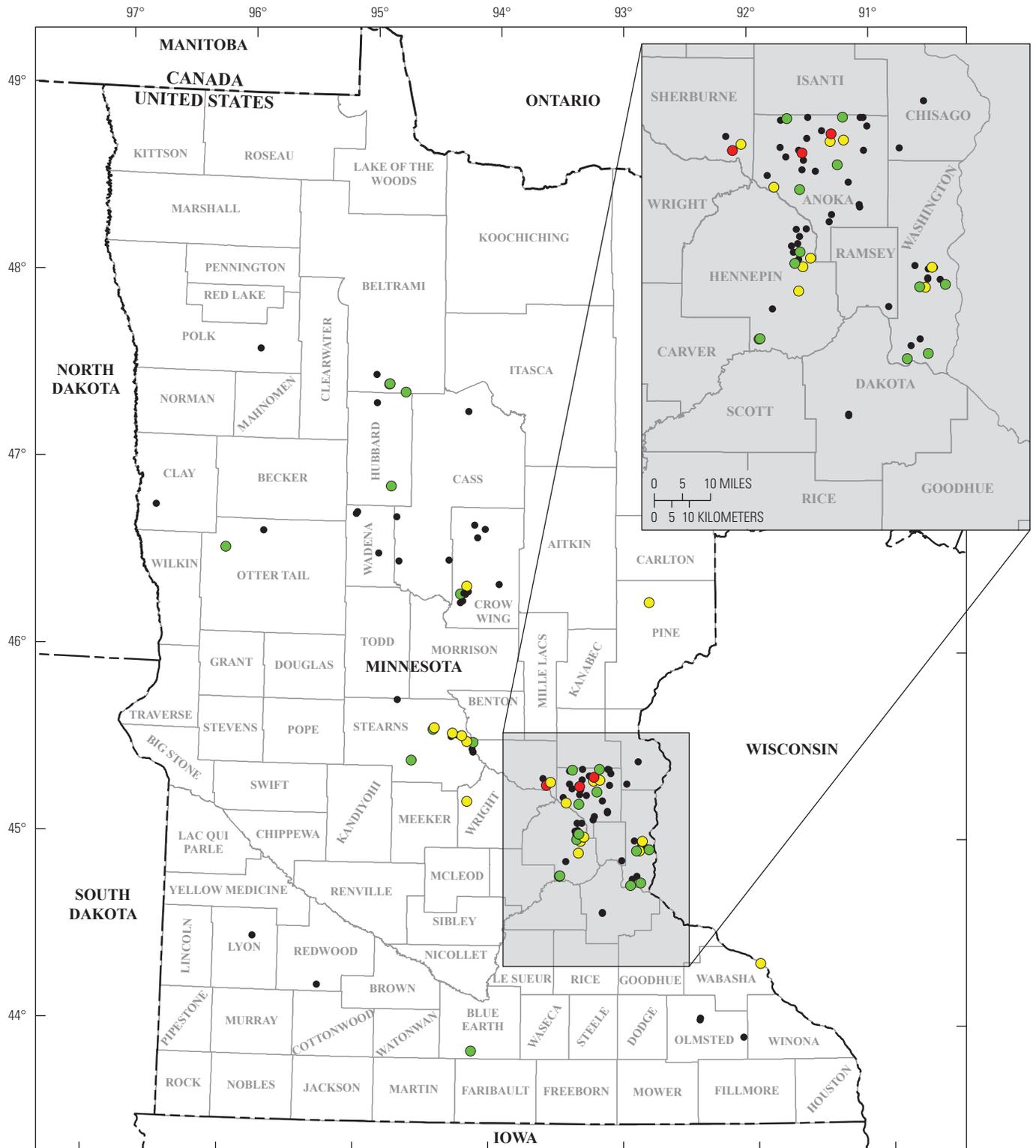
### Comparison of Concentrations with Health-Based Water-Quality Standards and Benchmarks

Of the 127 chemicals analyzed for this study, only 28 have established enforceable or non-enforceable health-based water-quality standards or benchmarks (table 11). The two types of enforceable water-quality standards are Minnesota Department of Health (MDH) Health Risk Limits (HRLs; Minnesota Department of Health, 2013) and U.S. Environmental Protection Agency (EPA) Maximum Contaminant Levels (MCLs) (U.S. Environmental Protection Agency, 2013). The non-enforceable water-quality standards are MDH Health-Based Values (HBVs) (Minnesota Department of Health, 2013) and USGS Health-Based Screening Levels (HBSLs) (Toccalino and others, 2012).

The HRLs are enforceable, promulgated guidance values adopted through a formal rulemaking process authorized in the State of Minnesota 1989 Groundwater Protection Act

(State of Minnesota, 2013). The HBVs are non-enforceable but are developed as interim guidance until MDH can adopt an HRL through rulemaking. The HBVs meet the same data requirements as HRLs. If a contaminant has been detected in groundwater, then HBVs for water may become HRLs at the time that MDH next amends the Health Risk Limits for Groundwater rule (Minnesota Department of Health, 2013). The MCLs are the EPA enforceable standards that apply to public water systems, which protect public health by limiting the levels of contaminants in drinking water. The HBSLs are non-enforceable benchmark concentrations of unregulated contaminants in water that may be of potential concern for human health, if exceeded. The HBSLs were developed by the USGS in collaboration with the EPA and others (Toccalino and others, 2012).

Fourteen of the 35 detected compounds have established health-based water-quality standards, whereas 21 detected compounds have no established standard or benchmark. All detections in this study were less than health-based water-quality standards. Although most detections were well below



Base from Minnesota Department of Natural Resources, 1993, 1:24,000 and 1:100,000 digital data  
 Universal Transverse Mercator projection, zone 15

0 25 50 75 100 MILES  
 0 25 50 75 100 KILOMETERS

**EXPLANATION**  
 Number of detections  
 ● 0  
 ● 1  
 ● 2 to 5  
 ● 6 to 10

**Figure 4.** Sampled well locations and number of detection of contaminants of emerging concern in groundwater samples from urbanized areas of Minnesota, 2009–12.

**Table 11.** Health-based water quality standards and benchmarks<sup>1</sup> for chemicals analyzed for this study.

[ $\mu\text{g/L}$ , micrograms per liter; MDH, Minnesota Department of Health; HRL, Health Risk Limit, subscript denotes year when value was established<sup>2</sup>; USGS, U.S. Geological Survey; HBSL, Health-Based Screening Level<sup>3</sup>; HBV, Health-Based Value, subscript denotes year when value was established<sup>2</sup>; EPA, U.S. Environmental Protection Agency; MCL, Maximum Contaminant Level<sup>4</sup>]

Chemical	Standard or benchmark ( $\mu\text{g/L}$ )	Standard source	Detected in this study? <sup>1</sup>
1,4-Dichlorobenzene	10	MDH HRL <sub>1994</sub>	Yes
2-Methylnaphthalene	30	USGS HBSL	No
Acetaminophen	200	MDH HBV <sub>2011</sub>	Yes
Acetophenone	700	USGS HBSL	No
Acetyl-hexamethyl-tetrahydro-naphthalene (AHTN)	20	MDH HRL <sub>2013</sub>	No
Anthracene	2000	MDH HRL <sub>1993</sub>	No
Benzo[ <i>a</i> ]pyrene	0.2	EPA MCL	No
Benzo[ <i>a</i> ]pyrene	0.06	MDH HBV <sub>2012</sub>	No
Bisphenol A	20	MDH HBV <sub>2014</sub>	Yes
Carbamazepine	40	MDH HRL <sub>2013</sub>	Yes
Carbaryl	40	USGS HBSL	No
Chlorpyrifos	0.6	MDH HBV <sub>2013</sub>	No
<i>N,N</i> -Diethyl- <i>meta</i> -toluamide (DEET)	200	MDH HRL <sub>2013</sub>	Yes
Diazinon	1	USGS HBSL	No
Fluoranthene	300	MDH HRL <sub>1993</sub>	Yes
Isophorone	100	MDH HRL <sub>1993</sub>	No
Isopropyl benzene (cumene)	300	MDH HRL <sub>1993</sub>	Yes
Metalaxyl	500	USGS HBSL	No
Methanol	3000	MDH HRL <sub>1994</sub>	No
Metolachlor	300	MDH HRL <sub>2011</sub>	Yes
Naphthalene	70	MDH HRL <sub>2013</sub>	No
<i>p</i> -Cresol	3	MDH HRL <sub>1994</sub>	Yes
Phenol	2000	USGS HBSL	No
Pyrene	200	MDH HRL <sub>1993</sub>	Yes
Sulfamethazine	100	MDH HBV <sub>2013</sub>	Yes
Sulfamethoxazole	100	MDH RAA <sub>2013</sub>	Yes
Tetrachloroethene	5	EPA MCL	Yes
Tris(2-chloroethyl) phosphate (FYROL CEF)	5	MDH HRL <sub>2013</sub>	Yes
Triclosan	50	MDH HBV <sub>2014</sub>	No

<sup>1</sup>Twenty-one analytes detected in this study do not have established drinking water or other water-quality targets.

<sup>2</sup> Minnesota Department of Health (2014).

<sup>3</sup> Toccalino and others (2012).

<sup>4</sup> U.S. Environmental Protection Agency (2013).

established standards, one detected compound, *p*-cresol, was detected at site 452055093105401 (map number 62, fig. 2, table 1), a monitoring well in septic residential land use, at a concentration of 2.9  $\mu\text{g/L}$ , as compared with the MDH HRL of 3  $\mu\text{g/L}$ . Four of the six most frequently detected compounds—azithromycin, diphenhydramine, tributyl phosphate, and lincomycin—have no health-based water-quality standards or benchmarks.

## Environmental Implications

Samples from bedrock wells, most of which are drinking water wells that are deeper than glacial wells, had a higher percentage of CEC detections than samples from glacial wells. Samples from drinking water wells with detections had three or fewer distinct chemicals detected. One drinking water well completed in a bedrock aquifer had five detections, but

sulfamethoxazole and carbamazepine were each detected by two analytical methods (table 10, tables 1–2 through 1–5). The higher DO concentrations and lower specific conductance for the bedrock wells sampled indicate relatively shorter duration flow paths from the land surface to the wells than for glacial wells. Homes that rely on a domestic well as a drinking water source often have a septic system for wastewater disposal (DeSimone and others, 2009; Bremer and Harter, 2012). The frequency of CEC detections in these bedrock drinking water wells is an indicator that these wells are indeed vulnerable to anthropogenic contaminants, potentially from septic system wastewater.

The antibiotic sulfamethoxazole, the most frequently detected CEC, was detected in 11.4 percent of the samples. Most of the detections (11 of 14, or 79 percent) of sulfamethoxazole were in samples from domestic wells or monitoring wells located in areas where septic systems are prevalent. Sulfamethoxazole was detected in 30 percent (8 of 27) of all domestic wells sampled; these wells likely are in proximity to a septic system or leaking sewer lines (Bremer and Harter, 2012). Because of its physical properties, sulfamethoxazole is mobile and not readily degraded in the subsurface environment (National Center for Biotechnology Information, 2013). Although the detections of sulfamethoxazole in samples for this study were several orders of magnitude less than the HBV of 100 µg/L (table 11), the detections nonetheless indicate that this antibiotic is present in domestic wastewater, is mobile in groundwater, and that shallow aquifers in Minnesota are vulnerable to anthropogenic contamination. The antibiotic azithromycin was detected in samples from 5 wells, 3 of which were domestic wells. Like sulfamethoxazole, azithromycin is an antibiotic used by people, and it is expected to be mobile and degrade slowly in the environment (Pfizer, 2012).

Conversely, bisphenol A, the chemical detected second most frequently, was detected primarily in wells in proximity to closed landfills. The only chemical detected at a concentration nearing a health-based water quality standard (*p*-cresol), was detected at a monitoring well located in septic residential land use. Diphenhydramine, an antihistamine, was detected in samples from 5 wells, 4 of which were monitoring wells not in areas with substantial septic system presence. One of the wells with detectable diphenhydramine was a domestic well completed in a bedrock aquifer.

The greater frequency of detection in wells located in developed land-use settings indicate that domestic, commercial, or industrial wastewater or infiltrating stormwater runoff are likely sources of some of the CECs. Other potential sources of CECs to shallow, vulnerable groundwater include transport and infiltration of CECs present on the land surface from atmospheric deposition, leaking municipal sewer lines, or other unknown sources. The CECs enter wastewater streams from domestic, commercial, industrial, and agricultural sources, and chemicals not removed through a treatment system are discharged to the environment (Ternes and others, 1999).

## Summary

A study of contaminants of emerging concern (CECs) in ambient groundwater in urbanized areas of Minnesota was completed by the U.S. Geological Survey in cooperation with the Minnesota Pollution Control Agency. For this study, water samples were collected from November 2009 through June 2012 from 118 wells located in different land-use settings. The sampled wells primarily were screened in vulnerable sand and gravel aquifers (surficial and buried glacial aquifers) or vulnerable bedrock aquifers such as the Prairie du Chien-Jordan aquifer. Sampled well depths ranged from 9 to 285 feet below land surface. Water samples were collected by Minnesota Pollution Control Agency staff. The water samples were analyzed for steroidal hormones, human-use pharmaceutical compounds, human- and animal-use antibiotics, and a broad suite of organic compounds associated with wastewater at U.S. Geological Survey laboratories. Reported detections were censored and not counted as detections in the data analyses if the chemical was detected in a laboratory or field blank at a similar concentration.

During this study, 38 out of 127 CECs analyzed were detected among all water samples collected. Three of the detected CECs, however, were analyzed using two different analytical methods (2080 and 1433 or LCAB), so 35 distinct chemicals were detected. The number of detections of CECs in individual water samples ranged from 0 to 10. The three wells in proximity to landfills had the most CEC detections. One or more CECs were detected in a total of 43 samples (35 percent); no CECs were detected in 80 samples.

CECs detected in more than one sample were azithromycin, carbamazepine, diphenhydramine, lincomycin, sulfamethazine, sulfadiazine, and sulfamethoxazole (human and animal-use pharmaceuticals); camphor (flavor, fragrance); 4-cumylphenol and 4-*tert*-octylphenol (nonionic detergent metabolites); bromacil (herbicide active ingredient); carbazole and metolachlor (pesticide active ingredients); tris(2-chloroethyl) phosphate (plasticizer and flame retardant); cotinine (nicotine metabolite); bisphenol A (plastic and resin component); fluoranthene and pyrene (asphalt components, combustion products); 1,4-dichlorobenzene (moth repellent); tributyl phosphate (corrosion inhibitor); and isopropylbenzene (fuel and paint thinner component). The hormone or hormone metabolites *cis*-androsterone and mestranol were each detected in one sample. Also detected in one sample each were a fecal indicator and a fecal stench component, 3-*beta*-coprostanol and 3-methyl-1*H*-indole, respectively. Chemicals detected in one sample and found in products used for ingestion or topical use were acetaminophen, caffeine, *N,N*-Diethyl-*meta*-toluamide (DEET), hexahydrohexamethylcyclopentabenzopyran (HHCB), and triethyl citrate. The chemicals indole, 5-methyl-1*H*-benzotriazole, *p*-cresol, tetrachloroethene, and triphenyl phosphate, which are components of different types of industrial products, also were each detected in one sample.

The chemical DEET was detected at the highest concentration of any CEC, at 7.9 micrograms per liter. The antibiotic

sulfamethoxazole was the most frequently detected CEC, detected in a total of 14 of 123 samples (11.4 percent) by one or both analytical methods that include sulfamethoxazole as an analyte. Most (11 of 14, or 79 percent) of the detections of the antibiotic sulfamethoxazole were in samples from domestic wells or monitoring wells located in areas where septic systems or leaking municipal sewer lines are prevalent. Conversely, bisphenol A, the chemical detected second most frequently, and DEET, detected at the highest concentration, were detected primarily in wells in proximity to closed landfills.

Of the 127 chemicals included for analysis in this study, 28 have established enforceable or non-enforceable health-based water-quality standards or benchmarks. Fourteen of the 35 detected compounds have established health-based water-quality standards, whereas 21 detected compounds have no established standard or benchmark. All detections in this study were less than health-based water-quality standards. Although most detections were well below established standards, one detected compound, *p*-cresol, was detected at a monitoring well located in septic residential land use at a concentration of 2.9 micrograms per liter ( $\mu\text{g/L}$ ), as compared with the Health Risk Limit of 3  $\mu\text{g/L}$ . Four of the six most frequently detected compounds—azithromycin, diphenhydramine, tributyl phosphate, and lincomycin—have no health-based water-quality standards or benchmarks.

Samples from bedrock wells, most of which are drinking water wells that are deeper than glacial wells, had a higher percentage of CEC detections than samples from glacial wells. Samples from all but one drinking water well with detections had only one or two chemicals detected; one drinking water well completed in a bedrock aquifer had detections of five chemicals. The higher dissolved oxygen concentrations and lower specific conductance for the bedrock wells sampled indicate relatively shorter duration flow paths from the land surface to these wells than for glacial wells. Homes that rely on a domestic well as a drinking water source often have a septic system for wastewater disposal. The frequency of CEC detections in these bedrock drinking water wells is an indicator that these wells are indeed vulnerable to anthropogenic contaminants, potentially from septic system wastewater.

The greater frequency of detection in wells located in developed land-use settings indicate that domestic, commercial, or industrial wastewater or infiltrating stormwater runoff are likely sources of some of the CECs. Other potential sources of CECs to shallow, vulnerable groundwater include transport and infiltration of CECs present on the land surface from atmospheric deposition, leaking municipal sewer lines, or other unknown sources. The CECs enter wastewater streams from domestic, commercial, industrial, and agricultural sources, and chemicals not removed through a treatment system are discharged to the environment.

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# Appendix

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## Appendix 1.

Appendix tables 1–1 through 1–5 that are presented in this section of the report are provided as separate worksheets in a single Microsoft® Excel spreadsheet available at [http://pubs.usgs.gov/sir/2014/5096/downloads/appendix\\_tables](http://pubs.usgs.gov/sir/2014/5096/downloads/appendix_tables).

**Table 1–1.** Physical properties in unfiltered groundwater, Minnesota, 2009–12.

**Table 1–2.** Concentrations of chemicals in water samples analyzed at the U.S. Geological Survey National Water Quality Laboratory using laboratory schedule 1433 for analysis of wastewater indicator chemicals, 2009–12.

**Table 1–3.** Concentrations of chemicals in water samples analyzed at the U.S. Geological Survey National Water Quality Laboratory using laboratory schedule 2080 for analysis of pharmaceutical chemicals, 2009–12.

**Table 1–4.** Concentrations of chemicals in water samples analyzed at the U.S. Geological Survey National Water Quality Laboratory using laboratory schedule 2434 for analysis of hormones and related chemicals, 2010–12.

**Table 1–5.** Concentrations of chemicals in water samples analyzed at the U.S. Geological Survey Organic Geochemistry Research Laboratory using laboratory schedule LCAB for analysis of antibiotic and other chemicals, 2009–12.

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