

Prepared in cooperation with the Triangle Area Water Supply Monitoring Project Steering Committee

Occurrence of Organic Wastewater Compounds in Selected Surface-Water Supplies, Triangle Area of North Carolina, 2002–2005

Scientific Investigations Report 2007–5054



Cover. Cane Creek Reservoir dam, Orange County, North Carolina *(photograph by M.J. Giorgino, U.S. Geological Survey).*

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By M.J. Giorgino, R.B. Rasmussen, and C.A. Pfeifle

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**U.S. Department of the Interior
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Conversion Factors

Inch/Pound to SI

Multiply	By	To obtain
	Length	
foot (ft)	0.3048	meter (m)
mile (mi)	1.609	kilometer (km)
	Area	
square mile (mi ²)	2.590	square kilometer (km ²)
	Volume	
quart (qt)	0.9464	liter (L)
	Flow rate	
million gallons per day (Mgal/d)	0.04381	cubic meter per second (m ³ /s)
	Mass	
ounce, avoirdupois (oz)	28.35	gram (g)

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

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

Acronyms and abbreviations used in this report:

AHTN	acetyl-hexamethyl tetrahydronaphthalene
DEET	N,N-diethyl-meta-toluamide
EDC	endocrin-disrupting compound
GC/MS	gas chromatography/mass spectrometry
HHCB	hexahydro-hexamethyl cyclopentabenzopyran
HIA	household, industry, and agriculture
HPLC/ESI-MS	high-performance liquid chromatography/electrospray ionization mass spectrometry
LHA	U.S. Environmental Protection Agency Lifetime Health Advisory
mL	milliliter
NWQL	U.S. Geological Survey National Water Quality Laboratory
OWC	organic wastewater compound
PAH	polycyclic aromatic hydrocarbon
QA/QC	quality assurance and quality control
RLR	reporting level
SPE	solid-phase extraction
TAWSP	Triangle Area Water Supply Monitoring Project
WWTP	wastewater-treatment plant
USGS	U.S. Geological Survey

Occurrence of Organic Wastewater Compounds in Selected Surface-Water Supplies, Triangle Area of North Carolina, 2002–2005

By M.J. Giorgino, R.B. Rasmussen, and C.A. Pfeifle

Abstract

Selected organic wastewater compounds, such as household, industrial, and agricultural-use compounds, sterols, pharmaceuticals, and antibiotics, were measured at eight sites classified as drinking-water supplies in the Triangle Area of North Carolina. From October 2002 through July 2005, seven of the sites were sampled twice, and one site was sampled 28 times, for a total of 42 sets of environmental samples.

Samples were analyzed for as many as 126 compounds using three laboratory analytical methods. These methods were developed by the U.S. Geological Survey to detect low levels (generally less than or equal to 1.0 microgram per liter) of the target compounds in filtered water. Because analyses were conducted on filtered samples, the results presented in this report may not reflect the total concentration of organic wastewater compounds in the waters that were sampled. Various quality-control samples were used to quality assure the results in terms of method performance and possible laboratory or field contamination.

Of the 108 organic wastewater compounds that met method performance criteria, 24 were detected in at least one sample during the study. These 24 compounds included 3 pharmaceutical compounds, 6 fire retardants and plasticizers, 3 antibiotics, 3 pesticides, 6 fragrances and flavorants, 1 disinfectant, and 2 miscellaneous-use compounds, all of which likely originated from a variety of domestic, industrial, and agricultural sources. The 10 most frequently detected compounds included acetyl-hexamethyl tetrahydronaphthalene and hexahydro-hexamethyl cyclopentabenzopyran (synthetic musks that are widely used in personal-care products and are known endocrine disruptors); tri(2-chloroethyl) phosphate, tri(dichloroisopropyl) phosphate, and tributyl phosphate (fire retardants); metolachlor (herbicide); caffeine (nonprescription stimulant); cotinine (metabolite of nicotine); acetaminophen (nonprescription analgesic); and sulfamethoxazole (prescription antibiotic).

The occurrence and distribution of organic wastewater compounds varied considerably among sampling sites, but at least one compound was detected at every location. The most organic wastewater compounds (19) were detected at the Neuse River above U.S. 70 at Smithfield, where two-thirds of the total number of samples were collected. The fewest organic wastewater compounds (1) were detected at the Eno River at Hillsborough. The detection of multiple organic wastewater compounds was common, with a median of 3.5 and as many as 12 compounds observed in individual samples. Some compounds, including acetaminophen, cotinine, tri(2-chloroethyl) phosphate, and metolachlor, were detected at numerous sites and in numerous samples, indicating that they are widely distributed in the environment. Other organic wastewater compounds, including acetyl-hexamethyl tetrahydronaphthalene and hexahydro-hexamethyl cyclopentabenzopyran, were detected in numerous samples but at only one location, indicating that sources of these compounds are more site specific. Results indicate that municipal wastewater may be a source of antibiotics and synthetic musks; however, the three sites in this study that are located downstream from wastewater discharges also receive runoff from agricultural, urban, and rural residential lands. Source identification was not an objective of this study.

Concentrations of individual compounds generally were less than 0.5 microgram per liter. No concentrations exceeded Federal drinking-water standards or health advisories, nor water-quality criteria established by the State of North Carolina; however, such criteria are available for only a few of the compounds that were studied.

Compared with other surface waters that have been sampled across the United States, the Triangle Area water-supply sites had fewer detections of organic wastewater compounds; however, differences in study design and analytical methods used among studies must be considered when making comparisons. In general, concentrations of organic wastewater compounds detected in the Triangle Area were within ranges of concentrations reported for other areas.

Maximum concentrations of pharmaceuticals, antibiotics, pesticides, fragrances, and disinfectants observed in the Triangle Area—even at sites downstream from major municipal wastewater discharges—generally were lower than those reported for other surface-water sites in the United States. In contrast, maximum concentrations of fire retardants that were detected in this study were consistent with concentrations observed elsewhere.

Introduction

Newly recognized classes of organic compounds that often are associated with wastewater recently have been documented in the world's waterways. For example, Kolpin and others (2002) reported that at least one organic wastewater compound was detected in 80 percent of 139 streams sampled across the United States. These organic wastewater compounds (OWCs) include chemicals widely used by households, industry, and agriculture (HIAs), sterols, pharmaceutical compounds, and antibiotics. OWCs are characterized by high usage rates, potential health effects, and a potential for continuous release through human activity (Daughton and Ternes, 1999). While many of these compounds are indicators of contamination from human sources, a few also occur naturally. Some, such as those that disrupt the endocrine systems of fish and other aquatic organisms, may have important implications for human and environmental health.

Relatively little is known about the occurrence of OWCs in the environment because they are not measured routinely in most monitoring programs and because they may be present in very low concentrations. Even less is known about the potential effects on humans and aquatic organisms that may be exposed to individual compounds or to mixtures of these chemicals (Sumpter and Johnson, 2005). OWCs have been shown to enter the aquatic environment through various pathways, such as stormwater runoff from urban, industrial, residential, and agricultural lands; wastewater effluent; and ground-water seepage. Wastewater and drinking-water treatment systems may not completely remove these compounds (Richardson and Bowron, 1985; Stumpf and others, 1996; Ternes, 1998; Stackelberg and others, 2004). In addition, some compounds may be transformed or metabolized into potentially harmful degradation products (Boxall and others, 2004).

The U.S. Geological Survey (USGS) recently developed laboratory methods to analyze for broad suites of these compounds, including HIAs, sterols, pharmaceutical compounds, and antibiotics, at very low concentrations—generally at or below 1 microgram per liter ($\mu\text{g/L}$) (equivalent to 1 part per billion)—in water. These methods are being used to investigate the occurrence of OWCs at various locations in the

United States and in various media, including surface water, ground water, drinking water, and wastewater. Additional methods are under development for analyzing OWCs in sediment, biosolids, and biological tissue.

The USGS, through its National Water-Quality Assessment Program and in cooperation with local agencies, has conducted two studies of the occurrence of OWCs at eight sites classified for drinking-water supply in the Triangle Area of North Carolina. Because results were obtained from separate studies, numbers of samples that were collected varied among sites. Differences in the number of OWCs detected among sites may be due to differences in the number of samples collected, as well as differences in site characteristics.

Previous Investigations

Previous investigations have documented the widespread presence of trace amounts of OWCs in water resources across the United States (summarized by Focazio and others, 2004). Reconnaissance studies like those in the Triangle Area have focused on collecting baseline occurrence data within a defined geographic area and have been conducted at local, state, and national scales. Selected reconnaissance investigations with strong surface-water components are described below. Other types of investigations related to OWCs that currently are being conducted include: source studies, which seek to identify and characterize the chemical signature of specific sources; transport, fate, and treatment studies, which examine the movement and transformation of compounds through the environment or treatment systems; and ecological-effects studies, which characterize effects of exposure on organisms.

Several reconnaissance investigations have reported concentrations of OWCs in streams, rivers, ground water, wastewater-treatment plant (WWTP) effluents, and potable drinking water in the United States. Differences in objectives, sampling approach, targeted compounds, analytical techniques, and reporting levels make it difficult to extrapolate results among studies. For example, HIAs were variously analyzed in filtered and unfiltered water samples, depending on the analytical technique that was selected. Some studies did not include antibiotics or pharmaceutical drugs except for caffeine, cotinine, and a few others. Nevertheless, reconnaissance studies continue to be a key source of information regarding the occurrence of OWCs.

In a landmark national-reconnaissance study, Kolpin and others (2002) reported the occurrence of OWCs in a network of 139 streams across 30 states during 1999–2000. Site selection was biased intentionally toward streams downstream from intense urbanization and/or livestock production. At least one OWC was detected in 80 percent of the streams sampled. A total of 95 OWCs were analyzed and 82 OWCs were detected,

including HIAs, pharmaceuticals, and antibiotics. HIAs were analyzed in unfiltered water samples.

Subsequent reconnaissance studies often focused on metropolitan areas where wastewater inputs are likely. Glassmeyer and others (2005) investigated OWCs in WWTP effluents and receiving streams for 10 municipalities across the United States. The network included 9 upstream sites, 11 WWTP effluents, and 20 downstream sites. Sampling was conducted primarily during low- to normal-flow conditions. HIAs were analyzed in unfiltered water samples, and sampling included antibiotics and pharmaceuticals. Of the 110 compounds that were analyzed, 78 were detected. The number of OWCs detected in individual samples ranged from 0 at a reference location to 50 in an effluent sample. The maximum number of compounds detected in a surface-water sample was 47 (Glassmeyer and others, 2005). The investigators concluded that wastewater discharges affected both the number and total concentration of OWCs that were detected in receiving waters and that the number and total concentration of OWCs decreased with distance downstream from the WWTPs.

Sando and others (2005) studied OWCs in the Big Sioux River basin of South Dakota. Samples of three WWTP effluents and nine surface-water sites located upstream or downstream from each wastewater discharge were collected during low-flow and runoff conditions. Sampling included antibiotics, pharmaceuticals, and HIAs; the latter were analyzed in unfiltered samples. Of the 125 OWCs that were analyzed, 45 had acceptable method performance and were detected at reportable concentrations. Of the 45 detected OWCs, 39 were detected at surface-water sites and 42 were detected in effluent samples. The influence of wastewater discharges on OWCs in receiving waters varied depending on the size of the discharge relative to river flow (that is, the effluent contribution to total streamflow). One WWTP increased the number and total concentration of OWCs at a river site immediately downstream during both low-flow and runoff conditions and was an apparent source of antibiotics. The remaining two WWTPs probably influenced the occurrence of OWCs in the Big Sioux River downstream but did not substantially contribute to total OWC concentrations, especially during runoff conditions. Nonpoint agricultural sources also were indicated as primary contributors of OWCs, with sterols likely originating from livestock and herbicides likely originating from cropland.

Kolpin and others (2004) evaluated OWCs at stream sites upstream and downstream from 10 urban areas in Iowa during high-, normal-, and low-flow conditions. Four additional urban areas were sampled only during low-flow conditions. HIAs were analyzed in unfiltered water samples, and sampling included antibiotics and pharmaceuticals. Of the 105 OWCs that were analyzed, 62 were detected. Pesticides, sterols, and nonprescription drugs were the most frequently detected compounds during all flow conditions. The urban contribution

of OWCs to streams became less pronounced as streamflow increased. During low-flow conditions, total concentrations of antibiotics, prescription drugs, fire retardants, fragrances, plasticizers, detergents, pesticides, and sterols were significantly higher downstream than upstream from urban areas. During normal flow, only total concentrations of nonprescription drugs were statistically higher downstream. During high flow, no significant differences in OWCs were observed between sites upstream and downstream from urban centers.

Organic wastewater compounds were analyzed at 26 sites located upstream, within, and downstream from Atlanta, Georgia, as part of five different studies summarized by Frick and Zaugg (2003). Sampling sites included WWTP effluents, Chattahoochee River sites, tributary sites, and raw and finished municipal drinking water. Unfiltered water samples were analyzed for HIAs, but sampling did not include antibiotics or pharmaceutical drugs. The number of OWCs that were analyzed varied among samples but generally ranged from 42 to 51. Of these, 39 different OWCs were detected in surface-water (river and tributary) samples. The number and concentrations of OWCs observed at various types of sites decreased from effluent samples, to wet-weather stream samples, to baseflow stream samples, to drinking-water samples. Patterns of detection indicated that nonpoint-source runoff was the primary source of polycyclic aromatic hydrocarbons and insecticides. In contrast, detections of plasticizers, fire retardants, and detergent metabolites indicated the presence of wastewater.

Sprague and Battaglin (2005) investigated the occurrence of HIAs at 16 stream sites and 87 wells in Colorado. Stream sites included 15 in urban areas and 1 in a minimally developed watershed. HIAs were analyzed in filtered samples using the same analytical method as in the Triangle Area of North Carolina. Antibiotics and pharmaceuticals were not sampled. In the urban streams, 57 of the 62 HIAs were detected at least once. In the minimally developed stream, fewer (11 of 62) HIAs were detected and at concentrations lower than those observed in the urban streams.

Lee and others (2004) reported OWC concentrations at 65 sites in Minnesota, including samples of surface water (32 sites), ground water, drinking water, landfill leachate, and seepage from livestock lagoons. HIAs were analyzed in filtered water, using the same analytical method as in the Triangle Area, and in unfiltered water. Results from the two methods showed reasonable agreement for OWC detection, and the investigators concluded that data from both methods could be used to describe OWC presence and distribution. Concentrations of most OWCs tended to be greater in filtered samples than in unfiltered samples, but differences generally were within the laboratory analytical error for selected OWCs. The Minnesota study included sampling for pharmaceutical drugs and antibiotics. Of the 91 OWCs that were analyzed, 56 were detected among all surface-water samples, averaging

6 OWCs per sample. The total number of OWCs detected ranged from 0 at a reference site to 28 at a site downstream from a WWTP discharge. Lee and others (2004) concluded that WWTP effluent was a major pathway of OWCs to surface waters. High numbers of OWCs also were observed in landfill leachate and in ground water underlying a waste dump and animal feedlots. Three lakes were sampled in this study, including a reference lake (0 detected OWCs), a lake not directly influenced by WWTP discharge (2 detected OWCs), and Lake Superior near a major municipal WWTP discharge (13 detected OWCs).

The objective of the Triangle Area reconnaissance was to document the occurrence of OWCs at eight sites classified as drinking-water supplies. The quality of these waters is of great interest to the public and to water-resource managers in local and State governments. Six of the eight sampling sites were located in reservoirs, which are hydrologically distinct from free-flowing streams. In contrast, most previous investigations have focused on streams and rivers, and comparative data for OWCs in reservoirs currently are limited. Because sites were limited to water-supply sources, results from this study should not be considered representative of all types of water bodies nor of the diverse environmental settings that are present in North Carolina.

Purpose and Scope

This report summarizes the occurrence of OWCs in filtered surface water at eight locations in the Triangle Area of North Carolina during October 2002 through July 2005. All sampling sites are classified as drinking-water supplies. At seven sites, two sets of samples were analyzed for HIAs, sterols, pharmaceutical compounds, and an array of antibiotics. At the remaining site, 28 samples were collected for HIAs, sterols, and a limited number of pharmaceuticals, but antibiotics were not sampled. Results were evaluated to address the following questions:

- What types of OWCs were detected?
- How were the detected OWCs distributed?
- How did concentrations compare with water-quality benchmarks?
- How did results compare with those from other areas of the country?

Study Area and Methods

The study area encompasses a five-county (Chatham, Durham, Johnston, Orange, and Wake) region in the Piedmont Physiographic Province of North Carolina, and includes portions of the Neuse and Cape Fear River basins (fig. 1). The area is referred to as “the Triangle,” because the three large municipalities of Raleigh, Durham, and Chapel Hill are located in relative proximity. Additional towns in the study area include Hillsborough, Carrboro, Apex, Cary, Morrisville, Wake Forest, Garner, and Smithfield. Multiple municipal and county agencies manage public drinking-water and wastewater utilities within the region. Population in the five-county area grew 16 percent from 2000 to 2005, and is projected to increase by an additional 70 percent from 2005 to 2030 (North Carolina State Demographics, 2006). This ongoing, rapid growth is placing increasing demands on these agencies and the water resources on which they rely.



Figure 1. Location of the study area, sampling sites, relevant municipal wastewater-treatment plants, and land cover in the Piedmont Physiographic Province of North Carolina.

Sampling Sites

As part of two different water-quality programs, the USGS collected 42 sets of samples for OWCs from eight sites in the Triangle Area from October 2002 to July 2005 (fig. 1; table 1). Two sites were riverine, six were reservoir sites, and all were located in waters classified as drinking-water supplies. Drainage areas for the sites ranged from 30 to 1,200 square miles. Drainage area was not computed for site 7 because it is located in a reservoir cove rather than along a defined stream channel. Water quality at this location usually is similar to that at site 6, which is located up-reservoir. Although the study area includes several urban centers, only three of the eight sampling locations were located downstream from major (discharging more than 100,000 gallons per day) municipal wastewater discharges. Land-cover patterns also differed among sites. Sites 1, 2, 3, 4, and 5 were located in predominantly rural watersheds dominated by forested and agricultural land cover; sites 6, 7, and 8 drained larger proportions of developed land (fig. 1; table 1).

Sites 1 through 7 (fig. 1; table 1) were sampled as part of the Triangle Area Water Supply Monitoring Project, a long-term cooperative effort between the USGS and local government partners (table 2). Since 1988, data have been collected to support the evaluation of trends in nutrients, sediment, major ions, and trace elements, and to document

the occurrence of synthetic organic compounds at project sites (project information is available online at <http://nc.water.usgs.gov/triangle/>). To supplement existing water-quality information, each of these sites was sampled for OWCs once in April 2004 and once in August 2004 during normal to low-flow hydrologic conditions. Two samples from these sites were ruined during laboratory analyses for HIAs and sterols; therefore, replacement samples for these OWCs were collected at site 2 in October 2004 and at site 6 in April 2005. Because only two full sets of samples were analyzed for each location, the results provide a limited reconnaissance of conditions at sites 1 through 7 and should be considered preliminary in nature.

The Neuse River above U.S. Highway 70 at Smithfield (site 8) was sampled as part of a USGS National Water-Quality Assessment Program study on the quality of water-supply sources across the United States. This site was sampled 28 times from October 2002 through July 2005 during a range of hydrologic conditions. At site 8, samples were collected for sterols and HIAs (including caffeine); however, no other pharmaceuticals or antibiotics were sampled at this location (table 2). Although this sampling approach differed from that used at sites 1 through 7, results from site 8 were useful for expanding the geographic scope of the OWC evaluation and for providing additional quality-control samples that helped to assess data bias and precision.

Table 1. Locations and characteristics of water-supply sites sampled for organic wastewater compounds between October 2002 and July 2005 in the Triangle Area of North Carolina.

[USGS, U.S. Geological Survey; WWTPs, major municipal wastewater-treatment plants; n/a, not applicable; nd, not determined but presumed to be similar to site 6]

Site number (fig. 1)	USGS station number	Site location in North Carolina	Drainage area (square miles)	Number of upstream WWTPs	Watershed land cover (percent) ^a			
					Agricultural	Developed	Forested/ herbaceous	Water/wetlands/ other
1	02085000	Eno River at Hillsborough	66.0	0	26	12	60	2
2	0208524845	Little River Reservoir at dam near Bahama	97.7	0	28	7	63	2
3	02086490	Lake Michie at dam near Bahama	167	0	28	6	64	2
4	0209684980	Cane Creek Reservoir at dam near White Cross	31.4	0	26	5	66	3
5	0209749990	University Lake at dam near Chapel Hill	30	0	15	7	76	2
6	0209799150	B.E. Jordan Lake above U.S. 64 near Wilsonville	285	3	7	27	56	9
7	0209801100	B.E. Jordan Lake at Bells Landing near Griffins Crossroads	n/a	3	nd	nd	nd	nd
8	0208755215	Neuse River above U.S. 70 at Smithfield	1,200	6	17	22	56	5

^aBased on the 2001 National Land Cover Database (Homer and others, 2004).

Table 2. Triangle Area sampling sites, monitoring programs and partners, duration, and classes of organic wastewater compounds, North Carolina.

[OWC, organic wastewater compound; HIAs, household, industrial, and agricultural-use compounds; USGS, U.S. Geological Survey; ns, not sampled]

Site (fig. 1)	Program and partners	Duration of OWC sampling	Number of samples collected for each class of OWCs			
			HIAs	Sterols	Pharma- ceuticals	Anti- biotics
1–7	Triangle Area Water Supply Monitoring Project ^a	April 2004 to April 2005	2	2	2	2
8	USGS National Water-Quality Assessment Program	October 2002 to July 2005	28	28	ns	ns

^aParticipants in the Triangle Area Water Supply Monitoring Project:

Town of Apex	Chatham County
Town of Cary	Orange County
City of Durham	Wake County
Town of Hillsborough	USGS Cooperative Water Program
Town of Morrisville	
Orange Water and Sewer Authority (serving Chapel Hill and Carrboro)	

Sample Collection and Processing

Water samples were collected using established protocols and procedures to avoid sample contamination and obtain representative samples (U.S. Geological Survey, variously dated). In addition, sampling teams adhered to special requirements for the collection of wastewater, pharmaceutical, and antibiotic compounds, which included avoiding contact with insect repellents, antibacterial cleansers, caffeine, tobacco, and targeted pharmaceuticals (U.S. Geological Survey, variously dated). Source-solution blanks, field blanks, replicate samples, and matrix spike samples were collected for quality assurance.

Depending on site conditions, samples were collected from boats, water-intake platforms, or by wading. All samples were collected with inert materials, such as Teflon or stainless steel. Collectors used a Teflon container and a weighted bottle sampler to obtain three to five depth-integrated subsamples at sites 2–8. At site 1, depth-integrated subsamples were collected from 10 to 15 equally spaced intervals across the channel. At every site, subsamples were composited in a Teflon container and chilled prior to processing.

Each sample was filtered through a pre-baked 0.7-micron glass fiber filter within 2 hours of collection, either in the field or in the USGS North Carolina Water Science Center laboratory in Raleigh. Approximately 10 to 20 milliliters (mL) of reagent-grade blank water and 100 mL of filtrate were used to prepare and flush the filtration system before sample collection. The samples then were collected into pre-cleaned amber glass bottles and vials, chilled, and shipped for analysis.

Because all analyses were conducted on filtered samples, the results presented in this report may not reflect the total concentration of OWCs in the waters that were sampled. Some OWCs, especially those that are hydrophobic (have low solubility in water), tend to associate with particulate matter in surface waters, and this fraction was removed during filtra-

tion. Therefore, the results presented in this study represent conservative estimates of the concentrations of these OWCs at the sampling locations.

Laboratory Analysis

Three laboratory analytical methods were used in this study to test for as many as 126 compounds in each sample, including 57 HIAs, 4 sterols, 18 pharmaceutical compounds, and 47 antibiotics (table 3). Additional information for the analytes, including their common uses and sources, is provided in Appendix 1. Specific compounds that were analyzed and their reporting limits varied among samples because the methods continued to be refined during the study period. Laboratory set blanks, spikes, and surrogates were routinely run for all analytical methods, and these data were used to evaluate bias and variability that may occur in the environmental data as a result of laboratory activities.

Five OWCs, including caffeine, cotinine, and three antibiotics (erythromycin, sulfamethoxazole, and trimethoprim), were analyzed by more than one method. For these compounds, quality-assurance and quality-control (QA/QC) results were evaluated to determine the analytical method with the best performance. Only results for the selected method are included in the discussion related to the occurrence of OWCs in this study.

Analytical method 1 was a USGS-approved production method that analyzed for 63 organic compounds typically found in domestic and industrial wastewater. OWCs analyzed by this method spanned diverse classes of chemicals, including 57 HIAs (such as fire retardants, non-ionic surfactants, plasticizers, solvents, disinfectants, polycyclic aromatic hydrocarbons (PAHs), and high-use domestic pesticides), 4 sterols, and 2 pharmaceutical compounds (caffeine and cotinine). These

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Table 3. Summary of results for dissolved organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005.

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration below reporting level. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Number of detections	Total number of samples	Frequency of detection (percent)
Household, industrial, and agricultural use compounds							
1	1,4-dichlorobenzene	0.5	—	—	0	42	0
1	1-methylnaphthalene	0.5	—	—	0	42	0
1	2,6-dimethylnaphthalene	0.5	—	—	0	42	0
1	2-methylnaphthalene	0.5	—	—	0	42	0
1	3-methyl-1H-indole (skatol)	1.0	E0.064	E0.064	1	42	2.4
1 ^a	3-tert-butyl-4-hydroxyanisole (BHA)	5.0	failed method performance criteria				
1	4-cumylphenol	1.0	—	—	0	42	0
1	4-normal-octylphenol	1.0	—	—	0	42	0
1	4-tert-octylphenol	1.0	—	—	0	42	0
1 ^a	5-methyl-1H-benzotriazole	2.0	failed method performance criteria				
1	Acetophenone	0.5	—	—	0	42	0
1	Acetyl-hexamethyl tetrahydronaphthalene (AHTN)	0.5	E0.014	E0.084	19	42	45.2
1	Anthracene	0.5	—	—	0	42	0
1	Anthraquinone	0.5	E0.11	E0.11	1	42	2.4
1	Benzo[a]pyrene	0.5	—	—	0	42	0
1	Benzophenone	0.5	—	—	0	42	0
1 ^a	Bisphenol-A	1.0	failed method performance criteria				
1 ^a	Bromacil	0.5	failed method performance criteria				
1	Bromoform	0.5	—	—	0	42	0
1	Camphor	0.5	E0.006	E0.021	6	42	14.3
1 ^a	Carbaryl	1.0	failed method performance criteria				
1	Carbazole	0.5	—	—	0	42	0
1	Chlorpyrifos	0.5	—	—	0	42	0
1	Diazinon	0.5	E0.016	E0.02	2	42	4.8
1 ^a	Dichlorvos	1.0	failed method performance criteria				
1 ^a	<i>d</i> -Limonene	0.5	failed method performance criteria				
1	Fluoranthene	0.5	—	—	0	42	0
1	Hexahydro-hexamethyl cyclopentabenzopyran (HHCB)	0.5	E0.049	E0.23	26	42	61.9
1	Indole	0.5	E0.006	E0.006	1	42	2.4
1	Isoborneol	0.5	—	—	0	42	0
1	Isophorone	0.5	—	—	0	42	0
1 ^a	Isopropylbenzene (cumene)	0.5	failed method performance criteria				
1	Isoquinoline	0.5	—	—	0	42	0
1	Menthol	0.5	E0.023	E0.16	2	42	4.8
1	Metalaxyl	0.5	—	—	0	42	0
1	Methyl salicylate	0.5	—	—	0	42	0
1	Metolachlor	0.5	E0.012	E0.11	21	42	50
1	Naphthalene	0.5	—	—	0	42	0
1	N,N-diethyl- <i>meta</i> -toluamide (DEET)	0.5	—	—	0	42	0
1	Nonylphenol diethoxylate (NP2EO)	5.0	—	—	0	42	0
1	Octylphenol diethoxylate (OP2EO)	1.0	—	—	0	42	0
1 ^a	Octylphenol monoethoxylate (OP1EO)	1.0	failed method performance criteria				
1	<i>para</i> -Cresol	1.0	E0.39	E0.39	1	42	2.4
1 ^a	<i>para</i> -nonylphenol (NP)	5.0	failed method performance criteria				
1 ^a	Pentachlorophenol	2.0	failed method performance criteria				
1	Phenanthrene	0.5	—	—	0	42	0
1	Phenol	0.5	—	—	0	42	0

Table 3. Summary of results for dissolved organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration below reporting level. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Number of detections	Total number of samples	Frequency of detection (percent)
1	Prometon	0.5	E0.04	E0.094	2	42	4.8
1	Pyrene	0.5	—	—	0	42	0
1 ^a	Tetrachloroethylene	0.5	failed method performance criteria				
1	Tri(2-butoxyethyl) phosphate	0.5	E0.13	3.7	8	42	19.0
1	Tri(2-chloroethyl) phosphate	0.5	E0.033	0.7	26	42	61.9
1	Tri(dichloroisopropyl) phosphate	0.5	E0.031	E0.15	16	42	38.1
1	Tributyl phosphate	0.5	E0.016	E0.12	12	42	28.6
1	Triclosan	1.0	E0.066	E0.098	4	42	9.5
1	Triethyl citrate (ethyl citrate)	0.5	E0.020	E0.098	8	42	19.0
1	Triphenyl phosphate	0.5	E0.005	E0.052	8	42	19.0
Sterols							
1 ^a	3- <i>beta</i> -coprostanol	2.0	failed method performance criteria				
1 ^a	beta-sitosterol	2.0	failed method performance criteria				
1 ^a	<i>beta</i> -stigmastanol	2.0	failed method performance criteria				
1 ^a	Cholesterol	2.0	failed method performance criteria				
Pharmaceuticals							
2	1,7-dimethylxanthine	0.144	—	—	0	14	0
2	Acetaminophen	0.036	E0.010	0.037	4	14	28.6
1 ^b	Caffeine	0.5	E0.017	E0.17	20	42	47.6
2	Caffeine	0.016	—	—	0	14	0
2	Carbamazepine	0.011	—	—	0	14	0
2	Cimetidine	0.012	—	—	0	14	0
2	Codeine	0.015	—	—	0	14	0
1 ^a	Cotinine	1.0	failed method performance criteria				
2 ^b	Cotinine	0.014	E0.007	0.020	6	14	42.9
2	Dehydronifedipine	0.015	—	—	0	14	0
2	Diltiazem	0.016	—	—	0	14	0
2	Diphenhydramine	0.015	—	—	0	14	0
2	Fluoxetine	0.014	—	—	0	14	0
2	Gemfibrozil	0.013	—	—	0	14	0
2	Ibuprofen	0.042	—	—	0	14	0
2 ^a	Miconazole	0.018	failed method performance criteria				
2	Ranitidine	0.013	—	—	0	14	0
2	Salbutamol	0.023	—	—	0	14	0
2	Thiabendazole	0.011	—	—	0	14	0
2	Warfarin	0.012	—	—	0	14	0
Antibiotics (*degradation product)							
3	Amoxicillin	0.20, 0.01	—	—	0	14	0
3	Ampicillin	0.10, 0.01	—	—	0	14	0
3	Anhydro-chlorotetracycline*	0.10, 0.01	—	—	0	14	0
3	Anhydro-erythromycin*	0.05, 0.005	0.042	0.055	2	14	14.3
3	Anhydro-tetracycline *	0.20, 0.01	—	—	0	14	0
2 ^a	Azithromycin	0.004	failed method performance criteria				
3	Carbadox	0.005	—	—	0	14	0
3	Cefotaxime	0.10, 0.01	—	—	0	14	0
3	Chlorotetracycline	0.10, 0.01	—	—	0	14	0
3	Ciprofloxacin	0.05, 0.005	—	—	0	14	0
3	Clinafloxacin	0.05, 0.005	—	—	0	14	0
3	Cloxacillin	0.10, 0.01	—	—	0	14	0
3	Demeclocycline	0.10, 0.01	—	—	0	14	0

10 Occurrence of Organic Wastewater Compounds in Selected Surface-Water Supplies, Triangle Area of NC, 2002–2005

Table 3. Summary of results for dissolved organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration below reporting level. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Number of detections	Total number of samples	Frequency of detection (percent)
3	Doxycycline	0.10, 0.01	—	—	0	14	0
3	Epi-anhydro-chlorotetracycline*	0.10	—	—	0	14	0
3	Epi-anhydro-tetracycline*	0.10	—	—	0	14	0
3	Epi-chlorotetracycline*	0.10	—	—	0	14	0
3	Epi-oxytetracycline*	0.10	—	—	0	14	0
3	Epi-tetracycline*	0.10	—	—	0	14	0
2 ^a	Erythromycin	0.009	failed method performance criteria				
3 ^b	Erythromycin	0.10, 0.005	—	—	0	14	0
3	Flumequine	0.05, 0.005	—	—	0	14	0
3	Iso-chlorotetracycline*	0.10	—	—	0	14	0
3	Iso-epi-chlorotetracycline*	0.10	—	—	0	14	0
3	Lincomycin	0.05, 0.005	—	—	0	14	0
3	Lomefloxacin	0.05, 0.005	—	—	0	14	0
3	Minocycline	0.20, 0.01	—	—	0	14	0
3	Norfloxacin	0.05, 0.005	—	—	0	14	0
3	Ofloxacin	0.05, 0.005	—	—	0	14	0
3	Ormetoprim	0.05, 0.005	—	—	0	14	0
3	Oxacillin	0.10, 0.01	—	—	0	14	0
3	Oxolinic Acid	0.05, 0.005	—	—	0	14	0
3	Oxytetracycline	0.10, 0.01	—	—	0	14	0
3	Penicillin G	0.10, 0.01	—	—	0	14	0
3	PenicillinV	0.10, 0.01	—	—	0	14	0
3	Roxithromycin	0.10, 0.005	—	—	0	14	0
3	Sarafloxacin	0.05, 0.005	—	—	0	14	0
3	Sulfachloropyridazine	0.05, 0.005	—	—	0	14	0
3	Sulfadiazine	0.05, 0.005	—	—	0	14	0
3	Sulfadimethoxine	0.05, 0.005	—	—	0	14	0
3	Sulfamerazine	0.05, 0.005	—	—	0	14	0
3	Sulfamethazine	0.05, 0.005	—	—	0	14	0
2	Sulfamethoxazole	0.064	—	—	0	14	0
3 ^b	Sulfamethoxazole	0.05, 0.005	0.006	0.033	3	14	21.4
3	Sulfathiazole	0.05, 0.005	—	—	0	14	0
3	Tetracycline	0.10	—	—	0	14	0
2	Trimethoprim	0.013	—	—	0	14	0
3 ^b	Trimethoprim	0.05, 0.005	E0.003	E0.003	1	14	7.1
3	Tylosin	0.10, 0.005	—	—	0	14	0
3	Virginiamycin	0.10, 0.005	—	—	0	14	0

^a Results for laboratory reagent-spike samples and(or) field matrix-spike samples unacceptable; results not reported.

^b Method with the best quality-assurance/quality-control performance for this compound.

analyses were conducted at the USGS National Water Quality Laboratory (NWQL) in Denver, Colorado. Target compounds were extracted by using solid-phase extraction (SPE) and analyzed by capillary-column gas chromatography/mass spectrometry (GC/MS; Zaugg and others, 2002).

Analytical method 2 was a research method that measured 22 commonly used pharmaceutical compounds, such as over-the-counter painkillers, selected prescription drugs, caffeine, and a nicotine metabolite. These analyses were conducted at the USGS NWQL by using SPE combined with high-performance liquid chromatography/electrospray ionization mass spectrometry (HPLC/ESI-MS; Cahill and others, 2004).

Analytical method 3 was a research method that analyzed for as many as 46 human and veterinary antibiotics and selected degradation products, and was performed by the USGS Organic Geochemistry Research Laboratory in Lawrence, Kansas. Samples were extracted by using tandem SPE, and extracts were separated and measured by HPLC/ESI-MS (Hirsch and others, 1998; Meyer and others, 2000; Kolpin and others, 2002). Samples collected prior to April 20, 2004, were analyzed by using a single-quadrupole HPLC mass spectrometer. Samples collected on or after April 20, 2004, were analyzed by using a triple-quadrupole HPLC mass spectrometer that reduced reporting levels (RLs) by a factor of about 10 (table 3).

All three methods are considered to be “information-rich;” that is, they have qualifying information that enhances identification of the analytes (Childress and others, 1999). For these mass spectral methods, ion abundance ratios are used as confirmation that analytes are present. Results for information-rich methods are not restricted to censoring at analyte RLs (values set by the laboratory to avoid reporting false positives). When a compound is identified at a concentration less than the RL, its result is labeled with an “E” to indicate that the concentration is estimated. Estimated concentrations, especially values considerably below the RL, must be interpreted with caution. In these cases, one could have confidence that the compound was identified qualitatively in the sample but less confidence in the reported concentration. It should be noted that some results routinely are reported as estimated values, including all concentrations above or below the calibration curve, concentrations for analytes with average recoveries less than 60 percent, analytes routinely detected in laboratory blanks, and analytes with reference standards prepared from technical mixtures (Kolpin and others, 2002).

Quality Assurance

The bias and variability associated with data must be known in order to interpret environmental conditions adequately. Therefore, QA/QC samples were collected to assess data accuracy (lack of bias) and precision. QA/QC activities included both laboratory and field samples.

Laboratory QA/QC included laboratory blanks, spikes, and surrogate samples. Laboratory blanks consist of reagent-grade (ultra-pure), organic-free water that is processed and analyzed in the laboratory identically to environmental samples. Laboratory blanks are used to ensure that the laboratory equipment, environment, and analytical procedures do not introduce target compounds into the samples. Laboratory spikes, which consist of reagent-grade water fortified with known concentrations of target analytes, are analyzed in conjunction with sets of environmental samples. Laboratory spikes are used to monitor the general proficiency of the analytical method by comparing the concentrations of analytes that are “recovered” with the amounts that were added. At least one laboratory blank and one fortified spike were analyzed with each set of 10 to 15 environmental samples; therefore, they are sometimes referred to as set blanks and set spikes. Surrogates are chemicals that have properties similar to target compounds but do not interfere with the quantitation of the target compounds. Surrogates are added to every QC and environmental sample in known concentrations prior to extraction. The recovery percentage of each surrogate is used to monitor the accuracy of the extraction and analytical method for specific environmental matrices.

Field QA/QC samples included source-solution and field blanks, field replicate samples, and field (environmental-matrix) spikes. Source-solution blanks were prepared in the USGS North Carolina Water Science Center laboratory by pouring reagent-grade, organic-free water directly into a sample bottle. Source-solution blanks are used to identify contaminants in the blank water used to prepare other field QC samples. Field blanks indicate the potential for contamination during field sampling and processing. Field blanks were prepared onsite during scheduled sampling trips by processing blank water through the same equipment used to collect and process the field samples. Field replicate samples indicate data precision; that is, the variability of detections and concentrations that result from field and laboratory processing. Field spikes, which are prepared by adding known concentrations of target compounds to a field replicate sample, indicate the relative loss or gain of target analytes due to water-matrix characteristics, and(or) all subsequent field processing, shipping, storage, and laboratory analytical procedures.

Performance of the analytical methods for individual compounds was evaluated based on recovery percentages for laboratory spikes, laboratory surrogates, and field spikes. Method performance was deemed acceptable when the median recovery for spikes and surrogates was between 30 and 120 percent, and when the relative standard deviation of the laboratory spikes was less than 40 percent. Twenty analytes did not meet method-performance criteria, including 12 HIAs, all 4 sterols, 2 pharmaceutical compounds, and 2 antibiotics. Acceptable results for one of the pharmaceutical compounds (cotinine) and one of the antibiotics (erythromycin) were available from alternate analytical methods; the remaining 18 compounds that failed were excluded from further consideration (table 3).

For compounds with acceptable method performance, results for laboratory, source-solution, and field blanks were evaluated for indications of possible sample contamination. Forty-two OWCs were detected at least once among all of the laboratory blanks that were analyzed. Most of these were detected at concentrations far below reporting levels; hence, they were qualified as estimated values. In order to prevent possible laboratory contamination from biasing the results, all environmental-sample results were compared to their corresponding laboratory set blanks. If an OWC concentration in an environmental sample was less than 10 times the concentration in its corresponding set blank, the sample result was reported as a nondetection; that is, it was re-censored to the RL. In addition, six OWCs were detected in at least 30 percent of the laboratory blanks at concentrations similar to those reported for environmental samples, which indicates the possibility of chronic laboratory contamination. These OWCs included 1,4-dichlorobenzene, acetophenone, nonylphenol diethoxylate, octylphenol diethoxylate, phenanthrene, and phenol. Estimated detections of these constituents in environmental samples were reported as nondetections. Estimated detections of fluoranthene, isophorone, and methyl salicylate also were re-censored to their RLs because these compounds were detected much more frequently or at higher concentrations in laboratory blanks than in environmental samples.

In this study, benzophenone and N,N-diethyl-metoluamide (DEET) were detected in multiple field blanks at concentrations similar to those reported for environmental samples. All detections of benzophenone and DEET were estimated concentrations below their respective RLs. To prevent possible field contamination from biasing the data, all environmental-sample concentrations for these compounds were re-censored to the RLs.

Occurrence of Organic Wastewater Compounds

During this study, 108 analytes met method-performance criteria. Twenty-four OWCs, including 18 HIAs, 3 pharmaceutical compounds, and 3 antibiotics, were detected in at least one sample and represented a wide range of domestic, industrial, and agricultural origins and uses. Of the OWC detections, 94 percent were less than the RLs and, therefore, were reported as estimated concentrations. Only three reported concentrations (all were fire retardants) were greater than 0.5 µg/L, and only one of these was greater than 1 µg/L (Appendix 2). Reporting levels, minimum and maximum observed concentrations, and frequencies of detection are provided (table 3). Complete results for detected OWCs are listed in Appendix 2.

Types of Compounds Detected

The 10 most frequently detected compounds included 2 synthetic musks (acetyl-hexamethyl tetrahydronaphthalene (AHTN) and hexahydro-hexamethyl cyclopentabenzopyran (HHCB)); 3 fire retardants (tri(2-chloroethyl) phosphate, tri(dichloroisopropyl) phosphate, and tributyl phosphate); an herbicide (metolachlor); a nonprescription stimulant (caffeine); a nicotine metabolite (cotinine); a nonprescription pain killer (acetaminophen); and an antibiotic typically used to treat urinary-tract infections (sulfamethoxazole). These compounds were each detected in more than 20 percent of the samples (table 3).

For interpretive purposes, the detected OWCs were grouped into seven categories that describe their general uses (table 4). Pharmaceuticals (3 compounds) and antibiotics (3 compounds) represented two categories. The remaining 18 detected OWCs represented five categories: fire retardants and plasticizers (6 compounds); fragrances and flavorants (6 compounds); pesticides (3 compounds); disinfectants (1 compound); and other uses (2 compounds).

When all 42 samples were considered collectively (fig. 2), pharmaceutical compounds were detected most frequently (at least one compound was present in 67 percent of the samples), followed by fire retardants and plasticizers (64 percent) and fragrances and flavorants (64 percent), pesticides (50 percent), antibiotics (29 percent), disinfectants (9.5 percent), and compounds with other uses (4.8 percent). At least one OWC was detected in 90 percent of the samples (38 of 42). The detection of multiple OWCs was common, with a median of 3.5 and as many as 12 compounds observed in individual samples. To avoid misinterpreting these statistics, one must consider the fact that many more samples were collected at site 8 (28 samples) than at sites 1–7 (2 samples each);

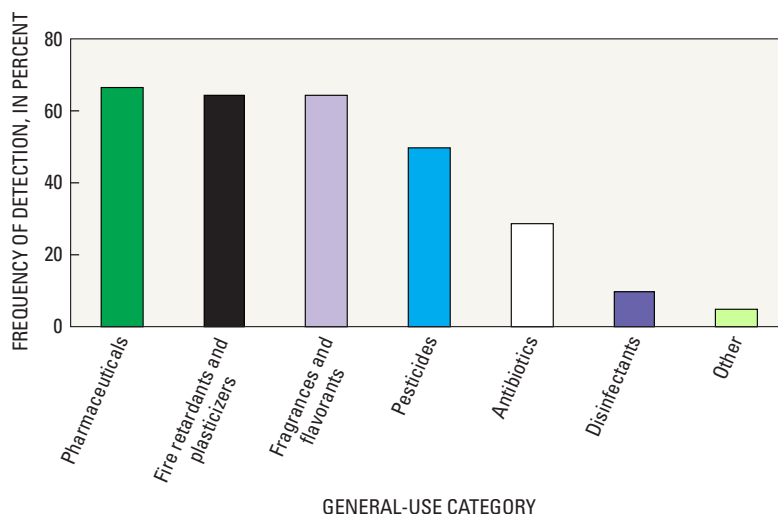


Figure 2. Frequency of detection of organic wastewater compounds in all study samples, Triangle Area of North Carolina, 2002–2005.

Table 4. Detections of dissolved organic wastewater compounds in the Triangle Area of North Carolina, by site and general-use category, 2002–2005.

[Compounds in **bold** text are known or suspected endocrine-disrupting compounds; n, number of samples collected at each site; ns, not sampled; PHARM, pharmaceutical; ANTI, antibiotic; FIRE, fire retardant; PLASTIC, plasticizer; FRAG, fragrance; FLAV, flavorant; PEST, pesticide; DISINF, disinfectant; OTHER, other use]

General-use category	Organic wastewater compound	Total number of detections	Total number of samples	Number of times compound was detected at each site (fig. 1; table 1)									
				Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8		
Pharmaceutical compounds													
PHARM	Acetaminophen	4	14	1	0	1	1	0	0	1	0	0	ns
PHARM	Caffeine (method 1)	20	42	0	0	0	0	0	0	0	0	1	19
PHARM	Cotinine (method 2)	6	14	0	1	1	0	0	0	0	2	2	ns
Antibiotics (*degradation product)													
ANTI	Anhydro-erythromycin*	2	14	0	0	0	0	0	0	0	1	1	ns
ANTI	Sulfamethoxazole (method 3)	3	14	0	0	0	0	0	0	0	1	2	ns
ANTI	Trimethoprim	1	14	0	0	0	0	0	0	0	0	1	ns
Fire retardants and plasticizers													
FIRE	Tri(2-butoxyethyl) phosphate	8	42	0	0	0	0	0	0	1	0	0	7
FIRE	Tri(2-chloroethyl) phosphate	26	42	0	1	0	0	0	0	0	2	2	21
FIRE	Tri(dichloroisopropyl) phosphate	16	42	0	0	0	0	0	0	0	0	1	15
FIRE	Tributyl phosphate	12	42	0	0	0	0	0	1	0	0	1	10
PLASTIC	Triethyl citrate (ethyl citrate)	8	42	0	0	0	0	0	0	0	0	0	8
PLASTIC	Triphenyl phosphate	8	42	0	0	0	0	0	0	1	0	0	7
Fragrances and flavorants													
FRAG	3-methyl-1H-indole (skatol)	1	42	0	0	0	0	0	0	0	0	0	1
FRAG	Acetyl-hexamethyl tetrahydro-naphthalene (AHTN)	19	42	0	0	0	0	0	0	0	0	0	19
FRAG	Hexahydro-hexamethyl cyclopentabenzopyran (HHCB)	26	42	0	0	0	0	0	0	0	0	0	26
FRAG	Indole	1	42	0	0	0	0	0	0	0	0	0	1
FLAV	Camphor	6	42	0	0	1	0	0	0	0	0	0	5
FLAV	Menthol	2	42	0	0	1	0	0	0	0	0	0	1
Pesticides													
PEST	Diazinon	2	42	0	0	0	0	0	0	0	0	0	2
PEST	Metolachlor	21	42	0	0	1	2	0	0	0	0	1	17
PEST	Prometon	2	42	0	0	1	0	0	0	0	0	0	1
Disinfectants													
DISINF	Triclosan	4	42	0	0	0	1	0	0	0	0	0	3
Other													
OTHER	Anthraquinone	1	42	0	0	0	0	0	0	0	0	0	1
OTHER	para-Cresol	1	42	0	0	0	0	0	0	0	0	0	1
Total number of organic wastewater compounds detected				1	2	6	3	3	3	5	9	9	19

therefore, the results for site 8 skew the frequencies of detection for some compounds. Additional details regarding the distribution of OWCs among the eight sites are provided later in this report.

The three pharmaceutical compounds detected in this study include caffeine, cotinine, and acetaminophen. Caffeine and cotinine accounted for the majority of the detections in this category and are categorized as pharmaceuticals because they are metabolically active. Generally, caffeine and cotinine are not administered for medicinal purposes but rather enter the waste stream primarily through the consumption of caffeinated beverages and tobacco products. Caffeine was detected in 20 samples but at only two of the eight sites. Acetaminophen and cotinine each were detected at four of the seven sites where they were sampled (table 4).

The six fire retardants and plasticizers that were detected include tri(2-butoxyethyl) phosphate, tri(2-chloroethyl) phosphate, tri(dichloroisopropyl) phosphate, tributyl phosphate, triethyl citrate, and triphenyl phosphate. These compounds are present in a broad range of consumer products, including automobile and electronic components, plastics, foams, textiles, and building materials (Appendix 1).

The six fragrances and flavorants that were detected include skatol, AHTN, HHCB, indole, camphor, and menthol. These compounds are used in a variety of personal-care and household products, foods, and beverages (Appendix 1). AHTN and HHCB, two polycyclic musk fragrances that are known endocrine disruptors, were detected in a large percentage (68 and 93 percent, respectively) of the 28 samples at site 8 but were not detected at other sites.

The three pesticides that were detected included two widely used herbicides (metolachlor and prometon) and one organophosphorus insecticide (diazinon). Triclosan is a disinfectant used in antibacterial soaps and other cleansers. The remaining two detected OWCs included anthraquinone, which is used in manufacturing dyes and in the paper industry, and *para*-cresol, which is used as a wood preservative and solvent and in manufacturing a variety of materials (Appendix 1).

During this study, antibiotics were sampled twice at sites 1–7, for a total of 14 samples. Three antibiotics were detected, and all detections occurred at sites 6 and 7 in Jordan Lake (table 4). Anhydro-erythromycin is a degradation product of erythromycin, which is widely prescribed for bacterial infections, particularly of the respiratory tract. Sulfamethoxazole and trimethoprim commonly are used in combination to treat urinary-tract infections.

Distribution of Detected Compounds

The distribution of OWCs varied among water-supply sites in the Triangle Area (fig. 3; table 4). Pharmaceuticals

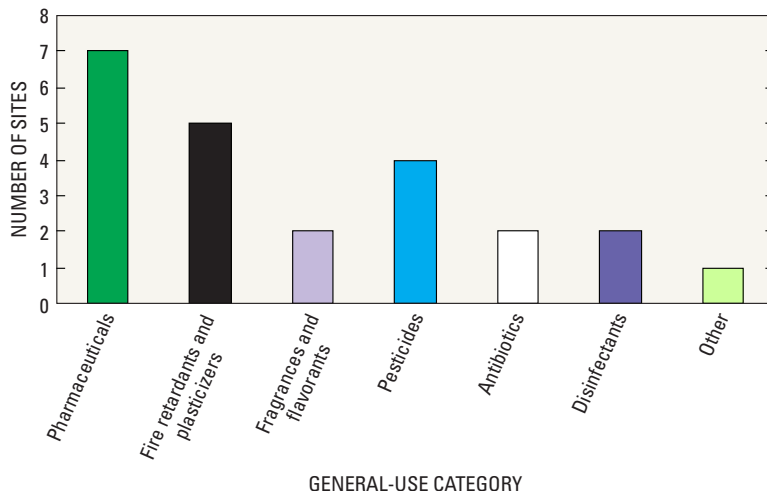


Figure 3. Number of sites in the Triangle Area of North Carolina at which each category of organic wastewater compounds was detected, 2002–2005.

were detected at seven of the eight sites. Fire retardants and plasticizers were detected at five sites, and pesticides were detected at four sites. The remaining four categories of OWCs (fragrances and flavorants, antibiotics, disinfectants, and other-use compounds) were detected at only one or two sampling locations.

Some individual compounds, including acetaminophen, cotinine, and metolachlor, were detected at several sites and in multiple samples, indicating that sources of these compounds are widely distributed throughout the study area. Other OWCs, including AHTN and HHCB, were detected in a high percentage of samples but at only one location, indicating that sources of these compounds are more site specific (table 4; Appendix 2).

At least one OWC was detected at every site that was sampled (fig. 4; table 4). For sites 1–7, the number of detected OWCs ranged from one compound at site 1 (Eno River at Hillsborough) to nine compounds at site 7 (Jordan Lake at Bells Landing). The number of OWCs detected in individual samples from these sites ranged from 0 to 6 with a median of 2 compounds. Because sites 1–7 were each sampled only twice, the data provided a preliminary reconnaissance of OWC occurrence at these locations.

Site 8 was sampled a total of 28 times over a range of hydrologic conditions. Nineteen OWCs were detected among all samples collected from this location (fig. 4; table 4). The number of OWCs detected in individual samples ranged from 0 to 12 with a median of 5 compounds. Samples from site 8 were analyzed using only method 1; therefore, the number of OWCs detected may have been greater if samples had been analyzed using all three methods.

The OWCs that were detected in the Triangle Area have multiple uses and sources (Appendix 1). Identifying specific sources was beyond the scope of this investigation; nevertheless, some general observations may be made regarding the distribution of OWCs among sampling locations. Results

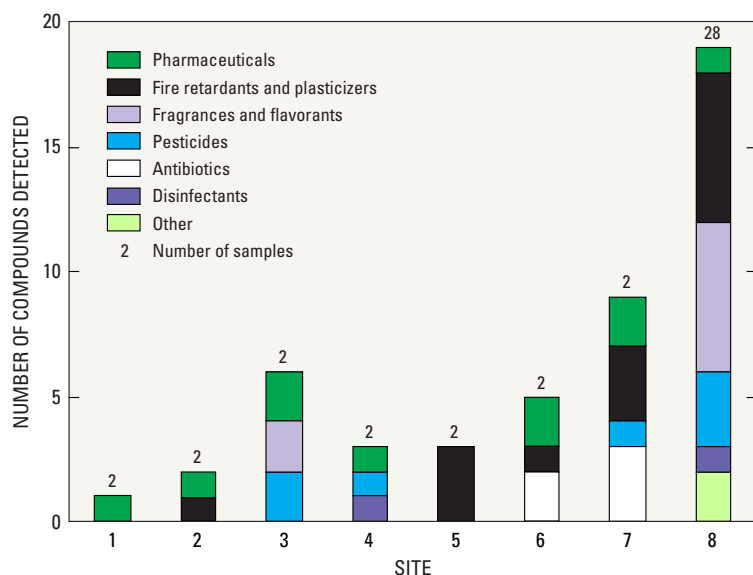


Figure 4. Number of organic wastewater compounds, grouped by general-use category, that were detected at each site in the Triangle Area of North Carolina, 2002–2005.

indicate that both point and nonpoint sources may contribute OWCs to Triangle Area water supplies.

Sites 6, 7, and 8 are located downstream from major wastewater discharges (fig. 1). Three municipal WWTPs are located upstream from sites 6 and 7 in Jordan Lake, and six WWTPs are located upstream from site 8 in the Neuse River (table 1). Caffeine was detected only at sites 7 and 8 (table 4). Site 8 had higher concentrations of fragrances and flavorants than sites 1–7 (Appendix 2), which indicates that wastewater may have been a significant contributor of these OWCs. Antibiotics were detected only at sites 6 and 7 and were not sampled at site 8 (table 4), which indicates that municipal WWTPs also may be a source of these compounds. Sampling for antibiotics at site 8 would provide valuable information on the distribution and potential sources of these compounds throughout the Triangle Area. It should also be noted that Jordan Lake is a State recreation area. Numerous public facilities, including restrooms, campgrounds, swimming beaches, dump stations, and boating access, are located near sites 6 and 7 (North Carolina Division of Parks and Recreation, 2006). Potential effects of these facilities on nearby water quality were not assessed as part of this study.

Fire retardants and plasticizers were detected frequently at sites downstream from wastewater point sources and during one sampling event at University Lake (table 4). A maximum concentration of 3.7 $\mu\text{g/L}$ of tri(2-butoxyethyl) phosphate was observed at site 5 (University Lake at dam near Chapel Hill) in April 2004. During the same sampling event, an additional fire retardant and a plasticizer also were detected at this site. Interestingly, a fire at an industrial cleaning-supply warehouse occurred upstream from University Lake 2 months prior to this sampling event. Water that was applied to control the fire entered a tributary to the lake. The effect of this event on the

presence of fire retardants in University Lake is not fully known, but it may have been a contributing factor. No fire retardants or other OWCs were detected at this site during August 2004 (Appendix 2).

Nonpoint sources also may have influenced the occurrence and distribution of OWCs among the Triangle Area sites. In general, a correlation was observed between the number of OWCs detected and the drainage area size (fig. 4; table 1), likely because large basins tend to contain more potential sources. For example, in addition to being influenced by upstream WWTPs, site 8 also receives runoff from large amounts of agricultural and developed land in its 1,200-square-mile watershed (fig. 1). The herbicide metolachlor was detected at numerous locations in the Triangle Area, and concentrations were similar among sites (table 4; Appendix 2), indicating that nonpoint sources are responsible for delivering this compound to surface waters. These results are consistent with the findings of previous reconnaissance studies, which also have indicated that herbicides likely originate from nonpoint-source inputs rather than treated wastewater (Kolpin and others, 2004; Lee and others, 2004; Sando and others, 2005).

Comparison with Water-Quality Benchmarks

No sample results exceeded Federal or State water-quality standards; however, human health or aquatic life benchmarks are not available for most of the OWCs included during this study. The U.S. Environmental Protection Agency (2004) has established enforceable drinking-water standards, also known as Maximum Contaminant Levels, for only five of the OWCs that were analyzed (Appendix 1), and none of these OWCs were detected in this study. The U.S. Environmental Protection Agency (2004) has established Lifetime Health Advisories (LHA) for 10 of the target OWCs (Appendix 1), and 3 of these were detected: diazinon (LHA = 0.6 $\mu\text{g/L}$); metolachlor (LHA = 100 $\mu\text{g/L}$); and prometon (LHA = 100 $\mu\text{g/L}$). The LHA is the concentration of a chemical in drinking water that is not expected to cause adverse, non-carcinogenic effects for a lifetime of exposure, and is not an enforceable standard.

North Carolina water-quality criteria are developed to protect human health and/or aquatic organisms. The North Carolina Department of Environment and Natural Resources has established ambient water-quality criteria for 23 of the OWCs included in this study, primarily for pesticides and polycyclic aromatic hydrocarbons (Appendix 1; Connie Brower, North Carolina Division of Water Quality, written commun., April 12, 2006). Three of these compounds were detected during this study, including diazinon (aquatic life criterion = 0.10 $\mu\text{g/L}$), metolachlor (water-supply criterion = 4,700 $\mu\text{g/L}$), and *para*-cresol (water-supply criterion = 170 $\mu\text{g/L}$). In comparison, the maximum concentrations reported for this study were 0.02 $\mu\text{g/L}$ for diazinon, 0.11 $\mu\text{g/L}$

for metolachlor, 0.094 µg/L for prometon, and 0.39 µg/L for *para*-cresol (table 3), all well below available benchmark concentrations.

Comparison with Results from Other Areas

Several differences among this study and previous investigations have been noted. These differences make it somewhat problematic to compare results obtained from the Triangle Area of North Carolina with findings from other areas. Some previous studies included results for WWTP effluents, ground water, drinking water, or other non-surface water samples (Frick and Zaugg, 2003; Lee and others, 2004; Glassmeyer and others, 2005; Sando and others, 2005; Sprague and Battaglin, 2005). When sufficient information was available, results for only surface-water sites were extracted for comparison with the Triangle Area study. In spite of these differences, previous studies provide a context for understanding the Triangle Area results, particularly for evaluating the occurrence of various OWCs.

Previous reconnaissance studies showed high rates of OWC detection, with 80 to 100 percent of samples containing one or more compound (Kolpin and others, 2002; Frick and Zaugg, 2003; Kolpin and others, 2004; Lee and others, 2004; Glassmeyer and others, 2005; Sando and others, 2005; Sprague and Battaglin, 2005). Wastewater, including treated municipal effluents, landfill leachate, and water underlying feedlot lagoons, consistently contained the greatest number and highest concentrations of OWCs, followed by urban streams. It should be noted that many of the previous studies focused on urban settings; therefore, data for rural areas were less prevalent. Sampling of reference sites also has been limited; however, the data that are available indicate that DEET and nonprescription drugs, including caffeine, acetaminophen, and methyl salicylate, have been detected at low concentrations in streams with minimal human influence (Glassmeyer and others, 2005; Sprague and Battaglin, 2005). No OWCs were detected at a reference lake located in Minnesota (Lee and others, 2004) and at a remote location in Montana (Glassmeyer and others, 2005).

Fewer OWCs were detected at the Triangle-area water-supply sites (24 of 126 analyzed) than in surface waters sampled in other areas of the United States. In Colorado, 57 of 62 OWCs were detected in streams (Sprague and Battaglin, 2005). Sixty-two of 105 OWCs were detected in Iowa streams (Kolpin and others, 2004), and 56 of 91 OWCs were detected in Minnesota streams (Lee and others, 2004). Thirty-nine of 46 OWCs were detected at surface-water sites in Georgia (Frick and Zaugg, 2003), and 39 of 125 OWCs were detected in South Dakota (Sando and others, 2005). Seventy-eight of 110 OWCs were detected at 10 urban areas across the United States (Glassmeyer and others, 2005); however, these detection rates included results for WWTP effluents.

Fire retardants, fragrances, metolachlor, triclosan, nonprescription pharmaceuticals, and antibiotics were

measured commonly in natural waters across the United States and also were observed at water-supply sites in the Triangle Area of North Carolina. These chemicals are widely used by households, industry, and agriculture. Concern is rising that fire retardants are persistent in the environment, are potentially carcinogenic, and bioaccumulate in living tissue—characteristics that are shared with now-banned PCBs. In the Triangle Area, fire retardants were measured primarily at sites downstream from municipal wastewater discharges and at a site downstream from an industrial fire. The fragrances AHTN and HHCb, which were detected at one location in the Triangle Area, are widely used in personal-care products and are known endocrine disrupting compounds. Triclosan is of concern because it may increase the antibacterial resistance of native microbes, and it is a suspected endocrine disrupting compound. Pharmaceutical compounds that were detected frequently in waters across the United States, as well as in the Triangle Area, include caffeine, acetaminophen, and cotinine, all of which are nonprescription substances. The prescription antibiotics sulfamethoxazole, trimethoprim, and anhydro-erythromycin (a metabolite of erythromycin) also were commonly detected in national and regional investigations of OWCs, as well as in the Triangle Area.

In contrast, other antibiotics and various prescription medications that were detected frequently in other areas of the United States were not detected at reportable concentrations at the Triangle Area sites. Several HIA compounds commonly detected in other areas (including cholesterol and other sterols, DEET, and detergent metabolites) either were not detected in this study or estimated concentrations were reported as nondetections because of QA concerns.

As noted previously, differences in types of sampling locations and analyses of filtered and unfiltered samples may account for some of the differences in OWC concentrations observed among studies. In general, concentrations of individual OWCs that were detected in the Triangle Area of North Carolina were within ranges reported for surface-water sites throughout the United States. Overall, concentrations of OWCs detected in North Carolina were most consistent with those observed in a study of South Dakota streams; however, the South Dakota streams had higher concentrations of antibiotics and acetaminophen (Sando and others, 2005).

Maximum concentrations of pharmaceuticals, antibiotics, pesticides, fragrances, and triclosan at all Triangle Area sites—even those downstream from WWTPs—generally were substantially lower than maximums observed in several areas of the United States (Kolpin and others, 2002; Frick and Zaugg, 2003; Kolpin and others, 2004; Lee and others, 2004; Glassmeyer and others, 2005; and Sprague and Battaglin, 2005). It should be noted that reconnaissance studies in Minnesota (Lee and others, 2004) and the Chattahoochee River system of Georgia (Frick and Zaugg, 2003) included results for wastewater effluent, so it is not surprising that maximum values reported from these studies were higher than those reported for the Triangle Area of North Carolina. In contrast, the maximum concentrations of the various fire retardants that

were detected throughout the Triangle Area were consistent with those observed elsewhere (Kolpin and others, 2002; Frick and Zaugg, 2003; Kolpin and others, 2004; Glassmeyer and others, 2005; Sando and others, 2005), with the exception of Colorado streams, which had higher maximums (Sprague and Battaglin, 2005).

Summary

Organic wastewater compounds are classes of compounds that include household, industrial, and agricultural-use compounds, pharmaceutical drugs, and antibiotics. OWCs are characterized by high usage rates, their potential toxicological effects, and continuous release into the environment through human activities. This study describes the occurrence of OWCs at eight river and reservoir sites classified for water-supply use in the Triangle Area of North Carolina, from October 2002 to July 2005. All analyses were performed on filtered water samples; thus, results may not represent the entire concentrations of some compounds, particularly those that tend to adsorb to particulate matter.

Of the 24 OWCs detected, 3 were nonprescription pharmaceutical compounds, 3 were antibiotics, 6 were fire retardants and plasticizers, 6 were fragrances and flavorants, 3 were pesticides, 1 was an antimicrobial disinfectant, and 2 compounds were used for other purposes. The 10 most frequently detected compounds were present in over 20 percent of the samples and included 2 synthetic musks (acetyl-hexamethyl tetrahydronaphthalene (AHTN) and hexahydro-hexamethyl cyclopentabenzopyran (HHCB)), 3 fire retardants (tri(2-chloroethyl) phosphate, tri(dichloroisopropyl) phosphate, and tributyl phosphate), an herbicide (metolachlor), caffeine, a nicotine metabolite (cotinine), a nonprescription pain killer (acetaminophen), and an antibiotic typically used to treat urinary-tract infections (sulfamethoxazole). Four OWCs detected in this study—AHTN, HHCB, diazinon, and triclosan—are known or suspected to disrupt endocrine systems in fish.

At least one OWC was detected at every sampling site. Site comparisons should be interpreted cautiously, however, because relatively few samples were collected at sites 1–7 and a disproportionately high number of samples was collected at site 8. The most (19) OWCs were detected at the Neuse River above U.S. 70 at Smithfield (site 8), where two-thirds of the total number of samples were collected. The fewest OWCs (1) were detected at the Eno River at Hillsborough (site 1). The detection of multiple OWCs was common, with a median of 3.5 and as many as 12 compounds observed in individual samples.

Some OWCs, including acetaminophen, cotinine, tri(2-chloroethyl) phosphate, and metolachlor, were detected at numerous sites and in numerous samples, indicating that they are widely distributed in the environment. Other OWCs, including AHTN and HHCB, were detected in a high percent-

age of samples but at only one location (site 8), indicating that sources of these compounds are more site specific. Antibiotics were detected at sites 6 and 7 in Jordan Lake. Results indicate that municipal wastewater may have been a source of the synthetic musks and antibiotics; however, the three sites in this study that were located downstream from wastewater discharges also received runoff from agricultural, urban, and rural residential lands. Source identification was beyond the scope of this study.

Concentrations of detected OWCs generally were less than 0.5 $\mu\text{g/L}$ and were within ranges observed at other surface-water locations across the United States. A maximum concentration of 3.7 $\mu\text{g/L}$ of tri(2-butoxyethyl) phosphate (and lesser concentrations of two other fire retardants and plasticizers) was observed at University Lake near Chapel Hill in April 2004. A fire occurred at an industrial cleaning-supply warehouse upstream from this site in February 2004 and may have contributed to the fire retardants observed in the lake. Maximum concentrations of fire retardants observed in the Triangle Area were similar to maximums reported for other areas of the United States, but maximum concentrations of most other OWCs tended to be lower at the Triangle Area sites than in other study areas. Some of these differences may be attributable to variations in site selection and analytical methodology.

This study demonstrated that OWCs are present in surface waters around the Triangle Area of North Carolina. Compounds that were detected have a variety of domestic, commercial and industrial, and agricultural uses and likely enter the environment through a variety of pathways. No compound concentrations in this study exceeded drinking-water criteria adopted by the U.S. Environmental Protection Agency or ambient water-quality criteria established by the North Carolina Department of Environment and Natural Resources; however, guidelines do not exist for most of the OWCs analyzed. Therefore, the potential implications of the presence of these compounds for human or aquatic health are not known. Samples frequently contained multiple OWCs, and the effects of exposure to mixtures of compounds is even more poorly understood. The OWCs that were analyzed represent only a small fraction of the chemicals being produced and used in the United States and around the world; however, this information is useful as a starting point for examining the occurrence and distribution of OWCs in the Triangle Area of North Carolina.

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Appendix 1. Common uses and sources of organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005.

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. MCL, maximum contaminant level; LHA, lifetime health advisory; µg/L, micrograms per liter; NC, North Carolina; CASRN, Chemical Abstracts Service Registry Number; nd, not determined. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	General-use category ^a	MCL (LHA) ^b (µg/L)	NC criterion (µg/L) ^c	CASRN ^d	Common use or source
1	1,4-dichlorobenzene	PEST	75	488	106-46-7	Deodorizer; moth repellent; fumigant; building and construction materials; suspected EDC
1	1-methylnaphthalene	PAH	nd	450	90-12-0	Component of gasoline, diesel, and crude; fossil fuel combustion
1	2,6-dimethylnaphthalene	PAH	nd	nd	581-42-0	Indicator of diesel and kerosene, but not a significant component in gasoline
1	2-methylnaphthalene	PAH	nd	72	91-57-6	Component of gasoline, diesel, and crude; fossil fuel combustion
1	3-methyl-1H-indole (skatol)	FRAG	nd	nd	83-34-1	Fragrance; feces and coal tar
1	3-tert-butyl-4-hydroxyanisole (BHA)	ANTIOX	nd	nd	25013-16-5	Food antioxidant and preservative; known EDC
1	4-cumylphenol	NID	nd	nd	599-64-4	Detergent/surfactant or metabolite; known EDC
1	4-normal-octylphenol	NID	nd	nd	1806-26-4	Detergent/surfactant or metabolite; known EDC
1	4-tert-octylphenol	NID	nd	nd	140-66-9	Detergent/surfactant or metabolite; known EDC
1	5-methyl-1H-benzotriazole	ANTIOX	nd	nd	136-85-6	Antioxidant in antifreeze and deicers; anticorrosive agent
1	Acetophenone	FRAG	nd	nd	98-86-2	Fragrance in personal care products and detergent; flavoring in foods, beverages, and tobacco; solvent for plastics and resins; paint and varnish removers
1	Acetyl-hexamethyl tetrahydro-naphthalene (AHTN)	FRAG	nd	nd	21145-77-7	Synthetic, polycyclic musk fragrance with widespread usage; known EDC
1	Anthracene	PAH	nd	9,600	120-12-7	Wood preservative; component of coal tar, diesel, and crude; suspected EDC
1	Anthraquinone	OTHER	nd	nd	84-65-1	Manufacturing of dyes and pigments; bird repellent for seeds; catalyst in pulp and paper industry
1	Benzo[a]pyrene	PAH	0.2	0.003	50-32-8	Combustion of fossil fuels and other organic material; known EDC
1	Benzophenone	OTHER	nd	nd	119-61-9	Fixative for perfumes and soaps; ultra-violet light blocker; flavoring agent; manufacturing medical products and insecticides; suspected EDC
1	Bisphenol-A	PLAS	nd	nd	80-05-7	Manufacturing of plastics and resins; antioxidant; known EDC
1	Bromacil	PEST	(90)	nd	314-40-9	Herbicide used primarily for grass and weed control; suspected EDC
1	Bromoform	DISINF	80	4.3	75-25-2	Trihalomethane by-product of water chlorination; MCL listed is for total trihalomethanes
1	Camphor	FLAV	nd	nd	76-22-2	Moth repellent; medicinal ointments; flavorant; manufacturing plastics and explosives; embalming
1	Carbaryl	PEST	(700)	38	63-25-2	Carbamate insecticide for crop and garden uses, and pet flea and tick products; known EDC
1	Carbazole	PEST	nd	1	86-74-8	Manufacturing of pharmaceuticals, agrochemicals, dyes, and pigments; also an insecticide
1	Chlorpyrifos	PEST	(20)	0.041	2921-88-2	Organophosphate insecticide used in domestic and agricultural settings for pest/termite control; highly restricted; known EDC
1	Diazinon	PEST	(0.6)	0.1	333-41-5	Organophosphate insecticide used in domestic and agricultural settings for a wide variety of insects and grubs; restricted use; known EDC

Appendix 1. Common uses and sources of organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. MCL, maximum contaminant level; LHA, lifetime health advisory; µg/L, micrograms per liter; NC, North Carolina; CASRN, Chemical Abstracts Service Registry Number; nd, not determined. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	General-use category ^a	MCL (LHA) ^b (µg/L)	NC criterion (µg/L) ^c	CASRN ^d	Common use or source
1	Dichlorvos	PEST	nd	17	62-73-7	Organophosphate insecticide used in food storage areas, greenhouses, and barns; also in pet collars and pest strips; veterinary treatment for parasitic worms; restricted use; suspected EDC
1	d-Limonene	FRAG	nd	nd	5989-27-5	Antimicrobial; antiviral; solvent; fragrance in aerosols
1	Fluoranthene	PAH	nd	300	206-44-0	Component of coal tar and asphalt
1	Hexahydro-hexamethyl cyclopentabenzopyran (HHCb)	FRAG	nd	nd	1222-05-5	Synthetic, polycyclic musk fragrance with widespread usage; known EDC
1	Indole	FRAG	nd	nd	120-72-9	Fragrance in perfumes; flavorant in beverages; inert ingredient in pesticides; occurs in coal tar and feces
1	Isoborneol	FRAG	nd	nd	124-76-5	Fragrance (pine odor), flavorant, and disinfectant; substitute for camphor
1	Isophorone	SOLV	(100)	36	78-59-1	Solvent for printing inks, paints, lacquers, adhesives, vinyl resins, copolymers, industrial coatings, and pesticides
1	Isopropylbenzene (cumene)	SOLV/PAH	nd	310	98-82-8	Solvent for paints, lacquers, and enamels; component of high-octane aviation fuel; used in styrene, acetone, and phenol production
1	Isoquinoline	FLAV	nd	nd	119-65-3	Flavor and fragrance; widely used in manufacturing
1	Menthol	FLAV	nd	nd	89-78-1	Flavor or fragrance in cigarettes, cough drops, mouthwash, liniments, and other pharmaceutical and personal care products
1	Metalaxyl	PEST	nd	nd	57837-19-1	Agricultural fungicide for soil pathogens, mildew, and blight; used on tobacco, ornamentals, conifers, turf, and some food crops, and as a seed treatment
1	Methyl salicylate	OTHER	nd	nd	119-36-8	Used in liniments, food, beverages, and ultra-violet-adsorbing lotions
1	Metolachlor	PEST	(100)	4,700	51218-45-2	High-use, broad-spectrum herbicide used on numerous food crops, ornamental plants, cotton, and highway rights-of-way; primarily used as a pre-emergence herbicide in agricultural settings
1	Naphthalene	PAH	(100)	78	91-20-3	Moth repellent; component of gasoline; also used in dyes, resins, paints, sealants, tanning agents, and insecticides; produced during combustion of fossil fuels, tobacco, or wood
1	N,N-diethyl-meta-toluamide (DEET)	PEST	nd	nd	134-62-3	Insect repellent, primarily used to prevent bites from mosquitoes and ticks
1	Nonylphenol diethoxylate (NP2EO)	NID	nd	nd	26027-38-2	Detergent/surfactant or metabolite; known EDC
1	Octylphenol diethoxylate (OP2EO)	NID	nd	nd	26636-32-8	Detergent/surfactant or metabolite; known EDC
1	Octylphenol monoethoxylate (OPIEO)	NID	nd	nd	26636-32-8	Detergent/surfactant or metabolite; known EDC
1	<i>para</i> -Cresol	OTHER	nd	170	106-44-5	Wood preservative; manufacturing disinfectants, deodorizers, perfumes, explosives, resins, and pesticides; metal cleaner; solvent; sources include auto exhaust, coal tar and metal refining, and wood pulping; suspected EDC
1	<i>para</i> -nonylphenol (NP)	NID	nd	6.6	84852-15-3	Detergent/surfactant or metabolite; known EDC

Appendix 1. Common uses and sources of organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. MCL, maximum contaminant level; LHA, lifetime health advisory; µg/L, micrograms per liter; NC, North Carolina; CASRN, Chemical Abstracts Service Registry Number; nd, not determined. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	General-use category ^a	MCL (LHA) ^b (µg/L)	NC criterion (µg/L) ^c	CASRN ^d	Common use or source
1	Pentachlorophenol (PCP)	PEST	1	0.28	87-86-5	Wood preservative for utility poles, railroad ties, and wharf pilings; also used as an insecticide and fungicide; suspected EDC
1	Phenanthrene	PAH	nd	0.7	85-01-8	Fossil fuel combustion; coal tar, diesel, and crude oil; manufacturing dyes, explosives, and drugs
1	Phenol	DISINF	(2,000)	300	108-95-2	Disinfectant; manufacturing of resins and numerous medical and industrial organic compounds and dyes
1	Prometon	PEST	(100)	nd	1610-18-0	Triazine herbicide generally used on non-agricultural land to control broadleaf and grassy weeds
1	Pyrene	PAH	nd	20	129-00-0	Combustion of fossil fuels, tobacco, foods, and other organic material; component of coal tar and asphalt; used to make dyes, plastics, pesticides, pharmaceuticals, and benz[a]pyrene
1	Tetrachloroethylene (PCE; PERC)	SOLV	5 (10)	0.8	127-18-4	Solvent and degreaser; used in dry-cleaning products and industrial metal cleaning and finishing
1	Tri(2-butoxyethyl) phosphate	FIRE	nd	nd	78-51-3	Flame retardant and plasticizer
1	Tri(2-chloroethyl) phosphate	FIRE	nd	nd	115-96-8	Flame retardant in plastics, including flexible and rigid foams, resins, PVC, adhesives, resins, and other materials; suspected EDC
1	Tri(dichloroisopropyl) phosphate	FIRE	nd	nd	13674-87-8	Flame retardant in plastics, including flexible and rigid foams, resins, PVC, adhesives, resins, and other materials; suspected EDC
1	Tributyl phosphate	FIRE	nd	nd	126-73-8	Flame retardant; plasticizer; solvent; antifoaming agent
1	Triclosan	DISINF	nd	nd	3380-34-5	Antimicrobial disinfectant; suspected EDC
1	Triethyl citrate (ethyl citrate)	PLAS	nd	nd	77-93-0	Plasticizer and solvent; used in cosmetics and hair-care products, pharmaceuticals, and lacquers, and as a food additive
1	Triphenyl phosphate	PLAS/FIRE	nd	nd	115-86-6	Flame retardant in many plastics and resins, especially in electronics and automobile parts; plasticizer in photographic and other cellulose acetate films, polyester, polyurethane, lacquers and varnishes, and roofing paper
Sterols						
1	3-beta-Coprostanol	STER	nd	nd	360-68-9	Fecal sterol; usually a carnivore indicator
1	beta-Sitosterol	STER	nd	nd	83-46-5	Plant sterol; known EDC
1	beta-Stigmasterol	STER	nd	nd	19466-47-8	Plant sterol
1	Cholesterol	STER	nd	nd	57-88-5	Animal/plant sterol; often a fecal indicator
Pharmaceuticals						
2	1,7-Dimethylxanthine	PHARM	nd	nd	611-59-6	Caffeine metabolite
2	Acetaminophen	PHARM	nd	nd	103-90-2	Antipyretic; analgesic; anti-inflammatory (nonprescription)
1, 2	Caffeine	PHARM	nd	nd	58-08-2	Stimulant; diuretic (nonprescription)
2	Carbamazepine	PHARM	nd	nd	298-46-4	Anticonvulsant; antiepileptic; antineuralgic (prescription)
2	Cimetidine	PHARM	nd	nd	51481-61-9	Antacid (nonprescription)
2	Codeine	PHARM	nd	nd	76-57-3	Analgesic (prescription)
1, 2	Cotinine	PHARM	nd	nd	486-56-6	Nicotine metabolite
2	Dehydronifedipine	PHARM	nd	nd	67035-22-7	Antianginal (prescription)

Appendix 1. Common uses and sources of organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. MCL, maximum contaminant level; LHA, lifetime health advisory; µg/L, micrograms per liter; NC, North Carolina; CASRN, Chemical Abstracts Service Registry Number; nd, not determined. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	General-use category ^a	MCL (LHA) ^b (µg/L)	NC criterion (µg/L) ^c	CASRN ^d	Common use or source
2	Diltiazem	PHARM	nd	nd	33286-22-5	Antihypertensive (prescription)
2	Diphenhydramine	PHARM	nd	nd	147-24-0	Antihistamine (prescription)
2	Fluoxetine	PHARM	nd	nd	54910-89-3	Antidepressant (prescription); known EDC
2	Gemfibrozil	PHARM	nd	nd	25812-30-0	Antihyperlipidemic (prescription)
2	Ibuprofen	PHARM	nd	nd	15687-27-1	Anti-inflammatory; analgesic; antipyretic (nonprescription)
2	Miconazole	PHARM	nd	nd	22916-47-8	Antifungal (nonprescription)
2	Ranitidine	PHARM	nd	nd	66357-35-5	Antacid (nonprescription)
2	Salbutamol	PHARM	nd	nd	18559-94-9	Antiasthmatic (prescription)
2	Thiabendazole	PHARM	nd	nd	148-79-8	Anthelmintic (human and veterinary; prescription)
2	Warfarin	PHARM	nd	nd	81-81-2	Anticoagulant (prescription)
Antibiotics and *degradation products						
3	Amoxicillin	ANTIB	nd	nd	61336-70-7	Antibiotic (human and veterinary; penicillin class)
3	Ampicillin	ANTIB	nd	nd	69-53-4	Antibiotic (human and veterinary; penicillin class)
3	Anhydro-erythromycin*	ANTIB	nd	nd	not available	Erythromycin degradate
3	Anhydro-chlorotetracycline*	ANTIB	nd	nd	4497-08-9	Chlorotetracycline degradate
3	Anhydro-tetracycline*	ANTIB	nd	nd	13803-65-1	Tetracycline degradate
2	Azithromycin	ANTIB	nd	nd	83905-01-5	Antibiotic (human and veterinary; macrolide class)
3	Carbadox	ANTIB	nd	nd	6804-07-5	Antibiotic (veterinary)
3	Cefotaxime	ANTIB	nd	nd	63527-52-6	Antibiotic (human and veterinary; cephalosporin class)
3	Chlorotetracycline	ANTIB	nd	nd	57-62-5	Antibiotic (veterinary; tetracycline class)
3	Ciprofloxacin	ANTIB	nd	nd	85721-33-1	Antibiotic (human and veterinary; fluoroquinolone class)
3	Clinafloxacin	ANTIB	nd	nd	105956-97-6	Antibiotic (human and veterinary; fluoroquinolone class)
3	Cloxacillin	ANTIB	nd	nd	61-72-3	Antibiotic (human and veterinary; penicillin class)
3	Demeclocycline	ANTIB	nd	nd	64-73-3	Antibiotic (human and veterinary; tetracycline class)
3	Doxycycline	ANTIB	nd	nd	564-25-0	Antibiotic (human and veterinary; tetracycline class)
3	Epi-anhydro-chlorotetracycline*	ANTIB	nd	nd	4497-08-9	Chlorotetracycline degradate
3	Epi-anhydro-tetracycline*	ANTIB	nd	nd	4465-65-0	Tetracycline degradate
3	Epi-chlorotetracycline*	ANTIB	nd	nd	14297-93-9	Chlorotetracycline degradate
3	Epi-oxytetracycline*	ANTIB	nd	nd	35259-39-3	Oxytetracycline degradate
3	Epi-tetracycline*	ANTIB	nd	nd	23313-80-6	Tetracycline degradate
2, 3	Erythromycin	ANTIB	nd	nd	114-07-8	Antibiotic (human and veterinary; macrolide class)
3	Flumequine	ANTIB	nd	nd	42835-25-6	Antibiotic (veterinary; fluoroquinolone class)
3	Iso-chlorotetracycline*	ANTIB	nd	nd	514-53-4	Chlorotetracycline degradate
3	Iso-epi-chlorotetracycline*	ANTIB	nd	nd	not available	Chlorotetracycline degradate
3	Lincomycin	ANTIB	nd	nd	154-21-2	Antibiotic (veterinary; lincosamide class)
3	Lomefloxacin	ANTIB	nd	nd	98079-51-7	Antibiotic (human and veterinary; fluoroquinolone class)
3	Minoocycline	ANTIB	nd	nd	10118-90-8	Antibiotic (human and veterinary; tetracycline class)
3	Norfloxacin	ANTIB	nd	nd	70458-96-7	Antibiotic (human and veterinary; fluoroquinolone class)
3	Oflloxacin	ANTIB	nd	nd	82419-36-1	Antibiotic (human and veterinary; fluoroquinolone class)

Appendix 1. Common uses and sources of organic wastewater compounds analyzed in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Analytical method 1 is an official U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Caffeine, cotinine, erythromycin, sulfamethoxazole, and trimethoprim were analyzed by more than one method. MCL, maximum contaminant level; LHA, lifetime health advisory; µg/L, micrograms per liter; NC, North Carolina; CASRN, Chemical Abstracts Service Registry Number; nd, not determined. **Bold** text indicates a known or suspected endocrine-disrupting compound (EDC)]

Analytical method	Organic wastewater compound	General-use category ^a	MCL (LHA) ^b (µg/L)	NC criterion (µg/L) ^c	CASRN ^d	Common use or source
3	Ormetoprim	ANTIB	nd	nd	6981-18-6	Antibiotic (veterinary; folic acid inhibitor)
3	Oxacillin	ANTIB	nd	nd	66-79-5	Antibiotic (human and veterinary; penicillin class)
3	Oxolinic Acid	ANTIB	nd	nd	14698-29-4	Antibiotic (veterinary; quinolone class)
3	Oxytetracycline	ANTIB	nd	nd	79-57-2	Antibiotic (veterinary; tetracycline class)
3	Penicillin G	ANTIB	nd	nd	61-33-6	Antibiotic (human and veterinary; penicillin class)
3	Penicillin V	ANTIB	nd	nd	87-08-1	Antibiotic (human and veterinary; penicillin class)
3	Roxithromycin	ANTIB	nd	nd	80214-83-1	Antibiotic (human and veterinary; macrolide class)
3	Sarafloxacin	ANTIB	nd	nd	98105-99-8	Antibiotic (veterinary; fluoroquinolone class)
3	Sulfachloropyridazine	ANTIB	nd	nd	80-32-0	Antibiotic (veterinary; sulfonamide class)
3	Sulfadiazine	ANTIB	nd	nd	68-35-9	Antibiotic (veterinary and human; sulfonamide class)
3	Sulfadimethoxine	ANTIB	nd	nd	122-11-2	Antibiotic (veterinary; sulfonamide class)
3	Sulfamerazine	ANTIB	nd	nd	127-79-7	Antibiotic (veterinary; sulfonamide class)
3	Sulfamethazine	ANTIB	nd	nd	57-68-1	Antibiotic (veterinary; sulfonamide class); suspected EDC
2, 3	Sulfamethoxazole	ANTIB	nd	nd	723-46-4	Antibiotic (human and veterinary; sulfonamide class); suspected EDC
3	Sulfathiazole	ANTIB	nd	nd	72-14-0	Antibiotic (veterinary; sulfonamide class)
3	Tetracycline	ANTIB	nd	nd	60-54-8	Antibiotic (human and veterinary; tetracycline class)
2, 3	Trimethoprim	ANTIB	nd	nd	738-70-5	Antibiotic (human and veterinary; folic acid inhibitor)
3	Tylosin	ANTIB	nd	nd	1401-69-0	Antibiotic (veterinary; macrolide class)
3	Virginiamycin	ANTIB	nd	nd	21411-53-0	Antibiotic (veterinary; streptogramin class)

^aPEST, pesticide; PAH, polycyclic aromatic hydrocarbons; FRAG, fragrance; ANTIOX, antioxidant; NID, nonionic detergent or metabolite; OTHER, other use; PLAS, plastic component; DISINF, disinfectant; FLAV, flavor; SOLV, solvent; FIRE, fire retardant; STER, plant or animal sterol; PHARM, pharmaceutical; ANTIB, antibiotic. Compounds generally have multiple uses and sources. In this table, compounds are categorized by their most common uses.

^bMaximum contaminant levels (MCL) and lifetime health advisories (LHA) are human-health related standards or advisories (U.S. Environmental Protection Agency, 2004). The MCL is the highest level of a contaminant that is allowed in drinking water. MCLs are enforceable Federal standards that apply to public drinking-water systems. The LHA, listed in parentheses, is the concentration of a chemical in drinking water that is not expected to cause any adverse, non-carcinogenic effects for a lifetime of exposure. LHAs are not legally enforceable standards, but serve as technical guidance to assist Federal, State, and local officials. MCLs and LHAs are available online at <http://www.epa.gov/waterscience/criteria/drinking/standards/dwstandards.pdf>.

^cNorth Carolina Department of Environment and Natural Resources water-quality standard or chronic toxicity criterion. Value listed is the most stringent of applicable criteria for these waters—generally, water-supply standards are most stringent, followed by chronic toxicity criteria for freshwater aquatic life. For more information, including criteria for non-water-supply waters, see http://h2o.enr.state.nc.us/csu/documents/critical103104_000.pdf.

^dNumber that is assigned to a substance when it enters the American Chemical Society's Chemical Abstracts Service (CAS) Registry database, available online at <http://www.cas.org/Support/lookup.html>.

Appendix 2. Results for dissolved organic wastewater compounds detected in water samples collected in the Triangle Area of North Carolina, 2002–2005.

[Compounds in **bold** text are known or suspected endocrine-disrupting compounds; all concentrations are micrograms per liter; concentrations in **bold** text represent the maximum concentration observed during the study; <, less than; E, estimated concentration below reporting level; ns, not sampled. *, two samples were ruined during laboratory analysis; additional samples were collected to replace the ruined samples]

Site number (fig. 1; table 1)	Site location	Sampling date	Organic wastewater compound concentrations (micrograms per liter)								
			3-Methyl-1H-indole	9,10-Anthraquinone	Acetyl-hexamethyl tetrahydro-naphthalene (AHTN)	Caffeine	Camphor	Diazinon	Hexahydro-hexamethyl cyclopenta-benzopyran (HHCB)		
1	Eno River at Hillsborough, NC	4/8/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
1	Eno River at Hillsborough, NC	8/19/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2	Little River Reservoir at dam near Bahama, NC	4/7/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2	Little River Reservoir at dam near Bahama, NC	8/18/2004	*	*	*	*	*	*	*	*	*
2	Little River Reservoir at dam near Bahama, NC	10/25/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
3	Lake Michie at dam near Bahama, NC	4/7/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
3	Lake Michie at dam near Bahama, NC	8/18/2004	<1	<0.5	<0.5	<0.5	<0.5	E0.006	<0.5	<0.5	<0.5
4	Cane Creek Reservoir at dam near White Cross, NC	4/8/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
4	Cane Creek Reservoir at dam near White Cross, NC	8/19/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
5	University Lake at dam near Chapel Hill, NC	4/5/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
5	University Lake at dam near Chapel Hill, NC	8/23/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/20/2004	*	*	*	*	*	*	*	*	*
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	8/24/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/21/2005	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	4/20/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	8/24/2004	<1	<0.5	<0.5	<0.5	E0.035	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	10/24/2002	<1	<0.5	E0.014	E0.017	<0.5	<0.5	E0.016	<0.5	E0.054
8	Neuse River above U.S. 70 at Smithfield, NC	11/13/2002	<1	<0.5	E0.025	E0.078	<0.5	<0.5	<0.5	<0.5	E0.063
8	Neuse River above U.S. 70 at Smithfield, NC	12/12/2002	<1	<0.5	E0.024	E0.086	<0.5	<0.5	<0.5	<0.5	E0.065
8	Neuse River above U.S. 70 at Smithfield, NC	1/16/2003	<1	<0.5	E0.052	E0.045	<0.5	<0.5	<0.5	<0.5	E0.2
8	Neuse River above U.S. 70 at Smithfield, NC	1/31/2003	<1	<0.5	E0.049	E0.17	<0.5	<0.5	<0.5	<0.5	E0.23
8	Neuse River above U.S. 70 at Smithfield, NC	2/11/2003	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	3/20/2003	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	E0.054
8	Neuse River above U.S. 70 at Smithfield, NC	4/9/2003	<1	<0.5	E0.035	E0.04	<0.5	<0.5	<0.5	<0.5	E0.092
8	Neuse River above U.S. 70 at Smithfield, NC	5/7/2003	<1	E0.11	E0.06	E0.097	<0.5	<0.5	<0.5	<0.5	E0.11
8	Neuse River above U.S. 70 at Smithfield, NC	6/4/2003	E0.064	<0.5	E0.036	E0.074	<0.5	<0.5	<0.5	<0.5	E0.068
8	Neuse River above U.S. 70 at Smithfield, NC	7/22/2003	<1	<0.5	E0.084	E0.09	<0.5	<0.5	<0.5	<0.5	E0.097
8	Neuse River above U.S. 70 at Smithfield, NC	8/21/2003	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	9/9/2003	<1	<0.5	E0.018	<0.5	<0.5	<0.5	<0.5	<0.5	E0.07
8	Neuse River above U.S. 70 at Smithfield, NC	10/6/2003	<1	<0.5	E0.018	E0.059	<0.5	<0.5	<0.5	<0.5	E0.12
8	Neuse River above U.S. 70 at Smithfield, NC	11/4/2003	<1	<0.5	E0.069	<0.5	<0.5	<0.5	<0.5	<0.5	E0.13
8	Neuse River above U.S. 70 at Smithfield, NC	12/23/2003	<1	<0.5	E0.043	E0.029	<0.5	<0.5	<0.5	<0.5	E0.049
8	Neuse River above U.S. 70 at Smithfield, NC	12/30/2003	<1	<0.5	E0.052	<0.5	<0.5	<0.5	<0.5	<0.5	E0.14
8	Neuse River above U.S. 70 at Smithfield, NC	6/22/2004	<1	<0.5	E0.051	<0.5	<0.5	E0.02	<0.5	<0.5	E0.12
8	Neuse River above U.S. 70 at Smithfield, NC	8/31/2004	<1	<0.5	<0.5	E0.11	<0.5	<0.5	<0.5	<0.5	E0.086
8	Neuse River above U.S. 70 at Smithfield, NC	10/12/2004	<1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	E0.21
8	Neuse River above U.S. 70 at Smithfield, NC	10/18/2004	<1	<0.5	<0.5	E0.12	<0.5	<0.5	<0.5	<0.5	E0.16
8	Neuse River above U.S. 70 at Smithfield, NC	12/29/2004	<1	<0.5	E0.024	E0.05	<0.5	<0.5	<0.5	<0.5	E0.16
8	Neuse River above U.S. 70 at Smithfield, NC	1/28/2005	<1	<0.5	<0.5	E0.08	<0.5	<0.5	<0.5	<0.5	E0.18
8	Neuse River above U.S. 70 at Smithfield, NC	2/2/2005	<1	<0.5	<0.5	E0.04	<0.5	<0.5	<0.5	<0.5	E0.17
8	Neuse River above U.S. 70 at Smithfield, NC	3/10/2005	<1	<0.5	E0.048	E0.07	E0.009	<0.5	<0.5	<0.5	E0.15
8	Neuse River above U.S. 70 at Smithfield, NC	4/14/2005	<1	<0.5	<0.5	E0.07	E0.021	<0.5	<0.5	<0.5	E0.11
8	Neuse River above U.S. 70 at Smithfield, NC	5/31/2005	<1	<0.5	E0.028	<0.5	E0.008	<0.5	E0.02	<0.5	E0.16
8	Neuse River above U.S. 70 at Smithfield, NC	7/12/2005	<1	<0.5	E0.04	E0.07	E0.011	<0.5	<0.5	<0.5	E0.12
	Minimum detected		E0.064	E0.11	E0.014	E0.017	E0.006	E0.016	E0.016	E0.049	
	Median detected		E0.064	E0.11	E0.04	E0.07	E0.010	E0.018	E0.018	E0.12	
	Maximum detected		E0.064	E0.11	E0.084	E0.17	E0.021	E0.02	E0.02	E0.23	
	Number of detections		1	1	19	20	6	2	2	26	

Appendix 2. Results for dissolved organic wastewater compounds detected in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Compounds in **bold** text are known or suspected endocrine-disrupting compounds; all concentrations are micrograms per liter; concentrations in **bold** text represent the maximum concentration observed during the study; <, less than; E, estimated concentration below reporting level; ns, not sampled. *, two samples were ruined during laboratory analysis; additional samples were collected to replace the ruined samples]

Site number (fig. 1; table 1)	Site location	Sampling date	Organic wastewater compound concentrations (micrograms per liter)						
			Indole	Menthhol	Metolachlor	p-Cresol	Prometon	Tributyl phosphate	Triclosan
1	Eno River at Hillsborough, NC	4/8/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
1	Eno River at Hillsborough, NC	8/19/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
2	Little River Reservoir at dam near Bahama, NC	4/7/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
2	Little River Reservoir at dam near Bahama, NC	8/18/2004	*	*	*	*	*	*	*
2	Little River Reservoir at dam near Bahama, NC	10/25/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
3	Lake Michie at dam near Bahama, NC	4/7/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
3	Lake Michie at dam near Bahama, NC	8/18/2004	<0.5	E0.023	E0.057	<1	E0.094	<0.5	<1
4	Cane Creek Reservoir at dam near White Cross, NC	4/8/2004	<0.5	<0.5	E0.088	<1	<0.5	<0.5	E0.073
4	Cane Creek Reservoir at dam near White Cross, NC	8/19/2004	<0.5	<0.5	E0.091	<1	<0.5	<0.5	<1
5	University Lake at dam near Chapel Hill, NC	4/5/2004	<0.5	<0.5	<0.5	<1	<0.5	E0.055	<1
5	University Lake at dam near Chapel Hill, NC	8/23/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/20/2004	*	*	*	*	*	*	*
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	8/24/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/21/2005	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	4/20/2004	<0.5	<0.5	E0.066	<1	<0.5	E0.059	<1
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	8/24/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	10/24/2002	<0.5	<0.5	E0.012	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	11/13/2002	<0.5	<0.5	E0.07	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	12/12/2002	<0.5	<0.5	E0.059	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	1/16/2003	<0.5	<0.5	<0.5	<1	<0.5	E0.12	<1
8	Neuse River above U.S. 70 at Smithfield, NC	1/31/2003	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	2/11/2003	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	3/20/2003	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	4/9/2003	<0.5	<0.5	E0.02	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	5/7/2003	<0.5	<0.5	E0.074	<1	<0.5	E0.046	<1
8	Neuse River above U.S. 70 at Smithfield, NC	6/4/2003	<0.5	E0.16	E0.066	E0.39	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	7/22/2003	E0.006	<0.5	E0.1	<1	<0.5	E0.11	<1
8	Neuse River above U.S. 70 at Smithfield, NC	8/21/2003	<0.5	<0.5	E0.05	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	9/9/2003	<0.5	<0.5	E0.04	<1	<0.5	E0.026	E0.066
8	Neuse River above U.S. 70 at Smithfield, NC	10/6/2003	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	11/4/2003	<0.5	<0.5	E0.06	<1	<0.5	E0.097	<1
8	Neuse River above U.S. 70 at Smithfield, NC	12/23/2003	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	12/30/2003	<0.5	<0.5	E0.04	<1	<0.5	0.091	E0.082
8	Neuse River above U.S. 70 at Smithfield, NC	6/22/2004	<0.5	<0.5	E0.04	<1	<0.5	E0.09	E0.098
8	Neuse River above U.S. 70 at Smithfield, NC	8/31/2004	<0.5	<0.5	E0.1	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	10/12/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	10/18/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	12/29/2004	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	1/28/2005	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	2/2/2005	<0.5	<0.5	<0.5	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	3/10/2005	<0.5	<0.5	E0.05	<1	<0.5	E0.05	<1
8	Neuse River above U.S. 70 at Smithfield, NC	4/14/2005	<0.5	<0.5	E0.04	<1	<0.5	<0.5	<1
8	Neuse River above U.S. 70 at Smithfield, NC	5/31/2005	<0.5	<0.5	E0.11	<1	<0.5	E0.016	<1
8	Neuse River above U.S. 70 at Smithfield, NC	7/12/2005	<0.5	<0.5	E0.02	<1	E0.04	E0.041	<1
	Minimum detected		E0.006	E0.023	E0.012	E0.39	E0.04	E0.016	E0.066
	Median detected		E0.006	E0.092	E0.059	E0.39	E0.067	E0.078	E0.078
	Maximum detected		E0.006	E0.16	E0.11	E0.39	E0.094	E0.12	E0.098
	Number of detections		1	2	21	1	2	12	4

Appendix 2. Results for dissolved organic wastewater compounds detected in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Compounds in **bold** text are known or suspected endocrine-disrupting compounds; all concentrations are micrograms per liter; concentrations in **bold** text represent the maximum concentration observed during the study; <, less than; E, estimated concentration below reporting level; ns, not sampled. *, two samples were ruined during laboratory analysis; additional samples were collected to replace the ruined samples]

Site number (fig. 1; table 1)	Site location	Sampling date	Organic wastewater compound concentrations (micrograms per liter)				
			Triethyl citrate	Triphenyl phosphate	Tri(2-bu-toxyethyl) phosphate	Tri(2-chlo-roethyl) phosphate	Tri(dichloro-isopropyl) phosphate
1	Eno River at Hillsborough, NC	4/8/2004	<0.5	<0.5	<0.5	<0.5	<0.5
1	Eno River at Hillsborough, NC	8/19/2004	<0.5	<0.5	<0.5	<0.5	<0.5
2	Little River Reservoir at dam near Bahama, NC	4/7/2004	<0.5	<0.5	<0.5	<0.5	*
2	Little River Reservoir at dam near Bahama, NC	8/18/2004	*	*	*	*	*
2	Little River Reservoir at dam near Bahama, NC	10/25/2004	<0.5	<0.5	<0.5	E0.056	<0.5
3	Lake Michie at dam near Bahama, NC	4/7/2004	<0.5	<0.5	<0.5	<0.5	<0.5
3	Lake Michie at dam near Bahama, NC	8/18/2004	<0.5	<0.5	<0.5	<0.5	<0.5
4	Cane Creek Reservoir at dam near White Cross, NC	4/8/2004	<0.5	<0.5	<0.5	<0.5	<0.5
4	Cane Creek Reservoir at dam near White Cross, NC	8/19/2004	<0.5	<0.5	<0.5	<0.5	<0.5
5	University Lake at dam near Chapel Hill, NC	4/5/2004	<0.5	E0.051	3.7	<0.5	<0.5
5	University Lake at dam near Chapel Hill, NC	8/23/2004	<0.5	<0.5	<0.5	<0.5	<0.5
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/20/2004	*	*	*	*	*
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	8/24/2004	<0.5	<0.5	<0.5	E0.095	<0.5
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/21/2005	<0.5	<0.5	<0.5	E0.026	<0.5
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	4/20/2004	<0.5	<0.5	<0.5	0.7	E0.12
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	8/24/2004	<0.5	<0.5	<0.5	E0.1	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	10/24/2002	<0.5	E0.0052	<0.5	E0.033	E0.031
8	Neuse River above U.S. 70 at Smithfield, NC	11/13/2002	<0.5	E0.04	E0.25	E0.059	E0.057
8	Neuse River above U.S. 70 at Smithfield, NC	12/12/2002	<0.5	<0.5	<0.5	E0.047	E0.049
8	Neuse River above U.S. 70 at Smithfield, NC	1/16/2003	<0.5	<0.5	<0.5	E0.056	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	1/31/2003	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	2/11/2003	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	3/20/2003	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	4/9/2003	<0.5	<0.5	<0.5	E0.05	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	5/7/2003	<0.5	<0.5	<0.5	E0.14	E0.078
8	Neuse River above U.S. 70 at Smithfield, NC	6/4/2003	<0.5	<0.5	<0.5	E0.079	E0.053
8	Neuse River above U.S. 70 at Smithfield, NC	7/22/2003	E0.098	<0.5	E0.24	E0.13	E0.12
8	Neuse River above U.S. 70 at Smithfield, NC	8/21/2003	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	9/9/2003	E0.028	<0.5	<0.5	E0.074	E0.089
8	Neuse River above U.S. 70 at Smithfield, NC	10/6/2003	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	11/4/2003	E0.081	E0.052	E0.13	E0.11	E0.1
8	Neuse River above U.S. 70 at Smithfield, NC	12/23/2003	<0.5	<0.5	<0.5	E0.086	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	12/30/2003	E0.063	E0.044	<0.5	E0.1	E0.072
8	Neuse River above U.S. 70 at Smithfield, NC	6/22/2004	E0.072	E0.048	E0.15	E0.14	E0.12
8	Neuse River above U.S. 70 at Smithfield, NC	8/31/2004	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	10/12/2004	<0.5	<0.5	<0.5	E0.18	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	10/18/2004	<0.5	<0.5	<0.5	<0.5	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	12/29/2004	<0.5	<0.5	<0.5	E0.23	E0.049
8	Neuse River above U.S. 70 at Smithfield, NC	1/28/2005	<0.5	<0.5	<0.5	E0.26	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	2/2/2005	<0.5	<0.5	<0.5	E0.22	E0.054
8	Neuse River above U.S. 70 at Smithfield, NC	3/10/2005	E0.029	E0.022	<0.5	E0.14	E0.069
8	Neuse River above U.S. 70 at Smithfield, NC	4/14/2005	<0.5	<0.5	<0.5	E0.039	<0.5
8	Neuse River above U.S. 70 at Smithfield, NC	5/31/2005	E0.02	<0.5	E0.16	E0.093	E0.098
8	Neuse River above U.S. 70 at Smithfield, NC	7/12/2005	E0.029	E0.012	E0.2	E0.1	E0.15
	Minimum detected		E0.02	0.005	E0.13	E0.033	E0.031
	Median detected		E0.046	0.042	E0.22	E0.1	E0.075
	Maximum detected		E0.098	0.052	3.7	0.7	E0.15
	Number of detections		8	8	8	26	16

Appendix 2. Results for dissolved organic wastewater compounds detected in water samples collected in the Triangle Area of North Carolina, 2002–2005. — Continued

[Compounds in **bold** text are known or suspected endocrine-disrupting compounds; all concentrations are micrograms per liter; concentrations in **bold** text represent the maximum concentration observed during the study; <, less than; E, estimated concentration below reporting level; ns, not sampled. *; two samples were ruined during laboratory analysis; additional samples were collected to replace the ruined samples]

Site number (fig. 1; table 1)	Site location	Sampling date	Organic wastewater compound concentrations (micrograms per liter)				
			Acetaminophen	Continine	Anhydro-erythromycin	Sulfamethoxazole	Trimethoprim
1	Eno River at Hillsborough, NC	4/8/2004	0.037	<0.014	<0.05	<0.05	<0.05
1	Eno River at Hillsborough, NC	8/19/2004	<0.036	<0.014	<0.005	<0.005	<0.005
2	Little River Reservoir at dam near Bahama, NC	4/7/2004	<0.036	<0.014	<0.05	<0.05	<0.05
2	Little River Reservoir at dam near Bahama, NC	8/18/2004	<0.036	E0.008	<0.005	<0.005	<0.005
2	Little River Reservoir at dam near Bahama, NC	10/25/2004	ns	ns	ns	ns	ns
3	Lake Michie at dam near Bahama, NC	4/7/2004	E0.01	<0.014	<0.05	<0.05	<0.05
3	Lake Michie at dam near Bahama, NC	8/18/2004	<0.036	E0.013	<0.005	<0.005	<0.005
4	Cane Creek Reservoir at dam near White Cross, NC	4/8/2004	E0.014	<0.014	<0.01	<0.005	<0.01
4	Cane Creek Reservoir at dam near White Cross, NC	8/19/2004	<0.036	<0.014	<0.005	<0.005	<0.005
5	University Lake at dam near Chapel Hill, NC	4/5/2004	<0.036	<0.014	<0.05	<0.05	<0.05
5	University Lake at dam near Chapel Hill, NC	8/23/2004	<0.036	<0.014	<0.005	<0.005	<0.005
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/20/2004	E0.005	E0.008	<0.01	0.033	<0.01
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	8/24/2004	<0.036	0.020	0.055	<0.005	<0.005
6	Jordan Lake above U.S. Hwy 64 at Wilsonville, NC	4/21/2005	ns	ns	ns	ns	ns
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	4/20/2004	<0.036	E0.007	<0.01	0.028	<0.01
7	Jordan Lake at Bells Landing near Griffins Crossroads, NC	8/24/2004	<0.036	0.020	0.042	0.006	E0.003
8	Neuse River above U.S. 70 at Smithfield, NC	10/24/2002	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	11/13/2002	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	12/12/2002	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	1/16/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	1/31/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	2/11/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	3/20/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	4/9/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	5/7/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	6/4/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	7/22/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	8/21/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	9/9/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	10/6/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	11/4/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	12/23/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	12/30/2003	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	6/22/2004	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	8/31/2004	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	10/12/2004	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	10/18/2004	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	12/29/2004	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	1/28/2005	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	2/2/2005	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	3/10/2005	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	4/14/2005	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	5/31/2005	ns	ns	ns	ns	ns
8	Neuse River above U.S. 70 at Smithfield, NC	7/12/2005	ns	ns	ns	ns	ns
Minimum detected			E0.005	E0.007	0.042	0.006	E0.003
Median detected			0.012	E0.010	0.049	0.028	E0.003
Maximum detected			0.037	0.020	0.055	0.033	E0.003
Number of detections			4	6	2	3	1

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