

Occurrence of Pharmaceuticals and Other Organic Wastewater Constituents in Selected Streams in Northern Arkansas, 2004



Prepared in cooperation with the
UNIVERSITY OF ARKANSAS and the
U.S. DEPARTMENT OF AGRICULTURE, AGRICULTURAL RESEARCH SERVICE

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

Front Cover: Photograph of water-quality sampling site on Mud Creek at Township Road, Fayetteville, Arkansas. Photograph by Joel M. Galloway, U.S. Geological Survey.

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By Joel M. Galloway, U.S. Geological Survey; Brian E. Haggard, U.S. Department of Agriculture, Agricultural Research Service; Michael T. Meyers, and W. Reed Green, U.S. Geological Survey

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U.S. Department of the Interior
Gale A. Norton, Secretary

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P. Patrick Leahy, Acting Director

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Conversion Factors and Datum

Multiply	By	To obtain
Length		
mile (mi)	1.609	kilometer (km)
Flow rate		
cubic foot per second (ft ³ /s)	0.02832	cubic meter per second (m ³ /s)

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

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

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

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

Occurrence of Pharmaceuticals and Other Organic Wastewater Constituents in Selected Streams in Northern Arkansas, 2004

By Joel M. Galloway¹, Brian E. Haggard², Michael T. Meyers¹, and W. Reed Green¹

Abstract

The U.S. Geological Survey, in cooperation with the University of Arkansas and the U.S. Department of Agriculture, Agricultural Research Service, collected data in 2004 to determine the occurrence of pharmaceuticals and other organic wastewater constituents, including many constituents of emerging environmental concern, in selected streams in northern Arkansas. Samples were collected in March and April 2004 from 17 sites located upstream and downstream from wastewater-treatment plant effluent discharges on 7 streams in northwestern Arkansas and at 1 stream site in a relatively undeveloped basin in north-central Arkansas. Additional samples were collected at three of the sites in August 2004. The targeted organic wastewater constituents and sample sites were selected because wastewater-treatment plant effluent discharge provides a potential point source of these constituents and analytical techniques have improved to accurately measure small amounts of these constituents in environmental samples.

At least 1 of the 108 pharmaceutical or other organic wastewater constituents was detected at all sites in 2004, except at Spavinaw Creek near Maysville, Arkansas. The number of detections generally was greater at sites downstream from municipal wastewater-treatment plant effluent discharges (mean = 14) compared to sites not influenced by wastewater-treatment plants (mean = 3). Overall, 42 of the 108 constituents targeted in the collected water-quality samples were detected. The most frequently detected constituents included caffeine, phenol, *para*-cresol, and acetyl hexamethyl tetrahydro naphthalene.

Introduction

Northwestern Arkansas is one of the fastest growing urban areas in the United States while at the same time it is one of the most productive poultry areas in the Nation. As a result, the water quality of northwestern Arkansas streams has become the

focus of environmental concern. Public and government concern about point and nonpoint source contributions of nutrients and other constituents to surface and ground waters has elevated to a point where legislation is being passed to establish numeric stream nutrient criteria, and lawsuits have been filed against the poultry industry and municipal wastewater-treatment plant (WWTP) effluent dischargers. Water-quality concerns in northwestern Arkansas mainly relate to nutrient enrichment and sediment. For example, the U.S. Environmental Protection Agency (USEPA) recently added 27 streams in northwestern Arkansas to the list of impaired water bodies because of increased nutrient and sediment concentrations (Arkansas Department of Environmental Quality, 2005). However, nationwide attention also has focused on the occurrence of organic wastewater constituents (OWCs) in surface and ground water. Kolpin and others (2002) conducted a nationwide reconnaissance of 95 antibiotics, pharmaceuticals, hormones, and other OWCs in streams and detected 82 constituents measured at numerous sites. OWCs were found in 80 percent of the streams sampled. However, little is known about the occurrence, extent, transport, and fate of many synthetic organic chemicals after their intended use, particularly hormonally active chemicals and antibiotics. Until recently, there have been few analytical methods capable of detecting these constituents at low concentrations that might be present in the environment. The development of these analytical methods has resulted in several new studies addressing the occurrence, fate, and transport of OWCs in the aquatic environment (Andreozzi and others, 2004; Barnes and others, 2004; Kolpin and others, 2004). Potential concerns related to the environmental presence of these constituents include abnormal physiological processes and reproductive impairment, increased incidences of cancer, the development of antibiotic-resistant bacteria and plasmid transfer, and the potential increased toxicity and carcinogenic activity of these chemicals and chemical mixtures.

The U.S. Geological Survey (USGS) in cooperation with the University of Arkansas and the U.S. Department of Agriculture, Agricultural Research Service collected data in 2004 to determine the occurrence and extent of pharmaceuticals and other OWCs in streams in northern Arkansas, especially those receiving WWTP effluent discharge. These constituents potentially are associated with human, industrial, and agricultural wastewaters and the use of prescription and nonprescription

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drugs, steroids, reproductive hormones, and personal care products. Some of the constituents are found in poultry manure, which is often applied to pastures as an organic fertilizer in northern Arkansas.

Purpose and Scope

The purpose of this report is to describe the occurrence of pharmaceuticals and other OWC's, including many constituents of emerging environmental concern, in selected streams in northern Arkansas. This report presents the occurrence of 108 OWCs and is focused on sites upstream and downstream from WWTP effluent discharges in the selected streams. Samples were collected in March and April 2004 from 17 sites located upstream and downstream from WWTP effluent discharges on 7 streams in northwestern Arkansas and at 1 stream site in a relatively undeveloped basin in north-central Arkansas. Samples also were collected again in August 2004 at three sites on one stream in northwestern Arkansas. The targeted OWCs and sample sites were selected because WWTP effluent discharge provides a potential point source of these constituents, and analytical techniques have improved to accurately measure small amounts of these constituents in environmental samples.

Description of Study Areas

Samples were collected from sites within two study areas in northern Arkansas (fig. 1; table 1). One study area includes streams in Benton and Washington Counties in northwestern Arkansas. The other study area is the North Sylamore Creek Basin in north-central Arkansas.

Northwestern Arkansas is one of the more densely populated areas of Arkansas. It also is one of the fastest growing urban areas in the United States (U.S. Census Bureau, 2005). The largest cities in the area are Fayetteville (62,078 people), Springdale (52,471 people), Rogers (42,795 people), and Bentonville (26,397 people) (U.S. Census Bureau, 2005).

Land use in the northwestern Arkansas study area generally consists of a mixture of urban, pasture, and forest. For example, the Illinois River Basin upstream from the Arkansas-Oklahoma State line, which includes Mud Creek, Spring Creek, and Osage Creek, is approximately 7 percent urban land, 29 percent forested, and 63 percent agricultural (mostly pasture) land. The Spavinaw Creek Basin upstream from the Arkansas-Oklahoma State line drains a pasture dominated watershed with a high density of poultry farms. Arkansas is the second largest producer of poultry in the Nation and Benton and Washington Counties produce approximately 20 percent of the poultry in the State (U.S. Department of Agriculture, 2005).

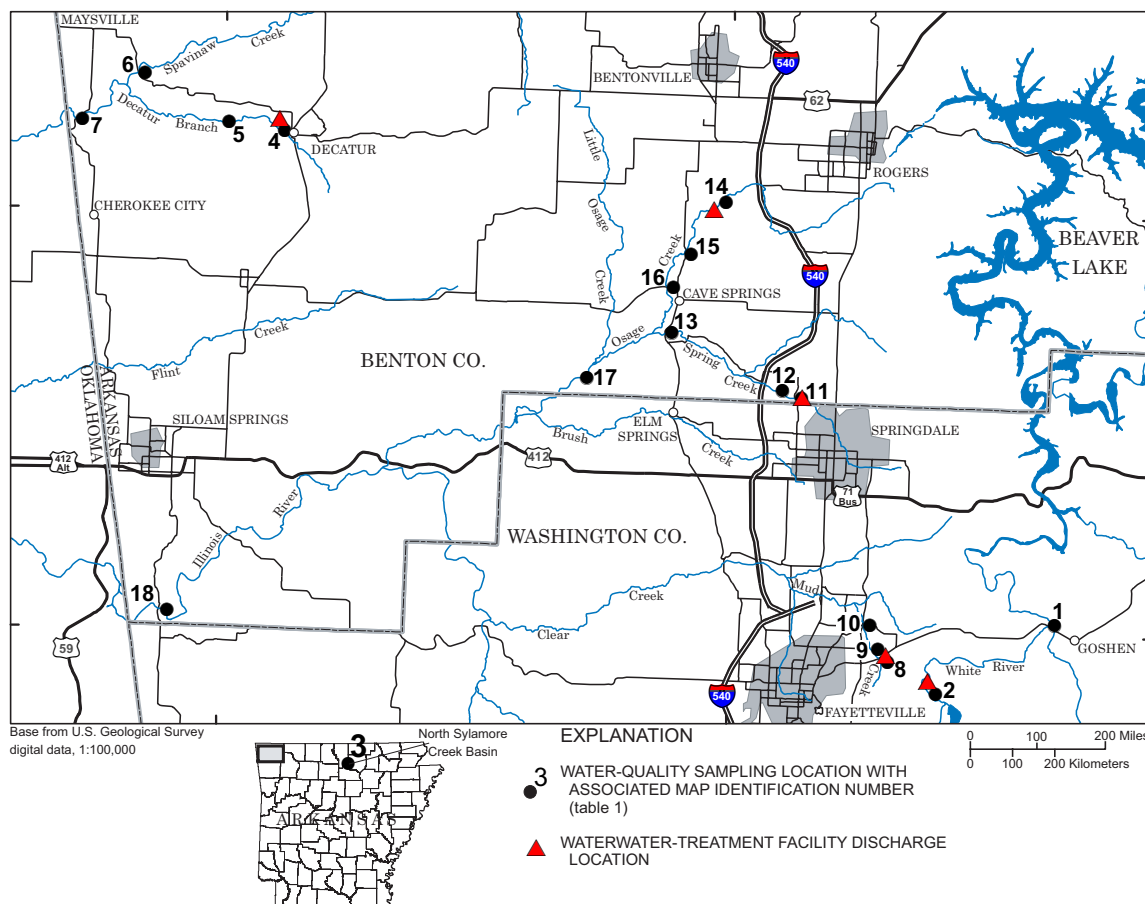


Figure 1. Study areas with water-quality sampling site locations.

Table 1. Stream sites sampled for pharmaceuticals and other organic wastewater constituents in northern Arkansas, 2004.

[WWTP, wastewater-treatment plant]

Map identification number (fig. 1)	Station identification number	Station name	Relation to wastewater-treatment plant discharges
1	07048600	White River near Fayetteville	Upstream from city of Fayetteville WWTP discharge
2	07048700	White River near Goshen	Downstream from city of Fayetteville WWTP discharge
3	07060710	North Sylamore Creek near Fifty-Six	U.S. Geological Survey Hydrologic Benchmark Network site, representative of an undeveloped basin
4	362016094280500	Decatur Branch at Decatur	Upstream from city of Decatur WWTP discharge
5	362030094300400	Decatur Branch near Maysville	Downstream from city of Decatur WWTP discharge
6	07191160	Spavinaw Creek near Maysville	Upstream from city of Decatur WWTP discharge
7	07191179	Spavinaw Creek near Cherokee City	Downstream from city of Decatur WWTP discharge
8	360516094063400	Mud Creek south of Highway 45 at Fayetteville	Upstream from city of Fayetteville WWTP discharge
9	360538094065500	Mud Creek at Township Road at Fayetteville	Downstream from city of Fayetteville WWTP discharge
10	360619094071200	Mud Creek at Old Wire Road at Fayetteville	Downstream from city of Fayetteville WWTP discharge
11	361248094094200	Spring Creek at Silent Grove Road near Springdale	Upstream from city of Springdale WWTP discharge
12	361301094102400	Spring Creek at North 40th Street near Springdale	Downstream from city of Springdale WWTP discharge
13	361438094141900	Spring Creek at Highway 112 near Cave Springs	Downstream from city of Springdale WWTP discharge
14	361823094122700	Osage Creek near County Road 51 near Rogers	Upstream from city of Rogers WWTP discharge
15	07194880	Osage Creek near Cave Springs	Downstream from city of Rogers WWTP discharge
16	361556094141600	Osage Creek at Highway 264 at Cave Springs	Downstream from city of Rogers WWTP discharge
17	07195000	Osage Creek near Elm Springs	Downstream from city of Rogers WWTP discharge
18	07195430	Illinois River south of Siloam Springs	Downstream from numerous WWTP discharges in the basin

Land use in the North Sylamore Creek Basin (58.1 square miles) is nearly 99 percent forested. A USGS streamflow and water-quality station on North Sylamore Creek near Fifty-Six is part of the USGS Hydrologic Benchmark Network, which includes sites across the Nation where long-term measurements of streamflow and water-quality data are collected in areas that are minimally affected by human activities.

Methods

Water-quality samples were collected at 17 stream sites in northwestern Arkansas, and at 1 stream site in north-central Arkansas in March and April 2004. A second set of samples also were collected in August 2004 at the three sites on Mud Creek (sites 8, 9, and 10; fig. 1). Samples were collected upstream and downstream from the effluent discharges from the city of Fayetteville WWTP (Mud Creek and the White River), the city of Springdale WWTP (Spring Creek), the city of Rogers WWTP (Osage Creek), and the city of Decatur WWTP (Deca-

tur Branch and Spavinaw Creek) (fig. 1 and table 1). Sample sites were selected at various distances downstream from the WWTPs, including the Illinois River south of Siloam Springs, Arkansas (site 18), which is downstream from WWTPs at the cities of Fayetteville, Rogers, and Springdale. North Sylamore Creek near Fifty-Six (site 3) in north-central Arkansas also was sampled to determine the occurrence of OWC's at a site draining a relatively undeveloped basin.

Samples were collected and processed using protocols described in Wilde and others (1998a, 1998b, 1998c, 1999a, 1999b). Water-quality samples were collected from a single vertical point in the stream because of well-mixed conditions, low velocities, and small cross-sectional areas at the streams. Samples were filtered at each site with a 0.7-micron pore size, baked glass-fiber filter and shipped to the laboratories in amber baked-glass bottles, chilled to 4 degrees Celsius. Physical properties (water temperature, pH, dissolved oxygen, and specific conductance) also were measured at each site using protocols described in Wilde and Radke (1998). Streamflow measurements were made at each site using an acoustic Doppler current profiler and methods described in Rantz and others (1982). To

minimize contamination of samples, use of personal care items, caffeinated products, pharmaceuticals, and tobacco were minimized during sample collection and processing.

One replicate and one blank sample were collected during March and April 2004 for quality control and assurance. The replicate sample was collected concurrently with the routine sample at Spavinaw Creek near Cherokee City, Arkansas (site 7). The blank sample was processed with laboratory-grade organic-free blank water at Mud Creek south of Highway 45 at Fayetteville, Arkansas (site 8) by using the same field methods and equipment used to collect the environmental samples at each of the stream sites. A blank sample also was collected at the same site in August 2004.

Samples were analyzed for antibiotics and antibiotic residuals by the USGS Organic Chemistry Research Group Laboratory in Lawrence, Kansas (table 2). Samples were analyzed for five classes of antibiotics (beta-lactams, macrolides, quinolones, sulfonamides, and tetracyclines) using two on-line solid-phase extraction methods and liquid chromatography/mass spectrometry (LC/MS). Samples were extracted for tetracyclines using a Spark-Holland glyphosate prosept cartridge and the quinolone, sulfonamide, beta-lactam, and macrolide antibiotics were extracted using Waters HLB prosept cartridges.

Samples for other pharmaceuticals and OWCs, excluding antibiotics, were analyzed by the USGS National Water Quality Laboratory in Lakewood, Colorado (table 2). The water samples were extracted at the laboratory by vacuum through disposable solid-phase extraction (SPE) cartridges that contain poly-

styrene-divinylbenzene resin. Cartridges were dried with nitrogen gas and then sorbed constituents were eluted with dichloromethane-diethyl ether. Constituent concentrations were determined by capillary-column gas chromatography/mass spectrometry (GC/MS) (Zaugg and others, 2002).

Occurrence of Pharmaceuticals and Other Organic Wastewater Constituents

At least one pharmaceutical or other OWC was detected at all sites in 2004, except at Spavinaw Creek near Maysville, Arkansas (site 6; fig. 2 and table 3). The number of detections at sites downstream from WWTP effluent discharges generally was greater than at sites not influenced by WWTP effluent discharges. The mean number of detections at sites upstream from WWTP effluent discharges was three (including the site at North Sylamore Creek, site 3, which is considered relatively undeveloped) for samples collected in March and April 2004. The mean number of detections at sites downstream from WWTP discharges was more than four times higher (14 detections). The greatest number of detections was found at Mud Creek (sites 9 and 10), Spring Creek (sites 12 and 13), and Osage Creek (sites 15, 16, and 17) at sites downstream from the cities of Fayetteville, Springdale, and Rogers WWTP effluent discharges, respectively.

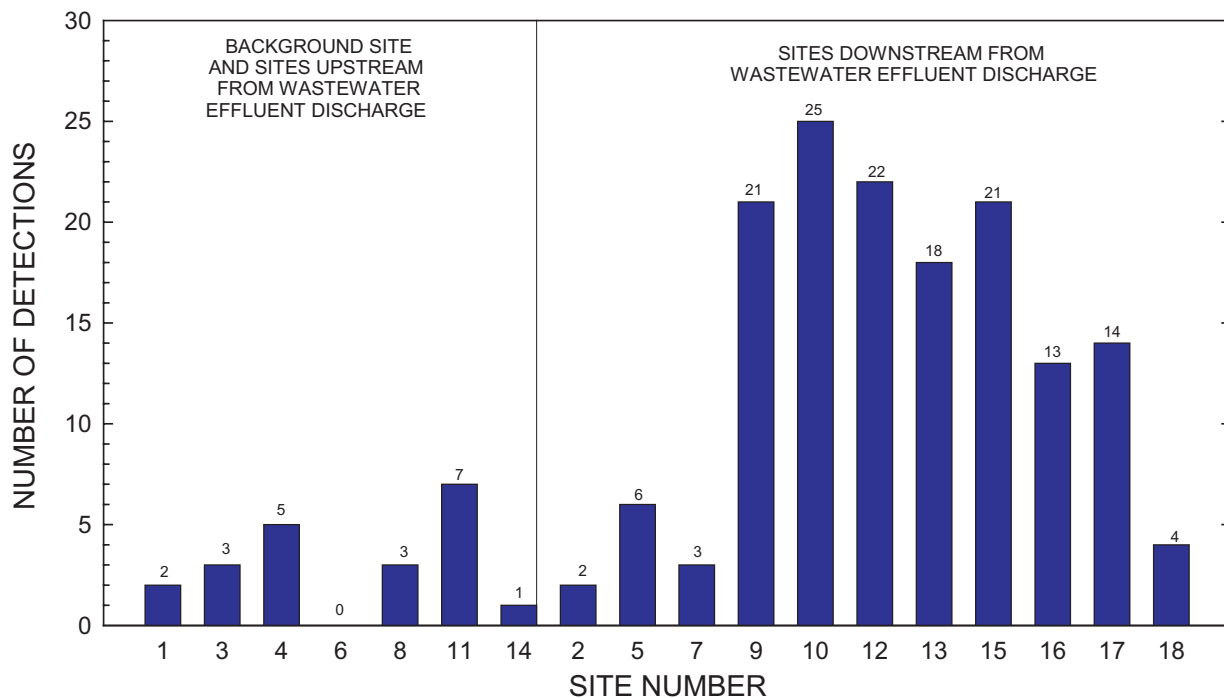


Figure 2. Summary of pharmaceuticals and other organic wastewater constituents detected from selected sites in northern Arkansas, March and April 2004.

Table 2. Pharmaceuticals and other organic wastewater constituents targeted by chemical analyses of water samples collected at selected sites in northern Arkansas, 2004.

[Information from Zaugg and others, 2002; *, reporting level for samples collected August 2004; µg/L, micrograms per liter; PAH, polycyclic aromatic hydrocarbon]

Constituent name	Laboratory reporting level (µg/L)	Constituent group	Uses or sources
1,4-Dichlorobenzene	0.5	Disinfectant	Moth repellent, fumigant, deodorant
1-Methylnaphthalene	0.5	Fuel	2-5 percent gasoline, diesel fuel, or crude oil
2,6-Dimethylnaphthalene	0.5	Fuel	Present in diesel and kerosene (trace in gasoline)
2-Methylnaphthalene	0.5	Fuel	2-5 percent gasoline, diesel fuel, or crude oil
3- <i>beta</i> -Coprostanol	2.0	Steroid	Carnivore fecal indicator
3-Methyl-1H-indole (skatol)	1.0	Fragrance/flower	Fragrance, stench in feces and coal tar
3- <i>tert</i> -Butyl-4-hydroxyanisole (BHA)	5.0	Antioxidant	Antioxidant, general preservative
4-Cumylphenol	1.0	Detergent metabolite	Nionic detergent metabolite
4- <i>n</i> -Octylphenol	1.0	Detergent metabolite	Nionic detergent metabolite
<i>para</i> -Nonylphenol (total)	0.5	Detergent metabolite	Nionic detergent metabolite
4- <i>tert</i> -Octylphenol	0.5	Detergent metabolite	Nionic detergent metabolite
5-Methyl-1H-benzotriazole	2.0	Antioxidant	Antioxidant in antifreeze and deicers
Anthraquinone	0.5	PAH	Manufacturing dye and textiles, seed treatment, bird repellent
Acetophenone	0.5	Fragrance/flower	Fragrance in detergent and tobacco, flavor in beverages
Acetyl hexamethyl tetrahydro naphthalene (AHTN)	0.5	Fragrance/flower	Musk fragrance (widespread usage)
Anthracene	0.5	PAH	Wood preservative, component of tar, diesel or crude oil, combustion product
Benzo[a]pyrene	0.5	PAH	Regulated polycyclic aromatic hydrocarbon, used in cancer research, combustion product
Benzophenone	0.5	Fragrance/flower	Fixative for perfumes and soap
<i>beta</i> -Sitosterol	2.0	Steroid	Plant sterol
<i>beta</i> -Stigmastanol	2.0	Steroid	Plant sterol
Bisphenol A	1.0	Plasticizer	Manufacturing polycarbonate resins, antioxidant, flame retardant
Bromacil	0.5	Insect repellent/ pesticide	Herbicide (general use pesticide), 80 percent noncrop usage on grass or brush
Caffeine	0.5	Nonprescription drug	Beverages, diuretic, very mobile and biodegradable
Camphor	0.5	Fragrance/flower	Flavor, odorant, ointments
Carbaryl	1.0	Insect repellent/ pesticide	Insecticide, crop and garden uses, low persistence
Carbazole	0.5	Insect repellent/ pesticide	Insecticide, manufacturing dyes, explosives, and lubricants

Table 2. Pharmaceuticals and other organic wastewater constituents targeted by chemical analyses of water samples collected at selected sites in northern Arkansas, 2004.—Continued

[Information from Zaugg and others, 2002; *, reporting level for samples collected August 2004; µg/L, micrograms per liter; PAH, polycyclic aromatic hydrocarbon]

Constituent name	Laboratory reporting level (µg/L)	Constituent group	Uses or sources
Chlorpyrifos	0.5	Insect repellent/ pesticide	Insecticide, domestic pest and termite control (domestic use restricted as of 2001)
Cholesterol	2.0	Steroid	Often a fecal indicator, also a plant sterol
Cotinine	0.5	Nonprescription drug	Primary nicotine metabolite
N,N-diethyl- <i>meta</i> -toluamide (DEET)	0.5	Insect repellent/ pesticide	Insecticide, urban uses, mosquito repellent
Diazinon	0.5	Insect repellent/ pesticide	Insecticide, greater than 40 percent nonagricultural usage, ants, flies
Nonylphenol, diethoxy-(total, NPEO2)	5.0	Detergent metabolite	Nionic detergent metabolite
Octylphenol, diethoxy-(OPEO2)	1.0	Detergent metabolite	Nionic detergent metabolite
<i>d</i> -Limonene	0.5	Fragrance/ flavor	Fungicide, antimicrobial, antiviral, fragrance in aerosols
Octylphenol, monoethoxy -(OPEO1)	1.0	Detergent metabolite	Nionic detergent metabolite
Fluoranthene	0.5	PAH	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), combustion product
Hexahydrohexamethyl cyclopentabenzopyran (HHCB)	0.5	Fragrance/ flavor	Musk fragrance (widespread usage)
Indole	0.5	Fragrance/ flavor	Pesticide inert ingredient, fragrance in coffee
Isoborneol	0.5	Fragrance/ flavor	Fragrance in perfume, in disinfectants
Isophorone	0.5	Solvent	Solvent for lacquer, plastic, oil, silicon, resin
Isopropylbenzene (cumene)	0.5	Fuel	Manufacturing phenol and acetone, fuels and paint thinner
Isoquinoline	0.5	Fragrance/ flavor	Flavors and fragrances
Menthol	0.5	Fragrance/ flavor	Cigarettes, cough drops, liniment, mouthwash
Metalaxyl	0.5	Insect repellent/ pesticide	Herbicide, fungicide (general use pesticide), mildew, blight, pathogens, golf and turf
Methyl salicylate	0.5	Liniment	Liniment, food, beverage, ultraviolet-absorbing lotion
Metolachlor	0.5	Insect repellent/ pesticide	Herbicide (general use pesticide), indicator of agricultural drainage
Naphthalene	0.5	PAH	Fumigant, moth repellent, major component (about 10 percent)
<i>para</i> -Cresol	1.0	Disinfectant	Wood preservative
Pentachlorophenol	2.0	Insect repellent/ pesticide	Herbicide, fungicide, wood preservative, termite control
Phenanthrene	0.5	PAH	Manufacturing explosives, component of tar, diesel fuel, or crude oil, combustion product
Phenol	0.5	Disinfectant	Disinfectant, manufacturing of several products, leachate
Prometon	0.5	Insect repellent/ pesticide	Herbicide (noncrop only), applied prior to blacktop

Table 2. Pharmaceuticals and other organic wastewater constituents targeted by chemical analyses of water samples collected at selected sites in northern Arkansas, 2004.—Continued

[Information from Zaugg and others, 2002; *, reporting level for samples collected August 2004; µg/L, micrograms per liter; PAH, polycyclic aromatic hydrocarbon]

Constituent name	Laboratory reporting level (µg/L)	Constituent group	Uses or sources
Pyrene	0.5	PAH	Component of tar and asphalt (only traces in gasoline or diesel fuel), combustion product
Tetrachloroethylene	0.5	Solvent	Solvent, degreaser, veterinary anthelmintic
Bromoform	0.5	Disinfectant	Wastewater ozonation byproduct, military explosives
Tributyl phosphate	0.5	Fire retardant	Antifoaming agent, flame retardant
Triclosan	1.0	Disinfectant	Disinfectant, antimicrobial (concern for acquired microbial resistance)
Triethyl citrate (ethyl citrate)	0.5	Plasticizer	Cosmetics, pharmaceuticals
Triphenyl phosphate	0.5	Plasticizer	Plasticizer, resin, wax, finish, roofing paper, flame retardant
Tri(2-butoxyethyl) phosphate	0.5	Fire retardant	Flame retardant
Tri(2-chloroethyl) phosphate	0.5	Fire retardant	Plasticizer, flame retardant
Tri(dichloroisopropyl) phosphate	0.5	Fire retardant	Flame retardant
Dichlorvos	1.0	Insect repellent/ pesticide	Insecticide, pet collars, flies, also a degradate of naled or trichlofon
Amoxicillin	0.20, 0.01*	Antibiotic	Antibiotic
Ampicillin	0.10, 0.01*	Antibiotic	Antibiotic
Cefotaxime	0.10, 0.01*	Antibiotic	Antibiotic
Cloxacillin	0.10, 0.01*	Antibiotic	Antibiotic
Oxacillin	0.10, 0.01*	Antibiotic	Antibiotic
Penicillin G	0.10, 0.01*	Antibiotic	Antibiotic
Penicillin V	0.10, 0.01*	Antibiotic	Antibiotic
Erythromycin	0.10, 0.01*	Antibiotic	Antibiotic
Anhydro-erythromycin	0.05, 0.005*	Antibiotic	Antibiotic degradation byproduct
Lincomycin	0.05, 0.005*	Antibiotic	Antibiotic
Ormetoprim	0.05, 0.005*	Antibiotic	Antibiotic
Roxithromycin	0.05, 0.005*	Antibiotic	Antibiotic
Trimethoprim	0.05, 0.005*	Antibiotic	Antibiotic
Tylosin	0.05, 0.005*	Antibiotic	Antibiotic
Virginiamycin	0.05, 0.005*	Antibiotic	Antibiotic
Ciprofloxacin	0.05, 0.005*	Antibiotic	Antibiotic
Clinafloxacin	0.05, 0.005*	Antibiotic	Antibiotic

Table 2. Pharmaceuticals and other organic wastewater constituents targeted by chemical analyses of water samples collected at selected sites in northern Arkansas, 2004.—Continued

[Information from Zaugg and others, 2002; *, reporting level for samples collected August 2004; µg/L, micrograms per liter; PAH, polycyclic aromatic hydrocarbon]

Constituent name	Laboratory reporting level (µg/L)	Constituent group	Uses or sources
Flumequine	0.05,0.005*	Antibiotic	Antibiotic
Lomefloxacin	0.05,0.005*	Antibiotic	Antibiotic
Norfloxacin	0.05,0.005*	Antibiotic	Antibiotic
Ofloxacin	0.05,0.005*	Antibiotic	Antibiotic
Oxolinic acid	0.05,0.005*	Antibiotic	Antibiotic
Sarfloxacin	0.05,0.005*	Antibiotic	Antibiotic
Sulfachloropyridazine	0.05,0.005*	Antibiotic	Antibiotic
Sulfadiazine	0.05,0.005*	Antibiotic	Antibiotic
Sulfadimethoxine	0.05,0.005*	Antibiotic	Antibiotic
Sulfamerazine	0.05,0.005*	Antibiotic	Antibiotic
Sulfamethazine	0.05,0.005*	Antibiotic	Antibiotic
Sulfamethoxazole	0.05,0.005*	Antibiotic	Antibiotic
Sulfathiazole	0.05,0.005*	Antibiotic	Antibiotic
Chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic
Anhydro-chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Epi-anhydro-chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Epi-chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Iso-chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Iso-epi-chlorotetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Demeclocycline	0.10, 0.01*	Antibiotic	Antibiotic
Doxycycline	0.10, 0.01*	Antibiotic	Antibiotic
Minocycline	0.10, 0.01*	Antibiotic	Antibiotic
Oxytetracycline	0.10, 0.01*	Antibiotic	Antibiotic
Epi-oxytetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Tetracycline	0.10, 0.01*	Antibiotic	Antibiotic
Anhydro-tetracycline	0.10, 0.01*	Antibiotic	Antibiotic degradation byproduct
Epi-anhydro-tetracycline	0.10	Antibiotic	Antibiotic degradation byproduct
Epi-tetracycline	0.10	Antibiotic	Antibiotic degradation byproduct

Table 3. Summary of pharmaceuticals and other organic wastewater constituents detected at selected sites in northern Arkansas, 2004.—Continued

[Values are in micrograms per liter; <, less than; E, estimated value below laboratory reporting limit, but greater than the detection limit; *, replicate sample; bold, detected concentration; number in parenthesis is map identification number (fig. 1); shaded indicates site is downstream from wastewater-treatment plant effluent discharge; --, not reported]

Constituent name	Sampling location (sample collection date)						
	(7) Spavinaw Creek near Cherokee City (4/06/04)*	(8) Mud Creek south of Hwy 45 at Fayetteville (3/30/04)	(8) Mud Creek south of Hwy 45 at Fayetteville (8/16/04)	(9) Mud Creek at Township Road at Fayetteville (3/30/04)	(9) Mud Creek at Township Road at Fayetteville (8/16/04)	(10) Mud Creek at Old Wire Road at Fayetteville (3/30/04)	(10) Mud Creek at Old Wire Road at Fayetteville (8/16/04)
Tributyl phosphate	<.50	<.50	<.50	E.29	.56	E.23	E.46
Triclosan	<1.0	<1.0	<1.0	E.13	E.25	E.12	E.20
Triethyl citrate (ethyl citrate)	<.50	<.50	<.50	E.26	E.26	E.22	E.24
Triphenyl phosphate	<.50	<.50	<.50	E.057	E.063	E.034	E.051
Tri(2-butoxyethyl) phosphate	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Tri(2-chloroethyl) phosphate	<.50	<.50	<.50	E.42	.69	E.34	.70
Tri(dichloroisopropyl) phosphate	<.50	<.50	<.50	E.20	E.40	E.20	E.40
Dichlorvos	<1.0	<1.0	--	<1.0	--	<1.0	--
Amoxicillin	<.20	<.20	<.01	<.20	<.01	<.20	<.01
Ampicillin	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Cefotaxime	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Cloxacillin	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Oxacillin	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Penicillin G	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Penicillin V	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Erythromycin	<.10	<.10	<.005	<.10	.175	<.10	.154
Anhydro-erythromycin	<.05	<.05	<.005	.20	1.21	.14	.962
Lincomycin	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Ormetoprim	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Roxithromycin	<.10	<.10	<.005	<.10	<.005	<.10	<.005
Trimethoprim	<.05	<.05	<.005	.19	.058	.15	.045
Tylosin	<.10	<.10	<.005	<.10	.012	<.10	.008
Virginiamycin	<.10	<.10	<.005	<.10	<.005	<.10	<.005
Ciprofloxacin	<.05	<.05	<.005	<.05	.039	<.05	.027
Clinafloxacin	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Flumequine	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Lomefloxacin	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Norfloxacin	<.05	<.05	<.005	<.05	<.005	<.05	<.005

Table 3. Summary of pharmaceuticals and other organic wastewater constituents detected at selected sites in northern Arkansas, 2004.—Continued

[Values are in micrograms per liter; <, less than; E, estimated value below laboratory reporting limit, but greater than the detection limit; *, replicate sample; bold, detected concentration; number in parenthesis is map identification number (fig. 1); shaded indicates site is downstream from wastewater-treatment plant effluent discharge; --, not reported]

Constituent name	Sampling location (sample collection date)						
	(7) Spavinaw Creek near Cherokee City (4/06/04)*	(8) Mud Creek south of Hwy 45 at Fayetteville (3/30/04)	(8) Mud Creek south of Hwy 45 at Fayetteville (8/16/04)	(9) Mud Creek at Township Road at Fayetteville (3/30/04)	(9) Mud Creek at Township Road at Fayetteville (8/16/04)	(10) Mud Creek at Old Wire Road at Fayetteville (3/30/04)	(10) Mud Creek at Old Wire Road at Fayetteville (8/16/04)
Ofloxacin	<.05	<.05	<.005	.10	.109	.10	.094
Oxolinic acid	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sarfloxacin	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sulfachloropyridazine	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sulfadiazine	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sulfadimethoxine	<.05	<.05	<.005	<.05	E.003	<.05	E.004
Sulfamerazine	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sulfamethazine	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Sulfamethoxazole	<.05	<.05	<.005	.500	.196	.42	.302
Sulfathiazole	<.05	<.05	<.005	<.05	<.005	<.05	<.005
Chlorotetracycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Anhydro-chlorotetracycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Epi-anhydro-chlorotetracycline	<.10	<.10	--	<.10	--	<.10	--
Epi-chlorotetracycline	<.10	<.10	--	<.10	--	<.10	--
Iso-chlorotetracycline	<.10	<.10	--	<.10	--	<.10	--
Iso-epi-chlorotetracycline	<.10	<.10	--	<.10	--	<.10	--
Demeclocycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Doxycycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Minocycline	<.20	<.20	<.01	<.20	<.01	<.20	<.01
Oxytetracycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Epi-oxytetracycline	<.10	<.10	--	<.10	--	<.10	--
Tetracycline	<.10	<.10	<.01	<.10	<.01	<.10	<.01
Anhydro-tetracycline	<.20	<.20	<.01	<.20	<.01	<.20	<.01
Epi-anhydro-tetracycline	<.10	<.10	--	<.10	--	<.10	--
Epi-tetracycline	<.10	<.10	--	<.10	--	<.10	--

Table 3. Summary of pharmaceuticals and other organic wastewater constituents detected at selected sites in northern Arkansas, 2004.—Continued

[Values are in micrograms per liter; <, less than; E, estimated value below laboratory reporting limit, but greater than the detection limit; *, replicate sample; bold, detected concentration; number in parenthesis is map identification number (fig. 1); shaded indicates site is downstream from wastewater-treatment plant effluent discharge; --, not reported]

Constituent name	Sampling location (sample collection date)							
	(11) Spring Creek at Silent Grove Road near Springdale (3/30/04)	(12) Spring Creek at North 40th St near Springdale (3/30/04)	(13) Spring Creek at Hwy 112 near Cave Springs (3/30/04)	(14) Osage Creek near Road 51 near Rogers (3/31/04)	(15) Osage Creek near Cave Springs (3/31/04)	(16) Osage Creek at Hwy 264 at Cave Springs (3/31/04)	(17) Osage Creek near Elm Springs (3/31/04)	(18) Illinois River south of Siloam Springs (4/05/04)
1,4-Dichlorobenzene	<.50	E.098	<.50	<.50	<.50	<.50	<.50	<.50
1-Methylnaphthalene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
2,6-Dimethylnaphthalene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
2-Methylnaphthalene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
3- <i>beta</i> -Coprostanol	<2.0	E1.5	E.81	<2.0	E.82	<2.0	<2.0	<2.0
3-Methyl-1H-indole (skatol)	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
3- <i>tert</i> -Butyl-4-hydroxyanisole (BHA)	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
4-Cumylphenol	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
4- <i>n</i> -Octylphenol	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
<i>para</i> -Nonylphenol (total)	<5.0	E1.2	<5.0	<5.0	E.68	E.51	E.64	<5.0
4- <i>tert</i> -Octylphenol	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
5-Methyl-1H-benzotriazole	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Anthraquinone	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Acetophenone	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Acetyl hexamethyl tetrahydro naphthalene (AHTN)	E.011	1.40	E.480	<.5	.50	E.330	E.220	E.030
Anthracene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Benzo[a]pyrene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Benzophenone	<.50	E.120	E.067	<.50	E.043	E.038	E.031	<.50
<i>beta</i> -Sitosterol	<2.0	<2.0	<2.0	<2.0	E.97	<2.0	<2.0	<2.0
<i>beta</i> -Stigmastanol	<2.0	E1.6	<2.0	<2.0	E.95	<2.0	<2.0	<2.0
Bisphenol A	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Bromacil	.65	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Caffeine	E.032	E.067	E.041	E.035	E.032	E.047	E.043	E.037
Camphor	<.50	<.50	<.50	<.50	<.50	<.50	<.500	<.5
Carbaryl	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Carbazole	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Chlorpyrifos	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Cholesterol	<2.0	E1.60	E1.00	<2.0	E.950	<2.0	<2.0	<2.0

18 Occurrence of Pharmaceuticals and Other Organic Wastewater Constituents in Selected Streams in Northern Arkansas, 2004

Table 3. Summary of pharmaceuticals and other organic wastewater constituents detected at selected sites in northern Arkansas, 2004.—Continued

[Values are in micrograms per liter; <, less than; E, estimated value below laboratory reporting limit, but greater than the detection limit; *, replicate sample; bold, detected concentration; number in parenthesis is map identification number (fig. 1); shaded indicates site is downstream from wastewater-treatment plant effluent discharge; --, not reported]

Constituent name	Sampling location (sample collection date)							
	(11) Spring Creek at Silent Grove Road near Springdale (3/30/04)	(12) Spring Creek at North 40th St near Springdale (3/30/04)	(13) Spring Creek at Hwy 112 near Cave Springs (3/30/04)	(14) Osage Creek near Road 51 near Rogers (3/31/04)	(15) Osage Creek near Cave Springs (3/31/04)	(16) Osage Creek at Hwy 264 at Cave Springs (3/31/04)	(17) Osage Creek near Elm Springs (3/31/04)	(18) Illinois River south of Siloam Springs (4/05/04)
Cotinine	<1.0	<1.0	E.036	<1.0	<1.0	<1.0	<1.0	<1.0
N,N-diethyl-meta-toluamide (DEET)	<.50	<.50	E.020	<.50	E.042	E.028	E.019	<.50
Diazinon	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Nonylphenol, diethoxy-(total, NPEO2)	<5.0	E7.4	E4.1	<5.0	E3.6	<5.0	<5.0	<5.0
Octylphenol, diethoxy-(OPEO2)	<1.0	E.33	E.20	<1.0	E.13	<1.0	<1.0	<1.0
<i>d</i> -Limonene	<.50	E.029	<.50	<.50	<.50	<.50	<.50	<.50
Octylphenol, monoethoxy -(OPEO1)	<1.0	<1.0	<1.0	<1.0	E.46	<1.0	<1.0	<1.0
Fluoranthene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Hexahydrohexamethyl cyclopentabenzopyran (HHCB)	<.50	E.240	E.078	<.50	E.083	E.050	E.026	<.50
Indole	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Isoborneol	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Isophorone	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Isopropylbenzene (cumene)	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Isoquinoline	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Menthol	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Metalaxyl	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Methyl salicylate	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Metolachlor	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Naphthalene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
<i>para</i> -Cresol	E.031	E.100	E.054	<1.0	E.048	E.093	E.049	<1.0
Pentachlorophenol	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Phenanthrene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Phenol	E.41	E.46	.99	<.50	.58	E.16	E.11	E.26
Prometon	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Pyrene	<.50	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Tetrachloroethylene	E.012	<.50	<.50	<.50	<.50	<.50	<.50	<.50
Bromoform	E.012	E.041	E.022	<.5	<.5	<.5	E.018	<.5

Overall, 42 of the 108 targeted constituents in the collected water-quality samples were detected, and the most frequently detected constituents included phenol, caffeine, *para*-cresol, and acetyl hexamethyl tetrahydro naphthalene (AHTN) (fig. 3) for samples collected in March, April, and August 2004. At sites upstream from WWTP effluent discharges, the most common constituents found were caffeine, *para*-cresol, and phenol. These constituents are relatively mobile and widely used in common, everyday products. For example, caffeine is found in numerous beverages; phenol is used as a disinfectant in manufacturing numerous products; *para*-cresol is a common wood preservative. Phenol and *para*-cresol also were found in streambed sediments at sites in northern Arkansas, southern Missouri, southeastern Kansas, and northeastern Oklahoma (Bell and others, 1997) and at relatively undisturbed sites in north-central Arkansas (Petersen, 1999).

Several pharmaceuticals and other OWCs were detected in samples collected at sites on Mud Creek in August 2004 that

were not detected in previous samples. Recent investigations have focused on the occurrence of antibiotics and some degradation products in streams and benthic sediment (Hirsch and others, 1999). Four different antibiotics (anhydro-erythromycin, trimethoprim, ofloxacin, and sulfamethoxazole) were detected in the samples collected in March 2004 at Mud Creek downstream from the WWTP effluent discharge (sites 9 and 10; tables 2 and 3). At Spring Creek (sites 12 and 13), anhydro-erythromycin and trimethoprim were present in detectable concentrations in the samples collected in March 2004 (tables 2 and 3). Sites at Mud Creek also were sampled again in August 2004 where eight different antibiotics (erythromycin, tylosin, ciprofloxacin, sulfamethoxine, and four antibiotics previously detected) were found in water samples collected downstream from the Fayetteville WWTP (sites 9 and 10). In addition, five other OWCs (5-methyl-1H-benzotriazole, 4-*tert*-octylphenol, 3-methyl-1H-indole, carbaryl, and octylphenol, monoethoxy) were detected in the August 2004 samples that were not

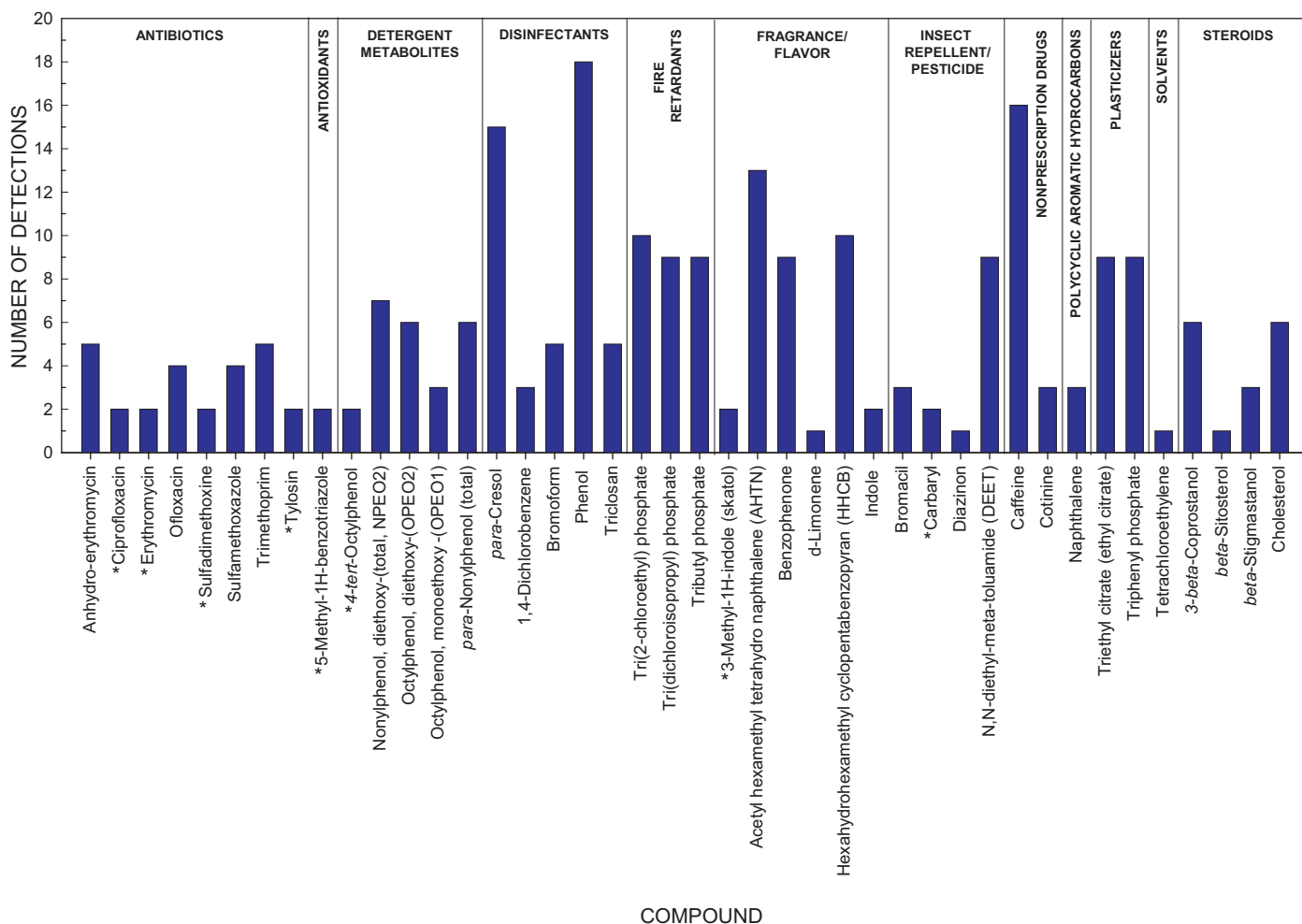


Figure 3. Detection frequencies of 42 pharmaceuticals and other organic wastewater constituents found at selected sites in northern Arkansas, March, April, and August 2004. Asterisk denotes constituents only detected in samples collected downstream from the wastewater-treatment plant effluent discharge on Mud Creek in August 2004.

detected in the March 2004 samples (table 3). Although octylphenol, monoethoxy was not detected in the samples collected at sites on Mud Creek in March 2004, it was detected at Osage Creek downstream from the Rogers WWTP effluent discharge (site 15). The other four OWCs were not detected at any of the sites in samples collected in March and April 2004. The increased number of detections of antibiotics may be explained by the decreases in laboratory detection limits of the methods used for the second set of samples (table 2). Although stream-flow conditions were similar for the two sampling events, higher water temperature and lower dissolved-oxygen concentrations were measured during the second sampling event compared to the first sampling event, and may have affected the mobilization of certain constituents (table 4).

A replicate sample collected at Spavinaw Creek near Cherokee City (site 7) indicated good reproducibility of the laboratory results with similar concentrations for two of the three detected constituents (*para*-cresol, phenol, and bromoform). Bromoform was detected in one of the samples but not the other. However, the concentration was below the laboratory reporting limit and was estimated. Two field blank samples collected, one during the routine sampling period (March 2004) and one during a resampling event at Mud Creek south of Arkansas State Highway 45 at Fayetteville (site 8) (August 2004), were compromised at the USGS National Water Quality Laboratory in Lakewood, Colorado, and did not yield sufficient results to determine the effectiveness of equipment cleaning procedures for the analysis of pharmaceuticals and other OWCs during sampling. This lack of quality assurance information decreases confidence in the environmental significance of OWC detections, especially at estimated concentrations. However, associated field blank samples sent to the USGS Organic Chemistry Research Group Laboratory in Lawrence, Kansas, for the analysis of antibiotics and antibiotic residuals did not yield any detections of the targeted constituents.

Summary

The purpose of this report is to describe the occurrence of pharmaceuticals and other OWC's, including many constituents of emerging environmental concern, in selected streams in northern Arkansas. Samples were collected in March and April 2004 from 17 sites located upstream and downstream from WWTP effluent discharges on 7 streams in northwestern Arkansas and at 1 site in a relatively undeveloped basin in north-central Arkansas. Samples also were collected again in August 2004 at three sites on Mud Creek. The targeted OWCs and sample sites were selected because WWTP effluent discharge provides a potential point source of these constituents, and analytical techniques have improved to accurately measure small amounts of these constituents in environmental samples. Potential concerns for the environmental presence of these constituents include abnormal physiological processes and reproductive impairment, increased incidences of cancer, the devel-

opment of antibiotic-resistant bacteria and plasmid transfer, and the potential increased toxicity and carcinogenic activity of the chemicals and mixtures of the constituents.

At least one pharmaceutical or other OWC was detected at all sites in 2004, except at Spavinaw Creek near Maysville, Arkansas. The number of detections generally was greater at sites downstream from WWTP effluent discharges compared to sites not influenced by municipal WWTP effluent discharges. The mean number of detections upstream from WWTP effluent discharges was 3, and the mean number of detections at sites downstream from WWTP effluent discharges was 14. Four antibiotics and five other OWCs were detected in samples collected on Mud Creek in August 2004 that were not detected in previous samples. Overall, 42 of the 108 constituents targeted in the collected water-quality samples were detected in samples collected in March, April, and August 2004. The most frequently detected constituents included phenol, caffeine, *para*-cresol, and acetyl hexamethyl tetrahydro naphthalene.

Table 4. Physical properties measured at selected sites associated with the collection of water-quality samples in northern Arkansas, 2004.

[ft³/s, cubic feet per second; mg/L, milligrams per liter; μS/cm, microsiemens per centimeter at 25 degrees Celsius; °C, degrees Celsius; --, no value; shaded column indicates site downstream from wastewater-treatment plant effluent discharge]

Map identificaton number	Station name	Date of sample collection	Sample time	Discharge (ft ³ /s)	Dissolved oxygen (mg/L)	Dissolved oxygen, percent of saturation	pH (standard units)	Specific conductance (μS/cm)	Water temperature (°C)
1	White River near Fayetteville	4/05/2004	1045	482	9.2	93	7.4	74	14
2	White River near Goshen	4/05/2004	1250	478	9.6	97	7.6	79	14
3	North Sylamore Creek near Fifty-Six	3/29/2004	1410	6.00	9.7	103	7.9	279	18
4	Decatur Branch at Decatur	4/06/2004	0800	2.10	9.5	92	7.5	232	12
5	Decatur Branch near Maysville	4/06/2004	0930	4.70	9.9	98	7.6	419	13
6	Spavinaw Creek near Maysville	4/06/2004	1050	77	10.0	98	7.5	282	13
7	Spavinaw Creek near Cherokee City	4/06/2004	1150	82	12.3	120	8.2	290	13
8	Mud Creek south of Highway 45 at Fayetteville	3/30/2004	0920	0.170	8.6	88	8.0	269	14
		8/16/2004	1055	<0.100	6.1	57	7.6	252	20
9	Mud Creek at Township Road at Fayetteville	3/30/2004	1030	6.20	9.9	103	8.1	665	15
		8/16/2004	1325	6.14	7.7	94	7.8	568	23
10	Mud Creek at Old Wire Road at Fayetteville	3/30/2004	1215	7.90	9.8	102	8.3	611	15
		8/16/2004	1520	5.75	7.8	95	8.0	581	23
11	Spring Creek at Silent Grove Road near Springdale	3/30/2004	1445	7.50	9.5	100	7.3	340	16
12	Spring Creek at North 40th Street near Springdale	3/30/2004	1615	27.0	9.7	105	7.6	673	17
13	Spring Creek at Highway 112 near Cave Springs	3/30/2004	1720	39.0	10.6	111	8.6	570	16
14	Osage Creek near County Road 51 near Rogers	3/31/2004	1050	17.0	9.2	90	7.3	295	12
15	Osage Creek near Cave Springs	3/31/2004	1150	39.0	10.9	110	7.8	388	14
16	Osage Creek at Highway 264 at Cave Springs	3/31/2004	1320	36.0	11.0	112	8.0	380	15
17	Osage Creek near Elm Springs	3/31/2004	1450	125	10.9	113	8.3	381	15
18	Illinois River south of Siloam Springs	4/05/2004	1525	346	11.6	121	8.1	315	16

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