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Compounds of Emerging Concern Detected in Water Samples from Potable Water and Wastewater Treatment Plants and Detected in Water and Bed-Sediment Samples from Sites on the Trinity River, Dallas, Texas, 2009–13

Scientific Investigations Report 2019–5019

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

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Conversion Factors

U.S. customary units to International System of Units

Multiply	By	To obtain
	Length	
inch (in.)	2.54	centimeter (cm)
foot (ft)	0.3048	meter (m)
mile (mi)	1.609	kilometer (km)
	Volume	
gallon (gal)	3.785	liter (L)
million gallons (Mgal)	3,785	cubic meter (m^3)
cubic foot (ft^3)	0.02832	cubic meter (m^3)
	Flow rate	
foot per second (ft/s)	0.3048	meter per second (m/s)
cubic foot per second (ft^3/s)	0.02832	cubic meter per second (m^3/s)
gallon per day (gal/d)	0.003785	cubic meter per day (m^3/d)
million gallons per day (Mgal/d)	0.04381	cubic meter per second (m^3/s)

Datum

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

Supplemental Information

Concentrations of chemical compounds in water are given in micrograms per liter (µg/L). Micrograms per liter is a unit expressing the concentration of chemical compounds in solution as weight of solute (micrograms) per unit volume (liter) of water.

Concentrations of chemical compounds in sediment are given in micrograms per kilogram (µg/kg).

Abbreviations

AHTN	acetyl-hexamethyl-tetrahydronaphthalene
ANOVA	analysis of variance
BPA	bisphenol A
CAFO	concentrated animal feeding operation
CECs	compounds of emerging concern
DEET	*N,N*-diethyl-*m*-toluamide
E	estimated
EDC	endocrine disrupting compound
EPA	U.S. Environmental Protection Agency
HHCB	hexahydro-hexamethyl-cyclopenta-benzopyran
LCAB	OGRL analysis for antibiotics by use of liquid chromatography/mass spectrometry
LRL	laboratory reporting level
LT-MDL	long-term method detection level
NWQL	U.S. Geological Survey National Water Quality Laboratory
OGRL	U.S. Geological Survey Organic Geochemistry Research Laboratory
PAH	polycyclic aromatic hydrocarbon
PWTP	potable water treatment plant
RPD	relative percent difference
SH	laboratory analyses for a group of compounds
SPE	solid-phase extraction
USGS	U.S. Geological Survey
WWTP	wastewater treatment plant

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By Christopher J. Churchill, Stanley Baldys III, Cathina L. Gunn, Craig A. Mobley, and Daniel P. Quigley

Abstract

The population in the Dallas-Fort Worth metropolitan area in northern Texas is rapidly growing, resulting in a rapid increase in the demand for potable water and an increase in the discharge of wastewater treatment plant effluent. An assessment of compounds of emerging concern (CECs) in samples collected at potable water and wastewater treatment plants in Dallas and downstream from Dallas in the Trinity River was completed by the U.S. Geological Survey in cooperation with the City of Dallas, Dallas Water Utilities. CECs are synthetic or naturally occurring chemicals that are not commonly monitored in the environment but can enter the environment and cause known or suspected adverse ecological or human health effects. CECs can enter the environment through nonpoint sources (for example, runoff) and point sources (for example, concentrated animal feeding operations and treated-effluent discharge from wastewater treatment plants), which can increase concentrations of CECs especially in highly populated areas. CECs include pharmaceuticals (prescription and nonprescription), steroidal hormones, stanols, sterols, detergents and detergent metabolites (hereinafter referred to as "detergents"), personal-use products, pesticides, polycyclic aromatic hydrocarbons (PAHs), flame retardants, plasticizers, and other organic compounds used in everyday domestic, agricultural, and industrial applications. The release of CECs to the environment went largely unrecognized until relatively recently. Increased loading of certain CECs to the environment, combined with advancements in laboratory analysis methods that resulted in appreciably lower detection levels, brought greater attention to the release of CECs. In addition, synthesis of new chemicals or changes in use and disposal of existing chemicals can create new sources of CECs. Some CECs are endocrine disrupting compounds (EDCs), which can elicit adverse effects on development, behavior, and reproduction of wildlife and can cause dysfunction of human and wildlife endocrine (hormone) systems.

Results of studies in the United States and Europe indicate that CECs, their metabolites, and industrial, agricultural, and household wastewater products are present in the aquatic environment, water treatment plants, and septic systems. CECs, especially pharmaceuticals, are of interest because of their persistence, widespread use, and potential to cause adverse effects in humans and nontargeted organisms. There is also concern that some CECs and EDCs resist degradation of water treatment processes at potable water treatment plants (PWTPs) and wastewater treatment plants (WWTPs) and that treated-effluent discharge could contain compounds that negatively affect biota living in receiving waters. Therefore, CECs and EDCs are more likely to be detected in environmental samples collected near areas of high population density where treated effluent from WWTPs can contribute substantially to receiving waters.

The U.S. Geological Survey, in cooperation with the City of Dallas, Dallas Water Utilities, evaluated the occurrence and concentrations of selected CECs in samples collected at PWTPs and WWTPs in Dallas and downstream from the Dallas-Fort Worth metropolitan area in the Trinity River, Texas, from August 2009 to December 2013. Water samples were collected at three PWTP sites, two WWTP sites, and five study sites on the Trinity River; all sites where samples were collected were in or downstream from Dallas. These water samples were analyzed for 120 CECs, including human-health pharmaceuticals (prescription and nonprescription), antibiotics, steroidal hormones, stanols, sterols, detergents, personal-use products (flavors and fragrances), pesticides and repellents, industrial wastewater compounds, disinfection compounds, PAHs, flame retardants, and plasticizers. Additionally, bed-sediment samples were collected at each of the five Trinity River sites. The bed-sediment samples were analyzed for 57 CECs.

In general, the water treatment processes at PWTPs and WWTPs were effective at reducing detections and concentrations of CECs to undetectable levels or transforming the compounds into degradates that were not analyzed. There were 14 and 73 CECs detected in raw water and in untreated-influent water at PWTPs and WWTPs, respectively. Of these, 11 of the 14 CECs detected in raw-water samples and 44 of the 73 CECs detected in untreated-influent samples were not

detected in finished water or in treated-effluent water samples, respectively, indicating that these compounds were removed or degraded to compounds that were not analyzed. Some CECs, however, are resistant to degradation and were detected in untreated and treated water at PWTPs and at WWTPs. The three CECs detected at PWTPs in raw-water and finished-water samples were tris(dichloroisopropyl)phosphate, benzophenone, and methyl salicylate. At WWTPs, 29 CECs were detected, including carbamazepine, sulfamethoxazole, 4-androstene-3,17-*dione*, 3-beta-coprostanol, acetyl-hexamethyl-tetrahydronaphthalene (AHTN), hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB), 1,4-dichlorobenzene, tribromomethane, benzophenone, and tris(dichloroisopropyl)phosphate, in untreated and treated water, indicating that treatment processes likely did not remove or degrade these compounds.

Of the 23 CECs detected in stream-water samples collected at 5 sites on the Trinity River in or near Dallas, 10 CECs (carbamazepine, sulfamethoxazole, caffeine, 3-beta-coprostanol, cholesterol, HHCB, benzophenone, triethyl citrate, tributyl phosphate, and tris(dichloroisopropyl) phosphate) were detected at all 5 sites. The 10 CECs detected in water samples collected at all 5 sites on the Trinity River were also detected in treated-effluent water at WWTPs.

Eleven of the 57 targeted CECs were detected in bed-sediment samples collected at study sites on the Trinity River. Of these 11 CECs, only 2 (beta-sitosterol and cholesterol) were detected in bed-sediment samples at all 5 sites on the Trinity River. Nine of these 11 CECs were not detected in any water-column sample, likely because of the strong hydrophobic characteristics of these compounds.

Results from water treatment plants indicate that the water treatment process is less effective for removing or degrading compounds that are engineered to be resistant to degradation. These results also indicate the presence of CECs and EDCs at locations upstream from PWTPs in Dallas. Results from Trinity River main-stem sites indicate that some compounds are naturally attenuated during transport, but a few are persistent throughout the study reach. Many CECs and EDCs are hydrophobic and were only detected in bed sediment, indicating multiple pathways through which CECs can persist in the environment.

In general, concentrations of CECs in the Dallas-Fort Worth metropolitan area were similar to those found in metropolitan areas nationwide.

Introduction

Dallas is in the upper Trinity River Basin in northern Texas and is the largest city in the Dallas-Fort Worth metropolitan area (Dallas-Fort Worth) with an estimated population of 1.26 million in 2015 (U.S. Census Bureau, 2015a). Dallas-Fort Worth includes 14 cities with populations greater than 100,000; the estimated population of the metropolitan area was 7.1 million in 2015 (U.S. Census Bureau, 2015b). Dallas-Fort Worth is projected to more than double in population to approximately 15 million by 2060 (Texas Water Development Board, 2010). The burgeoning population of the Dallas-Fort Worth area creates an increased demand for potable water supplies, and the increasing urbanization associated with rapid population growth increases water-quality concerns. Water supplies for Dallas and nearby cities that are served by Dallas Water Utilities come from surface-water reservoirs in the Trinity River Basin and in neighboring river basins (the Red, Sabine, and Sulphur River Basins). Several of these reservoirs can contain water diverted from regions downstream from Dallas-Fort Worth through water reuse and transfer programs. For example, North Texas Municipal Water District diverts water from the East Fork Trinity River through an artificial wetland before pipelines transfer the water to Lavon Lake; Tarrant Regional Water District diverts water from the Trinity River into an artificial wetland and returns treated water to reservoirs in Dallas-Fort Worth (Texas Water Development Board, 2011). Drought conditions during 2011–14 reduced water levels in these regional reservoirs, which adversely affected potable-water supplies. Regional water supplies were further reduced because of the presence of zebra mussels in Lake Texoma in the Red River Basin (Churchill and Baldys, 2012). The presence of zebra mussels precluded interbasin transfer of water from Lake Texoma to Dallas-Fort Worth. Drought conditions during 2011–14 also led to increased scrutiny on the quality of source and treated municipal waters, including raw water, finished water, untreated-influent and treated-effluent wastewater, and receiving waters of wastewater discharges.

The release of compounds of emerging concern (CECs) to the environment went largely unrecognized until the 1970s; laboratory analysis methods that resulted in appreciably lower detection levels and increased media coverage have brought increased attention to the issue of CECs in recent years (National Association of Clean Water Agencies, 2010). CECs have become a water-quality concern in many large urban areas such as Dallas-Fort Worth. CECs can be broadly defined as any synthetic or naturally occurring chemical that is not commonly monitored in the environment but has the potential to enter the environment and cause known or suspected adverse ecological or human health effects (U.S. Geological Survey [USGS], 2018a). CECs include pharmaceuticals (steroidal hormones and their metabolites and antibiotics), naturally occurring steroids, stanols, sterols, and other organic compounds used in everyday domestic, agricultural, and industrial applications. Releases of CECs to the environment were not widely recognized until laboratory analysis methods capable of detecting concentrations in the parts per billion range or sub parts per billion range were developed. In addition, synthesis and widespread use of new chemicals such as pharmaceuticals have increased the loading of CECs to the environment. Changes in use and disposal of existing chemicals can also create new sources of CECs. There is increasing interest in CECs, especially pharmaceuticals,

because of their persistence, widespread use, and potential to cause adverse effects in humans and nontargeted organisms (Daughton and Ternes, 1999; Kolpin and others, 2002; Kim and others, 2007; Brooks and others, 2009; Raghav and others, 2013). Many pharmaceutical compounds are designed to resist physical, chemical, and biological degradation, resulting in as much as 90 percent of the ingested drug being excreted in its biologically active form (Jjemba, 2006). The use of pharmaceuticals has steadily increased in the United States. The number of Americans who took at least one prescription drug increased from 44 to 48 percent from 1999 to 2008, and those who took two or more prescription drugs increased from 25 to 31 percent. Moreover, from 1990 to 2008, those who took five or more drugs increased from 6 to 11 percent (Gu and others, 2010). The increase in prescription drug use can concomitantly increase the potential for larger amounts of pharmaceuticals to enter potable water supplies and wastewater effluent (Buxton and Kolpin, 2002).

Results of studies in the United States and Europe indicate that CECs, their metabolites, and industrial, agricultural, and household wastewater products are widely present in the aquatic environment and are found in the water in water treatment plants and septic systems (Stackelberg and others, 2004; Clara and others, 2005; Glassmeyer and others, 2005; Godfrey and others, 2007; Phillips and others, 2010; Phillips and others, 2012). Buxton and Kolpin (2002) reported that several compounds, steroids, insect repellents, and nonprescription drugs were frequently detected in surface-water samples collected across the United States. Examples of recent reports documenting the presence of CECs in the aquatic environment in different parts of the United States include reports pertaining to the presence of CECs in North Carolina (Ferrell, 2009); Minnesota (Lee and others, 2010); Pennsylvania (Reif and others, 2012); and Wisconsin (Tomasek and others, 2012). Opsahl and Lambert (2013) also reported on the presence of CECs in the San Antonio River Basin in south-central Texas, and evaluated detections, concentrations, and distribution patterns of selected CECs downstream from effluent discharge locations in San Antonio.

Some CECs are endocrine disrupting compounds (EDCs), which can elicit adverse effects on development, behavior, and reproduction of wildlife and can cause dysfunction of human and wildlife endocrine (hormone) systems. Although adverse effects of EDCs on human health are not clearly understood and can be difficult to predict (Kolpin and others, 2002; Brooks and others, 2009), several studies have documented the effects of EDCs on ecological health. For example, Crain and Guillette (1997) found that EDCs can cause feminization in alligators, and Kirk and others (2003) found that EDCs are estrogenic in fish, birds, and mammals. To further study the effect that EDCs have on endocrine systems, the U.S. Environmental Protection Agency (EPA) instituted the Endocrine Disruptor Screening Program in 2014 that superseded the original comprehensive management plan issued in June 2012 with targeted objectives for 2014–19 (EPA, 2014).

CECs and EDCs are more likely to be detected in environmental samples collected near areas of high population density where treated effluent from wastewater treatment facilities can contribute substantially to receiving waters (Clara and others, 2005). An additional source of CECs can be from concentrated animal feeding operations (CAFOs), such as cattle or chicken feedlots, because of the widespread use of pharmaceutical compounds in animal feed (Campagnolo and others, 2002). Several poultry and cattle CAFOs are in the Trinity River Basin upstream from Dallas. Although most of the Trinity River Basin is serviced by enclosed sewer systems, rural systems can contribute CECs to tributaries of the Trinity River through rainfall runoff. Land application of biosolids has also been identified as a potential way for CECs to enter the environment (Reif and others, 2012; Raghav and others, 2013). The Trinity River Authority of Texas and the City of Fort Worth use biosolid application programs in the Trinity River Basin (City of Fort Worth, 2018; Trinity River Authority of Texas, 2018).

Some CECs are known to resist degradation during water treatment processes at wastewater treatment plants (WWTPs) (Venkatesan and Halden, 2014). If treated effluent discharged from WWTPs to the Trinity River contains CECs that were not degraded during treatment, these compounds could negatively affect the receiving waters of the Trinity River by causing adverse ecological or human health effects. Stream-water and bed-sediment data obtained from samples collected from the Trinity River could yield information regarding the transport, attenuation, and fate of CECs in this effluent-dominated river. To date, the occurrence, concentrations, and distributions of CECs in waters of the Trinity River Basin have not been studied. Therefore, the USGS, in cooperation with the City of Dallas, Dallas Water Utilities, assessed the occurrence, concentrations, and distributions of CECs in raw water and finished water at the City's potable water treatment plants (PWTPs), in untreated-influent and treated-effluent water at the City's WWTPs, and in water and bed sediment at five sites in the receiving waters of the Trinity River between Dallas and Trinidad, Tex.

Purpose and Scope

This report summarizes detections, concentrations, and distributions of CECs in raw- and finished-water samples collected at three PWTPs and in untreated-influent and treated-effluent water samples collected at two WWTPs in Dallas, Tex., and in stream-water and bed-sediment samples collected at five study sites on the main stem of the Trinity River during 2009–2013. The analyzed CECs were human-health prescription and nonprescription pharmaceuticals (14 compounds); antibiotics (30 compounds); steroidal hormones and their metabolites, stanols, and sterols (21 compounds); and organic compounds found in domestic and industrial wastewater (55 compounds). In addition to quality-control samples, seven environmental water samples were collected at the raw-water intake (inflow to plant) and

from the finished water (outflow from plant) of three PWTPs and at the untreated-influent intake and from treated effluent of two WWTPs. Environmental samples collected at each of the five study sites on the Trinity River consisted of seven width- and depth-integrated stream-water samples and seven bed-sediment samples. A total of 129 total samples were analyzed during the study; of these 129 samples, 105 were environmental samples, and 24 were quality-control samples. Results from the quality-control samples were used to help interpret and provide context for the results obtained from the environmental samples.

Description of Study Area

The Trinity River extends approximately 710 miles and flows generally southeast from near the Oklahoma border to Galveston Bay near Houston, Tex. (fig. 1). The river's headwaters are composed of the West Fork Trinity River (West Fork), Clear Fork Trinity River (Clear Fork), Elm Fork Trinity River (Elm Fork), and East Fork Trinity River (East Fork) in north-central Texas. Downstream from where Clear Fork flows into West Fork, the main stem of the Trinity River begins west of downtown Dallas at the confluence of West Fork and Elm Fork. The confluence of East Fork with the main stem is 12 miles southeast of Dallas and downstream from the treated-effluent discharge from WWTP-2, but upstream from USGS station 08062500 Trinity River near Rosser, Tex. (hereinafter referred to as the Trinity River Rosser site) and USGS station 08062700 Trinity River at Trinidad, Tex. (hereinafter referred to as the Trinity River Trinidad site). Streamflow upstream from the confluence of West Fork and Elm Fork is regulated by reservoirs that are mostly in the Dallas-Fort Worth area. These reservoirs include Benbrook Lake, Lake Bridgeport, Eagle Mountain Lake, Ray Roberts Lake, Lake Lewisville, Grapevine Lake, Lavon Lake, and Ray Hubbard Lake. Reservoirs in or near the Dallas-Fort Worth area store most of the potable water supply for Dallas-Fort Worth and are sustained by natural inflows and inter- and intrabasin diversions. The Trinity River downstream from Dallas-Fort Worth flows unregulated until it is impounded by Lake Livingston near Houston.

Of the 339 permitted WWTPs in the Trinity River Basin, 192 are upstream from the Trinity River Trinidad site (J.R. Lueg, Texas Commission on Environmental Quality, written commun., 2014). WWTPs with the capacity to discharge the largest amounts of treated effluent are in Dallas-Fort Worth between source-water reservoirs and the confluence of the East Fork Trinity River. Because of the large amount of treated-effluent discharges from the WWTPs in Dallas-Fort Worth, the Trinity River can be a wastewater-dominated system, especially during base-flow conditions.

The City of Dallas operates three PWTPs and two WWTPs (table 1). Raw-water intakes for the three PWTPs are upstream from the discharge points for the two WWTPs. Other WWTPs discharge into the tributaries of the Trinity River upstream from Dallas WWTP discharge points (Trinity River Authority of Texas, 2014). Treated effluent from other WWTPs that are upstream from the potable water-supply reservoirs were not sampled as part of this study but could be potential point sources for CECs in the influent waters of Dallas' PWTPs.

Potable Water Treatment Plants

The three PWTPs receive water for treatment from the following regional reservoirs: Grapevine Lake, Lake Fork Reservoir, Lake Lewisville, Lake Tawakoni, Ray Hubbard Lake, and Ray Roberts Lake (fig. 1). The combined treatment capacity of the three PWTPs is 900 million gallons per day (Mgal/d) with a record single-day water use of 789.6 million gallons on September 4, 2000 (Dallas Water Utilities, 2018). The largest PWTP, PWTP-1, receives source waters from Ray Hubbard Lake by direct intake and from Lake Tawakoni by interbasin transfer. The City of Dallas uses conventional treatment processes (such as partial softening) as the primary type of treatment at PWTP-2 and PWTP-3 (table 1). Partial softening alters the hardness of water to aid in the removal of heavy metals, radionuclides, and dissolved organics and the reduction of viruses and bacteria (Pontius, 1990). The City of Dallas also uses other processes including sedimentation, chlorination, lime softening, coagulation and flocculation, dual-media filtration using anthracite coal and sand, and post-filter corrosion control (Christopher Holmgren, Dallas Water Utilities, oral commun., 2014). PWTP-1 used partial softening as primary treatment until June 2012, when enhanced coagulation was started (Peter Stencel, Dallas Water Utilities, oral commun., 2014). Coagulation is a process that combines small particles into larger aggregates to facilitate their removal (Pontius, 1990). Enhanced coagulation is part of the process by which raw water is purified for human consumption; inorganic chemicals (ferric sulfate and polymers) are added to the water to cause suspended particles to bind together until flocculation occurs. Flocculation is a physical process that promotes interparticle contact and clumping of particles, which increase in size (Pontius, 1990). After flocculation occurs, large particles descend gravimetrically or are mechanically removed from suspension by using settling ponds or filtration. PWTPs use chlorination or ozonation as a disinfectant to treat finished water to the point where it is safe for human consumption. All PWTPs where samples were collected use chlorination and ozonation as primary and secondary disinfectants, respectively.

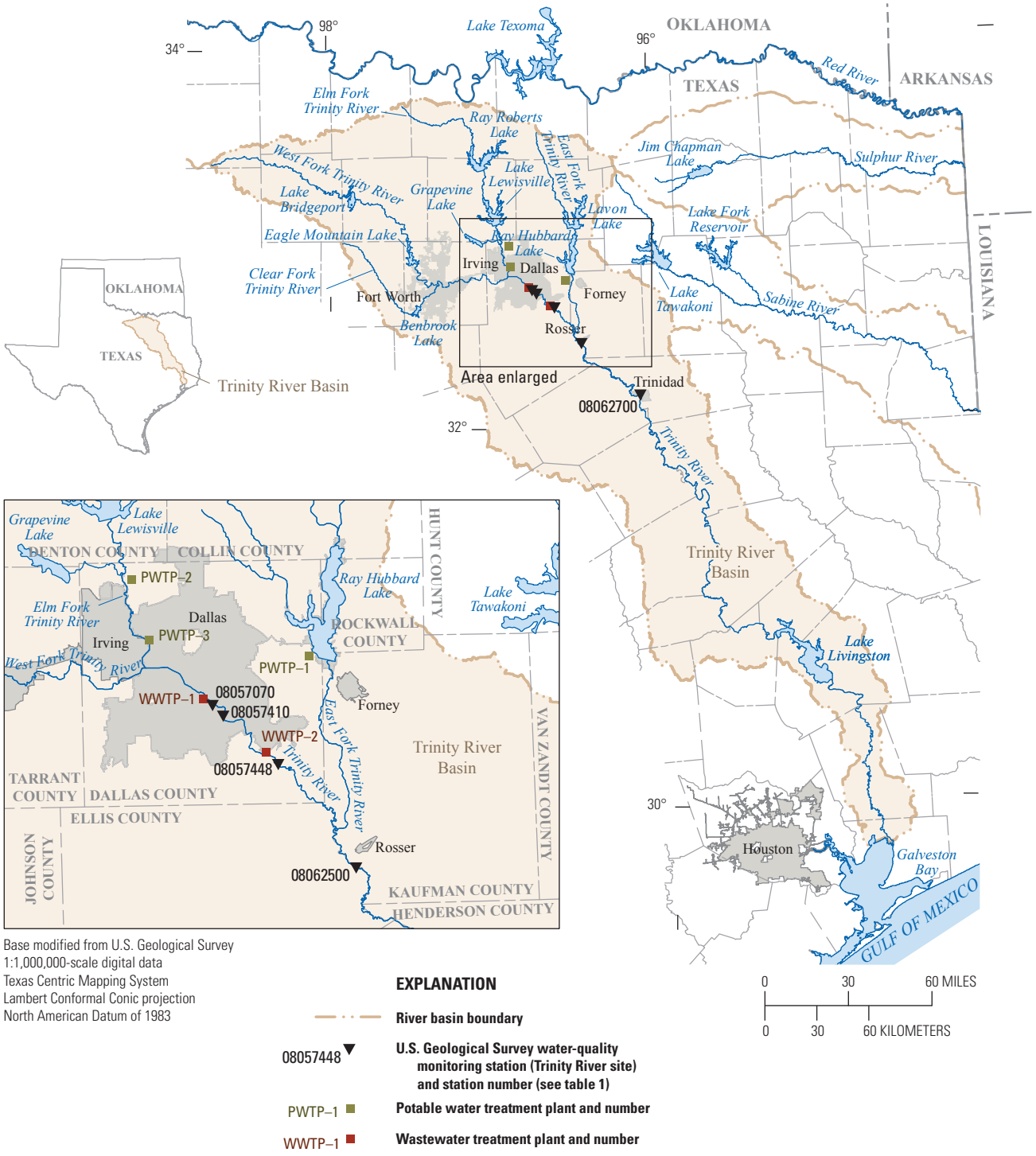


Figure 1. Locations of potable water and wastewater treatment plants and U.S. Geological Survey water-quality monitoring stations on the Trinity River in or near Dallas, Texas, where samples were collected during 2009–13.

Table 1. Water-quality study sites in or near Dallas, Texas, 2009–13.

[--, not applicable; USGS, U.S. Geological Survey]

Map identifier (see fig. 1)	Description of study site	Primary source of water	Primary type of treatment	Disinfectants used	Trinity River mile
		Potable water treatment plants			
PWTP-1	Raw water	Ray Hubbard Lake, Lake Tawakoni, and Lake Fork Reservoir	Partial softening/ enhanced coagulation[1]	--	--
	Finished water	--	--	Chlorine and ozone[1]	--
PWTP-2	Raw water	Lake Lewisville, Grapevine Lake, and Ray Roberts Lake	Partial softening[2]	--	--
	Finished water	--	--	Chlorine and ozone[2]	--
PWTP-3	Raw water	Lake Lewisville and Grapevine Lake	Partial softening[3]	--	--
	Finished water	--	--	Chlorine and ozone[3]	--
		Wastewater treatment plants			
WWTP-1	Untreated-influent water 1	--	Activated sludge[4]	--	--
	Untreated-influent water 2	--	Activated sludge[4]	--	--
	Treated-effluent water	--	--	Chlorine/sulphur dioxide	495.0
WWTP-2	Untreated-influent water	--	Activated sludge and sludge digestion[5]	--	--
	Treated-effluent water	--	--	Chlorine/sulphur dioxide	481.0

Map identifier (see fig. 1)	USGS station name (and short name)	Trinity River mile
	USGS water-quality monitoring stations (Trinity River sites)	
08057070	Trinity River at State Highway 310, Dallas, Tex. (Trinity River Highway 310 site)	494.6
08057410	Trinity River below Dallas, Tex. (Trinity River Dallas)	491.8
08057448	Trinity River near Wilmer, Tex. (Trinity River Wilmer)	478.4
08062500	Trinity River near Rosser, Tex. (Trinity River Rosser)	451.4
08062700	Trinity River near Trinidad, Tex. (Trinity River Trinidad)	391.2

[1]Primary type of treatment changed from partial softening to enhanced coagulation starting in June 2012 (Peter Stencel, Dallas Water Utilities, oral commun., 2014).

[2]Jim Crowley, Dallas Water Utilities, oral commun., 2014.

[3]Chaise Holmgren, Dallas Water Utilities, oral commun., 2014.

[4]Daniel Halter, Dallas Water Utilities, oral commun., 2014.

[5]Nosa Irenumaagho, Dallas Water Utilities, oral commun., 2014.

Wastewater Treatment Plants

The two WWTPs in Dallas receive wastewater from a wastewater system and from 11 wholesale water customers (Dallas Water Utilities, 2018). The two plants have a combined capacity of 260 Mgal/d and treated a combined mean of 72 Mgal/d during 2010–11 (52.8 billion gallons total) (Dallas Water Utilities, 2018). The sources of water to the two WWTPs are water that goes down drains (including water from toilet systems) in homes and businesses and wastewater that is delivered by pipeline from wholesale water customers. Stormwater is separated into another collection system that is not treated before it is released to the Trinity River. Generally, wastewater from the southern and southwestern areas of the city is treated at WWTP-2, and the remaining wastewater is treated at WWTP-1 (fig. 1). However, wastewater can be transferred to either plant as needed based on treatment demands and weather (Paul Sill, Dallas Water Utilities, oral commun., 2014). WWTP-1 had two different influent sampling locations at the time of sampling. Both sampling locations represented the same step in the water treatment process (untreated influent). Inflows to WWTP-1, regardless of sampling location within the plant, are considered to carry sewage from the same area of Dallas. WWTP-1 uses activated sludge as the primary method of treatment. Activated sludge treatment is a process for removing organic matter from sewage by saturating it with air and microorganisms that can break down the organic matter (EPA, 2004). WWTP-2 uses activated sludge followed by sludge digestion, during which the sludge is placed in tanks where volatile organic materials are decomposed by bacteria, resulting in partial gasification, liquefaction, and mineralization of volatile organic compounds (EPA, 2004). Sludge from WWTP-1 is pumped to WWTP-2 for sludge digestion. Treated effluent is disinfected by chlorine, and then sulfur dioxide is added to remove residual chlorine (Daniel Halter, Dallas Water Utilities, oral commun., 2014).

Trinity River Study Sites

The five study sites on the Trinity River span more than 100 river miles (table 1). In upstream to downstream order, the sites are as follows: (1) USGS station 08057070 Trinity River at State Highway 310, Dallas, Tex. (hereinafter referred to as the Trinity River Highway 310 site); (2) USGS station 08057410 Trinity River below Dallas, Tex. (hereinafter referred to as the Trinity River Dallas site); (3) USGS station 08057448 Trinity River near Wilmer, Tex. (hereinafter referred to as the Trinity River Wilmer site); (4) the Trinity River Rosser site; and (5) the Trinity River Trinidad site (fig. 1). The Trinity River Highway 310 site (river mile 494.6) is downstream from the treated-effluent discharge point for treated effluent from WWTP-1 (river mile 495.0) and downstream from discharge points for other WWTPs located upstream on the West Fork Trinity River. Large WWTPs on the West Fork Trinity River upstream from WWTP-1 include the Village Creek Water Reclamation Facility (166 Mgal/d) (City of Fort Worth, 2018) and the Trinity River Authority of Texas Central Regional Wastewater System (162 Mgal/d) (Trinity River Authority of Texas, 2018). The next site downstream from the Trinity River Highway 310 site is the Trinity River Dallas site. The Trinity River Highway 310 site and the Trinity River Dallas site are in a highly urbanized area of southeast Dallas, and there are no substantial inflows in the 2.8-mile reach between these sites. Treated-effluent discharge from WWTP-2 enters the main stem of the Trinity River at river mile 481.0, which is between the Trinity River Dallas site and the Trinity River Wilmer site. Flow from the East Fork Trinity River enters the main stem of the Trinity River downstream from the Trinity River Wilmer site and upstream from the Trinity River Rosser site. The farthest downstream site is Trinity River Trinidad (river mile 391.2).

Methods

A total of 105 environmental samples were collected by the USGS from 2009 to 2013 in the Dallas-Fort Worth study area. In addition to the environmental samples, 24 quality-control samples were collected, consisting of 11 replicate samples, 9 equipment blank samples, 4 matrix-spike samples (1 of which was split and analyzed in replicate). Replicate samples are “two or more water samples that are collected, prepared, and analyzed such that they are essentially identical in composition and analysis” (Mueller and others, 2015, p. 7). Equipment blanks “are samples that are intended to demonstrate that sample collection and processing equipment and equipment-cleaning procedures are not sources of contamination” (Mueller and others, 2015, p. 5). A spike sample is a “water sample fortified (spiked) with known concentrations of compounds and is defined by the location where the spike solution is added to the sample, either in the field or in the laboratory, and by the type of water that is spiked, either environmental (matrix) water or blank (reagent) water” (Mueller and others, 2015, p. 6). In conjunction with the collection of the environmental samples, selected water-quality field properties (water temperature, dissolved-oxygen concentration, pH, and specific conductance) were measured in the field at each study site by using methods described in the USGS National Field Manual for the Collection of Water-Quality Data (USGS, variously dated).

Sample-Collection Methods

Water samples collected for analysis of pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds are susceptible to contamination because many of the targeted compounds are ubiquitous in the environment. To ensure sample integrity, on the day of sampling activities, field personnel avoided contact with or consumption of products that contain targeted compounds

following methods as outlined in Chapter A5, Section 5.6.1F of the USGS National Field Manual (USGS, variously dated). Efficiency of sample-collection methods and laboratory analyses was assessed by using quality-control samples, such as field blanks, which are used to test for bias from the introduction of contamination into environmental samples.

Sample-collection equipment, including sample containers, filters, and associated tubing, is a possible source of field contamination (contamination that occurs before the sample is sent to analyzing laboratories). Sample-collection and sample-processing equipment were prepared by using standard methods described in the USGS National Field Manual (USGS, variously dated). Specifically, these methods included cleaning sample-collection equipment by washing with detergent and rinsing with tap water, then distilled water, then methanol, then organic-free blank water, then air drying.

Sample Collection at Potable Water Treatment Plants

Raw-water and finished-water samples were collected inside PWTPs. Raw-water grab samples were collected prior to the addition of ozone intake structure settling basins at PWTP-1 and PWTP-3 by using weighted bottle samplers and Teflon sample bottles. Raw-water samples were collected from an intake pipe at PWTP-2. Finished-water samples were collected from a pipe containing finished water prior to the application of chlorine for disinfection purposes at all PWTPs; samples were collected from the spigots after field properties had stabilized. Finished water from a PWTP is defined as water that has been fully treated by the processes of a plant but has not been placed in a pipeline to be delivered for consumption (Sacramento State Office of Water Programs, 2017). The time required to process a volume of water from start to finish (that is, residence time) in the PWTPs was not available. Raw- and finished-water samples were collected at the three PWTPs seven times during 2009–13. Seven environmental samples were collected from the intake (raw water) and outflow (finished water) from each of the 3 PWTPs, for a total of 42 environmental samples. Samples were filtered into sample bottles provided by the respective analyzing laboratory (the USGS National Water Quality Laboratory [NWQL] in Lakewood, Colorado or the USGS Organic Geochemistry Research Laboratory [OGRL] in Lawrence, Kansas) and shipped rapidly to meet all specified holding times (Sandstrom, 1995).

Sample Collection at Wastewater Treatment Plants

Untreated-influent grab samples at the WWTPs were collected from basins that contain wastewater prior to the step of the treatment process when surface skimmers remove large solid materials. Treated-effluent samples at WWTPs were collected from a pipe located in a laboratory at each plant. Treated-effluent samples were collected from WWTPs after chlorination but before the application of sulfur dioxide for dechlorination. Treated effluent from a WWTP is defined as water that has been fully treated by the processes of a plant but has not been released into a conveyance channel to the receiving waters. Treated-effluent samples from WWTPs were filtered into bottles that contained dechlorination reagents (ascorbic acid and sodium sulfite) in the event residual chlorine was in the treated water. A residence time of 10–14 hours in WWTPs was estimated by plant operators (Richard Seely and Mark Evers, Dallas Water Utilities, oral commun., 2009). Collection times for untreated-influent and treated-effluent samples collected after August 2009 at each WWTP were based on residence times to ensure that each sample contained the same volume of water. Seven environmental samples were collected from the intake (untreated-influent water), and seven environmental samples were collected from the outflow (treated-effluent water) at each of the two WWTPs between 2009 and 2013, for a total of 28 samples.

Sample Collection at Trinity River Sites

Seven width- and depth-integrated stream-water samples were collected during 2009–2013 at each of the five USGS water-quality monitoring stations on the main stem of the Trinity River. Samples represented periods of base flow except for one sampling event during December 11–26, 2009, when streamflow ranged from 1,450 to 2,800 cubic feet per second at the five sites (U.S. Geological Survey, 2018b). Stream-water samples were collected by using either equal-depth or equal-width integrated sampling techniques as described in the USGS National Field Manual (USGS, variously dated). Samples were usually processed onsite in a mobile USGS laboratory and shipped from the USGS North Texas Program Office laboratory in Fort Worth to the respective analyzing laboratory (NWQL or OGRL) within 1–2 days after collection to meet holding times established for each laboratory analysis. Because the channel bed was armored with large rocks under the bridges where bed-sediment samples were collected from several sites on the Trinity River, standard bed-sampling equipment could not be used. In these cases, bed-sediment samples were collected by wading from the bank and scooping bed sediment into the sample container.

Laboratory Analysis

Four laboratory analysis schedules, each targeting a specific group of compounds, were used for water samples collected at the three types of study sites (PWTPs, WWTPs, and Trinity River sites) and one laboratory analysis schedule was used for bed-sediment samples collected at Trinity River sites. Water samples were sent to the NWQL for analysis by using one of three different laboratory schedules targeting a different group of compounds: (1) human-health

pharmaceuticals (schedule SH2080); (2) steroidal hormones and metabolites (schedule SH2434); and (3) wastewater compounds: stanols, sterols, detergents and detergent metabolites, personal-use products, pesticides, disinfection compounds, flame retardants, and plasticizers (schedule SH1433) (table 2, at end of report). Part of each stream-water sample collected at the five Trinity River sites was sent to the OGRL for analysis of antibiotics by using their liquid chromatography/mass spectrometry antibiotics (LCAB) schedule (Meyer and others, 2007).

Analysis of wastewater compounds in bed-sediment samples collected at each stream site was done by the NWQL (schedule SH5433). About 120 compounds were analyzed in each bed-sediment sample; most of the same compounds were included in the different wastewater compound schedules for water. Methods for analyzing CECs in bed-sediment samples were developed in response to increasing concern about the effects of CECs in wastewater and wastewater-affected sediment on aquatic organisms (National Association of Clean Water Agencies, 2010). Occasionally, some compounds were not analyzed in a given sample for various reasons including internal laboratory quality-assurance data that identified compound concentrations that could not be determined because of nonspectroscopic interferences with the laboratory methods, referred to as matrix effects, that suppress or enhance the analytical signal or physically interfere with the analysis (Wolf and Adams, 2015).

Concentrations of compounds detected by the laboratory analysis schedules are reported with a numerical value (that is, a detection) if the value for the compound exceeds the laboratory reporting level (LRL). The analytical quantification procedure used by the NWQL for reporting results is based on the long-term method detection level (LT-MDL) and LRL. The LT-MDL is derived by determining the standard deviation of a minimum of 24 replicate spike-sample measurements near the previously reported LT-MDL over an extended period (typically 6 to 12 months) (Childress and others, 1999). The LT-MDL concentrations are defined as a censoring limit for most analytical methods at the NWQL, and their purpose is to limit false positives to less than or equal to 1 percent. A false positive measurement error occurs when a compound is incorrectly reported as present in the sample when it is not. The LRL is defined as twice the LT-MDL and is established to limit the occurrence of false negative detections to less than or equal to 1 percent. A false negative measurement error is the reporting of nondetection for a compound present in the sample (that is, not detecting a compound when it is present) (Childress and others, 1999). Generally, the probability of a false negative or false positive measurement error is predicted to be less than or equal to 1 percent when the compound concentration in the sample is equal to or less than the LRL (Childress and others, 1999). Values for several compounds were further censored to reduce the likelihood of reporting false positives for compound detections and are discussed in the "Blank Samples" section of this report. The LRL for some compounds varied during the study period (table 2). The use of variable LRLs by the laboratory increased the difficulty of computing detection frequencies because there was no single basis for detection when concentrations were reported as less than a maximum LRL. A constituent concentration was considered estimated by the laboratory when results were greater than the LT-MDL and less than the LRL; that is, a detection was considered likely, but quantification was considered questionable. These values were not considered detections. The remark code of "E" (estimated) was assigned by the laboratory for these results.

Human-Health Pharmaceuticals in Water Samples

Concentrations of human-health pharmaceutical compounds were determined at the NWQL by using laboratory schedule SH2080, except for ibuprofen, which was determined at the OGRL by using the LCAB schedule. Laboratory schedule SH2080, developed by the Methods Research and Development Program of the NWQL (Furlong and others, 2008), was used for analysis of 14 commonly used human-health pharmaceuticals by using solid-phase extraction (SPE) and high-performance liquid chromatography/mass spectrometry. For this study, human-health pharmaceuticals were separated into prescription and nonprescription pharmaceuticals (table 2). Concentrations for two compounds, the antibiotics sulfamethoxazole and trimethoprim, were determined by schedules SH2080 and LCAB, respectively, and are discussed in the "Antibiotics in Water Samples" section of this report. For the sample collected on November 15, 2010, the results from the LCAB schedule were not complete because the concentrations for several antibiotic compounds were not determined. During statistical analyses of these missing values, the sample count was reduced by one for each missing value.

Antibiotics in Water Samples

The antibiotic schedule, LCAB, was developed at the OGRL and was modified from the liquid chromatography/tandem mass spectrometry version of the online SPE method in Meyer and others (2007). Water samples were analyzed for 30 antibiotic compounds and their associated degradation products (6 quinolones, 6 macrolides, 5 sulfonamides, 9 tetracycline antibiotics, and 4 other antibiotics: lincomycin, trimethoprim, chloramphenicol, and ormetoprim). In addition, ibuprofen, a nonprescription pharmaceutical, was determined by using the LCAB schedule. Samples were analyzed by using online SPE and ultra-pressure liquid chromatography/tandem mass spectrometry with electrospray ionization with multiple reaction monitoring. Samples were analyzed in positive-ion mode except for chloramphenicol and ibuprofen, which were analyzed in negative-ion mode. Samples were extracted by using hydrophilic/lipophilic-balanced SPE cartridges (Waters Corp., Milford, Massachusetts).

Internal standards used are 13C3-15N-ciprofloxacin, clinafloxacin, 13C2-erythromycin, 13C2-erythromycin-H2O, meclocycline, simatone, 13C6- sulfamethoxazole, 13C3-trimethoprim, carbamazepine-*d*10, 13C3-ibuprofen, and 13C3- chloramphenicol. Surrogate standards included demeclocycline, nalidixic acid, oleandomycin, and 13C6-sulfamethazine. A replicate sample, a matrix-spike sample, and a carryover blank sample were analyzed after every 10th sample. A check standard was analyzed after every 20th sample. Also, two blank samples were interspersed for analysis between the environmental samples. All standard solutions, blanks, and matrix spikes were treated the same as the environmental water samples. LRLs ranged from 0.005 to 0.010 microgram per liter (µg/L) for all antibiotics analyzed except chloramphenicol (0.10 µg/L) and ibuprofen (0.05 µg/L).

Steroidal Hormones, Stanols, and Sterols in Water Samples

Concentrations of steroidal hormones, stanols, and sterols were determined primarily by laboratory schedule SH2434 (table 2). Laboratory schedule SH2434 was developed by the NWQL to detect a suite of 17 steroid hormones, 2 stanols, and 2 sterols by using SPE, derivatization (the conversion of compound into a different compound), and gas chromatography with tandem mass spectrometry (Foreman and others, 2012). Analysis is carried out by gas chromatography with tandem mass spectrometry by using calibration standards that are derivatized concurrently with the sample extracts (Li and others, 2017). Steroidal hormones were separated into categories of natural and synthetic estrogens, androgens, and progestins.

Wastewater Compounds in Water Samples

Laboratory schedule SH1433 was developed by the NWQL to determine concentrations of 57 compounds typically found in domestic and industrial wastewater, some of which could affect human health, by using polystyrene-divinylbenzene SPE and capillary-column gas chromatography/mass spectrometry (Zaugg and others, 2007) (table 2). Analyzed compounds were divided into the following classes: (1) stanols and sterols; (2) detergents and detergent metabolites (hereinafter referred to as "detergents"); (3) personal-use products; (4) pesticides; (5) industrial wastewater compounds; (6) disinfection compounds; (7) polycyclic aromatic hydrocarbons (PAHs); and (8) flame retardants and plasticizers. When the analysis of data for this report began in 2009, pentachlorophenol (parameter code 34459), BPA (parameter code 62069), caffeine (parameter code 50305), and cotinine (parameter code 62005) were analyzed by schedule SH1433. By the completion of the study, they were not included in schedule SH1433. Pentachlorophenol was removed from schedule SH1433 shortly after sample collection began; because it was not included in another schedule, this compound is not mentioned further in this report. Concentrations of caffeine (parameter code 50305) were determined by schedule SH1433. Concentrations of cotinine (parameter code 62005) were determined by schedule SH2080. Concentrations for 3-*tert*-butyl-4-hydroxyanisole (parameter code 62059) were reported for samples collected during the first round of sampling in 2009 and occasionally thereafter because of the highly variable nature of the compound when it was analyzed by schedule SH1433.

Wastewater Compounds in Bed-Sediment Samples

Laboratory schedule SH5433 uses methods developed by the NWQL for the detection of 57 compounds in environmental sediment and soil samples (table 2). Method development focused on the determination of many compounds that are targeted by schedule SH1433 but was designed for sediment-bound compounds by using pressurized solvent extraction, SPE, and capillary-column gas chromatography/mass spectrometry (Burkhardt and others, 2006). Compounds are extracted from sediment and soil samples by using a pressurized solvent extraction system. Compounds of interest are extracted from interfering matrix components by using high-pressure water/isopropyl alcohol extraction. Compounds are isolated by using disposable SPE cartridges containing chemically modified polystyrene-divinylbenzene resin. Cartridges are dried with nitrogen gas, and sorbed compounds are eluted with methylene chloride (80 percent)-diethyl ether (20 percent) through the SPE cartridge. Concentrations are determined by capillary-column gas chromatography/mass spectrometry.

The lists of compounds analyzed by schedules SH5433 and SH1433 are similar, with 52 compounds in each schedule. Cotinine, 5-methyl-1H-benzotriazole, tribromomethane (bromoform), caffeine, carbaryl, triethyl citrate, tetrachloroethylene, metalaxyl, 4-nonylphenol, and methyl salicylate were analyzed by schedule SH1433 or SH2080 but not by schedule SH5433. Compounds 2,2',4,4'-tetrabromodiphenyl ether, 4-nonylphenol monoethoxylate, atrazine, bis(2-ethylhexyl) phthalate, and diethyl phthalate were analyzed by schedule SH5433, but not by schedule SH1433. Some of these compounds have LRLs that changed during the study. Human-health pharmaceuticals, antibiotics, and hormones were not targeted in bed-sediment sample analyses.

Duplicate Analyses of Selected Compounds

Concentrations were determined by using two separate analysis schedules for each sample for the following seven selected compounds: carbamazepine, sulfamethoxazole, trimethoprim, caffeine, cotinine, 3-beta-coprostanol, and cholesterol (table 3). Preference was given to values determined by methods where the associated quality-control data support the decision to use one value instead of another (the process of determining which value used is explained further in the "Quality Control" section). A method comparison quality-control check was done by determining whether a detection reported by the preferred method with a concentration above the LRL of the other, nonpreferred method was reported as a detection by the nonpreferred method (table 3).

Research methods represented by the LCAB schedule provided lower LRLs for carbamazepine, sulfamethoxazole, and trimethoprim compared to the LRLs available for these compounds in the NWQL schedules. Concentrations of carbamazepine and sulfamethoxazole from schedule LCAB were selected as preferred values over values determined by schedule SH2080. Concentrations of carbamazepine in most samples were less than the LRL for schedule SH2080 (0.06 µg/L) and, therefore, were not reported as detections (table 3). Concentrations of carbamazepine determined by schedule LCAB were often greater than the LRL (0.005 µg/L), therefore, values from schedule LCAB were used in the analyses for carbamazepine. Concentrations of sulfamethoxazole determined by schedule SH2080 (parameter code 62021) were less than the LRL (0.10 µg/L) in all samples. Concentrations of sulfamethoxazole determined by schedule LCAB (parameter code 62775) were often greater than the LRL (0.005 µg/L) and were used during analyses instead of those from schedule SH2080. By using the method comparison quality-control check, carbamazepine and sulfamethoxazole were frequently detected by schedule LCAB at concentrations that were greater than the LRL of schedule SH2080 for samples collected at WWTPs and in stream-water samples collected in the Trinity River main stem (table 3). These disparities could partially be caused by the higher LRLs for schedule SH2080 than for schedule LCAB. Although schedules SH2080 and LCAB performed similarly for trimethoprim, more historical laboratory quality-control data were available for schedule SH2080 compared to schedule LCAB. Therefore, trimethoprim concentrations determined by schedule SH2080 were preferred over those determined by schedule LCAB.

Caffeine and cotinine concentrations were determined by schedules SH1433 and SH2080; on both schedules, the parameter codes for caffeine and cotinine were 50305 and 62005, respectively. Although the LRL for caffeine is 0.06 µg/L on both schedules used by the NWQL, the percent recovery for caffeine was slightly better when schedule SH1433 was used compared to the percent recovery when schedule SH2080 was used (Null and others, 2019). These percent recoveries apply to the analyses of caffeine in this report and for the analyses of caffeine reported as part of other studies nationwide during 2009–13. In addition, values for caffeine for schedule SH2080 were often assigned a remark code of "E" (estimated). Cotinine concentrations determined by schedule SH2080 were preferred over those determined by schedule SH1433 because the maximum LRL was lower for schedule SH2080 (0.04 µg/L) compared to the maximum LRL for schedule SH1433 (0.80 µg/L). Schedule SH2080 also had higher percent recoveries compared to schedule SH1433.

The CECs 3-beta-coprostanol and cholesterol are included in laboratory schedules SH2434 and SH1433. Results are separated by laboratory schedule because of method-specific LRLs. Results for 3-beta-coprostanol determined by schedule SH2434 are reported under parameter code 64512 (LRL 2.0 µg/L). Results determined for 3-beta-coprostanol by SH1433 are reported under parameter code 62057 (LRL 1.8 µg/L). Concentrations of 3-beta-coprostanol (parameter code 64512) determined by schedule SH2434 were preferred over those determined by schedule SH1433 because schedule SH2434 had higher percent recoveries (Null and others, 2019). In addition, laboratory analysts' notes suggested that results from SH1433 for this compound were highly variable. Results for cholesterol determined by schedule SH2434 are reported under parameter code 64514 (LRL 0.20–2.0 µg/L). Results for cholesterol determined by schedule SH1433 are reported under parameter code 62072 (LRL 2.0 µg/L). Cholesterol concentrations determined by schedule SH2434 (parameter code 64514) were preferred over those determined by schedule SH1433 (parameter code 62072) because schedule SH2434 had higher percent recoveries.

Quality Control

Quality-control samples collected in the field consisted of blank, replicate, and matrix-spike samples. In addition to quality-control samples collected in the field, the NWQL and OGRL analyze several types of quality-control samples during laboratory analysis, such as blank, replicate, spike, and surrogate samples. Data from the NWQL and the OGRL internal quality-control samples, such as percent recoveries for individual compounds by method that were used to evaluate data quality are available in Null and others (2019).

Table 3. Compounds analyzed by using more than one laboratory method in water samples collected at study sites in or near Dallas, Texas, 2009–13.

[LRL, laboratory reporting level; µg/L, micrograms per liter; LCAB, USGS Organic Geochemistry Research Laboratory liquid chromatography/mass spectrometry antibiotics schedule; SH, USGS National Water Quality Laboratory schedule; USGS, U.S. Geological Survey; OGRL, USGS Organic Geochemistry Research Laboratory in Lawrence, Kansas; NWQL, USGS National Water Quality Laboratory in Denver, Colorado; --, not applicable; β, beta]

Compound	Parameter code	Laboratory	Laboratory schedule	Minimum LRL (µg/L)	Total number of samples	Number of detections equal to or greater than the LRL	Detections equal to or greater than the LRL (percent)	Range of detected values (µg/L)	Quality-control check (percent)[1]
Potable water treatment plant									
Carbamazepine	62793	OGRL	[2]LCAB	0.005	42	15	36	0.006–0.024	0
	62793	NWQL	SH2080	0.06	42	0	0	0.002–0.015	
Sulfamethoxazole	62775	OGRL	[2]LCAB	0.005	42	17	40	0.009–0.217	5
	62021	NWQL	SH2080	0.1	42	0	0	0.004–0.009	
Trimethoprim	62023	NWQL	[2]SH2080	0.034	42	0	0	0.009–0.009	0
	62023	OGRL	LCAB	0.005	42	0	0	--	
Caffeine	50305	NWQL	[2]SH1433	0.06	42	3	7	0.015–0.320	2
	50305	NWQL	SH2080	0.06	42	2	5	0.014–0.153	
Cotinine	62005	NWQL	[2]SH2080	0.026	42	0	0	--	0
	62005	NWQL	SH1433	0.4	42	0	0	0.040–0.041	
3-β-Coprostanol	64512	NWQL	[2]SH2434	0.2	42	1	2	0.260–0.260	0
	62057	NWQL	SH1433	1.8	42	0	0	0.200–0.300	
Cholesterol	64514	NWQL	[2]SH2434	0.2	42	11	26	0.204–0.456	0
	62072	NWQL	SH1433	2.0	42	0	0	0.200–0.500	
Wastewater treatment plant									
Carbamazepine	62793	OGRL	[2]LCAB	0.005	28	28	100	0.034–0.384	79
	62793	NWQL	SH2080	0.06	28	5	18	0.003–0.104	
Sulfamethoxazole	62775	OGRL	[2]LCAB	0.005	28	22	79	0.008–1.90	46
	62021	NWQL	SH2080	0.1	28	0	0	0.025–0.07	
Trimethoprim	62023	NWQL	[2]SH2080	0.034	28	13	46	0.043–0.175	0
	62023	OGRL	LCAB	0.005	28	1	4	0.011–0.815	
Caffeine	50305	NWQL	[2]SH1433	0.06	28	19	68	0.037–64.0	7
	50305	NWQL	SH2080	0.06	28	17	61	0.019–134	
Cotinine	62005	NWQL	[2]SH2080	0.026	28	15	54	0.043–0.275	0
	62005	NWQL	SH1433	0.4	28	8	29	0.050–2.40	
3-β-Coprostanol	64512	NWQL	[2]SH2434	0.2	28	26	93	0.223–108	11
	62057	NWQL	SH1433	1.8	28	13	46	0.300–51.0	
Cholesterol	64514	NWQL	[2]SH2434	0.2	28	16	57	0.216–1120	7
	62072	NWQL	SH1433	2.0	28	14	50	0.200–70.0	
Trinity River stream water									
Carbamazepine	62793	OGRL	[2]LCAB	0.005	35	35	100	0.054–0.219	71
	62793	NWQL	SH2080	0.06	35	5	14	0.019–0.067	
Sulfamethoxazole	62775	OGRL	[2]LCAB	0.005	35	35	100	0.017–0.754	51
	62021	NWQL	SH2080	0.1	35	0	0	--	
Trimethoprim	62023	NWQL	[2]SH2080	0.034	35	0	0	--	0
	62023	OGRL	LCAB	0.005	35	3	9	0.005–0.036	
Caffeine	50305	NWQL	[2]SH1433	0.06	35	26	74	0.050–0.760	51
	50305	NWQL	SH2080	0.06	35	4	11	0.012–0.132	

Table 3. Compounds analyzed by using more than one laboratory method in water samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

[LRL, laboratory reporting level; µg/L, micrograms per liter; LCAB, USGS Organic Geochemistry Research Laboratory liquid chromatography/mass spectrometry antibiotics schedule; SH, USGS National Water Quality Laboratory schedule; USGS, U.S. Geological Survey; OGRL, USGS Organic Geochemistry Research Laboratory in Lawrence, Kansas; NWQL, USGS National Water Quality Laboratory in Denver, Colorado; --, not applicable; β, beta]

Compound	Parameter code	Laboratory	Laboratory schedule	Minimum LRL (µg/L)	Total number of samples	Number of detections equal to or greater than the LRL	Detections equal to or greater than the LRL (percent)	Range of detected values (µg/L)	Quality-control check (percent)[1]
Trinity River stream water—Continued									
Cotinine	62005	NWQL	[2]SH2080	0.026	35	1	3	0.012–0.012	0
	62005	NWQL	SH1433	0.4	35	0	0	0.040–0.600	
3-β-Coprostanol	64512	NWQL	[2]SH2434	0.2	35	11	31	0.294–0.792	0
	62057	NWQL	SH1433	1.8	35	0	0	0.200–0.700	
Cholesterol	64514	NWQL	[2]SH2434	0.2	35	27	77	0.210–1.350	0
	62072	NWQL	SH1433	2.0	35	0	0	0.200–0.800	

[1]Frequency at which a detection reported by the preferred method with a concentration above the LRL of the non-preferred method was not reported as a detection by the non-preferred method.

[2]Preferred values.

Blank Samples

Blank samples are used to quantify contamination by compounds that could have been introduced into environmental samples because of sampling-related activities (USGS, variously dated). Blank samples consist of laboratory-grade organic-free water processed through equipment used for collecting and processing environmental samples before the collection of environmental water samples (Mueller and others, 1997). Blank samples used in this assessment were subjected to the same aspects of sample collection, field processing and preservation, transportation, and laboratory handling as the environmental samples. Nine equipment blank samples were analyzed for the same CECs that were targeted in the water samples collected at the three types of sites sampled during the study. Results were compiled without regard as to the type of site from which the sample was collected. There were no detections of human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, or sterols in field-blank samples. However, there were detections of CECs in some of the blank samples that were analyzed with the wastewater compound schedule, SH1433 (table 4). Generally, concentrations reported from blank samples were very low. For example, the maximum concentrations of 12 compounds measured in blank samples were less than the respective minimum LRLs for the study period.

Detections of compounds in field-blank samples that were equal to or greater than the minimum LRL for the study period could have possible implications regarding the concentrations of these compounds measured in environmental samples. For 10 compounds detected in laboratory-grade organic-free water, the censoring level was the minimum LRL that applied during the study period (table 4). The censoring level for six compounds (acetyl-hexamethyl-tetrahydronaphthalene [AHTN], *N*,*N*-diethyl-*m*-toluamide [DEET], isophorone, phenol, tributyl phosphate, and tris(2-chloroethyl)phosphate) was determined by multiplying the maximum concentration detected in a blank sample by five. This method of determining the censoring level was generally used when the maximum concentration detected in a blank sample exceeded the lowest LRL for the compound. The censoring level for 4-nonylphenol (sum of all isomers) and *d*-limonene was set to the maximum concentration detected in a blank sample.

Replicate Samples

Replicate samples were collected to identify variability in the analytical results that could have been introduced during sample collection, processing, and analysis. Relative percent differences (RPDs) were calculated for each replicate pair having detectable concentrations by using the following equation:

$$RPD = [|C_1 - C_2|/((C_1 + C_2)/2)] \times 100 \quad (1)$$

where

C_1 is the constituent concentration, in micrograms per liter, from the environmental sample; and

C_2 is the constituent concentration, in micrograms per liter, from the replicate sample.

Table 4. Detected compounds and range of concentrations in laboratory-grade organic-free water in nine blank samples analyzed in conjunction with water-quality sampling in or near Dallas, Texas, 2009–13.

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

Compounds that were detected in more than 30 percent of blanks						
Compound	Parameter code	Detections in blanks (percent)	LRL range during study period (µg/L)	Range of detected concentrations (µg/L)	Maximum blank detection (µg/L)	Selected censor level (µg/L)
4-Nonylphenol (sum of all isomers)	62085	33	1–2	0.1–2.0	2.00	2.00
Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)	62065	56	0.028–0.500	0.005–0.015	0.015	0.075
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	62075	67	0.052–0.500	0.012–0.041	0.041	0.052
Menthol	62080	44	0.32–0.40	0.02–0.10	0.100	0.320
1,4-Dichlorobenzene	34572	44	0.040–0.080	0.014–0.036	0.036	0.040
N,N-diethyl-*meta*-toluamide (DEET)	62082	89	0.06–0.14	0.01–0.14	0.140	0.700
Benzophenone	62067	44	0.08–0.12	0.03–0.07	0.070	0.080
Isophorone	34409	56	0.032–0.080	0.015–0.058	0.058	0.290

Compounds that were detected in less than 30 percent of blanks						
Compound	Parameter code	Detections in blanks (percent)	LRL range during study period (µg/L)	Range of detected concentrations (µg/L)	Maximum blank detection (µg/L)	Selected censor level (µg/L)
4-Nonylphenol diethoxylate	62083	11	5.0	3.0	3.00	5.00
Camphor	62070	11	0.044–0.100	0.014	0.014	0.044
d-Limonene	62073	11	0.08–0.14	0.27	0.270	0.270
Methyl salicylate	62081	11	0.044–0.100	0.009	0.009	0.044
Phenol	34466	22	0.16–1.4	0.15–0.21	0.210	1.05
Phenanthrene	34462	11	0.016–0.080	0.005	0.005	0.016
Tributyl phosphate	62089	22	0.16–0.20	0.02	0.020	0.100
Triphenyl phosphate	62092	11	0.12	0.01	0.010	0.120
Tris(2-chloroethyl)phosphate	62087	22	0.10	0.07–0.12	0.12	0.60
Tris(dichloroisopropyl)phosphate	62088	11	0.10–0.16	0.02	0.020	0.100

All RPDs were rounded to the nearest percent. RPDs were reported as zero percent when there was no difference between paired replicate concentrations if both values were equal to or greater than the LRL. RPDs were not reported if either of the paired replicate concentrations was less than the LRL or less than the selected censoring level for the compound (table 5).

For this study, five sequential replicates were collected for finished water, raw water, or treated-effluent water; five sequential replicates were collected for stream-water samples; and one sequential replicate was collected for bed-sediment samples. Replicate samples were collected immediately after their associated environmental samples. Each environmental and replicate sample was analyzed for as many as 120 different compounds. There were 65 pairings between compounds for the environmental and replicate samples across all study sites and dates (table 5). For 7 of the 65 paired samples, RPDs were zero; for 30 of the 65 paired samples, RPDs were greater than 10. Of the seven compounds with at least four calculated RPD values, tribromomethane had the lowest mean RPD (6), and sulfamethoxazole had the highest mean RPD (35). Sulfamethoxazole and 4-androstene-3,17-*dione* were the only two compounds with two or more RPDs greater than 30.

Table 5. Relative percent differences of compounds detected in 11 replicate samples collected at water-quality study sites in or near Dallas, Texas, 2009–13.

[--, compound measured in concentration below reporting level or not present; sed, bed-sediment sample; β, beta]

Compound	PWTP-2 finished water	PWTP-1 raw water	PWTP-1 raw water	PWTP-1 finished water	WWTP-2 treated-effluent water	Trinity River below Dallas	Trinity River Wilmer	Trinity River Wilmer (sed)	Trinity River Wilmer	Trinity River Rosser	Trinity River Rosser
	8/18/2009	11/17/2009	3/2/2010	3/2/2010	8/26/2010	12/11/2009	3/8/2011	3/8/2011	8/30/2011	8/27/2009	7/11/2011
Human-health pharmaceuticals											
Caffeine	--	--	--	--	--	0	8	--	--	0	--
Carbamazepine	--	15	--	--	6	1	2	--	14	123	--
Dehydronifedipine	--	--	--	--	--	--	0	--	--	--	--
Trimethoprim	--	--	--	--	--	17	77	--	--	--	--
Antibiotics											
Sulfamethoxazole	--	--	57	--	94	7	15	--	1	--	--
Steroidal hormones and sterols											
3-β-Coprostanol	--	--	--	--	9	12	8	17	--	--	--
4-Androstene-3,17-*dione*	23	--	--	--	36	--	45	--	--	--	--
Cholesterol	--	--	--	--	--	8	14	13	10	--	--
Estrone	--	--	--	--	--	5	1	--	--	--	--
Wastewater compounds											
4-*tert*-Octylphenol monoethoxylate	--	--	--	--	--	67	--	--	--	--	--
5-Methyl-1H-benzotriazole	--	--	--	--	--	0	--	--	22	--	--
Acetyl-hexamethyl-tetrahydronaphthalene	--	--	--	--	29	--	10	--	--	--	--
1,4-Dichlorobenzene	--	--	--	--	17	--	--	--	--	--	--
Cotinine	--	--	--	--	--	--	--	--	5	--	--
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	--	--	--	--	21	11	5	--	9	17	--
Benzophenone	--	--	--	--	22	--	0	--	7	--	--
Tribromomethane (Bromoform)	6	--	--	--	9	6	7	--	4	--	--
Tributyl phosphate	--	--	--	--	29	--	6	--	--	--	--
Triclosan	--	--	50	--	--	--	--	--	--	--	--
Triethyl citrate	--	--	--	--	6	0	0	--	--	--	--
Phenol	--	40	--	--	--	--	--	--	--	--	--
Tris(2-butoxyethyl)phosphate	--	--	--	--	--	40	--	--	--	--	--
Tris(2-chloroethyl)phosphate	--	--	--	--	--		11	--	--	--	--
Tris(dichloroisopropyl)phosphate	--	--	--	170	8	6	1	--	13	3	--

Matrix-Spike Samples

Environmental-matrix spikes are environmental samples with known concentrations of compounds added in the field; one of the uses of environmental-matrix spikes is the assessment of any effects that the sample matrix could have on the concentration measured for each of the added compounds. Other uses include the assessment of possible bias associated with analytical methods and the degradation of compounds in the sample during sample shipment and storage (Mueller and others, 1997). Environmental-matrix spikes were analyzed by schedule SH1433 for treated-effluent samples collected at WWTPs on November 23, 2009, and March 26, 2013. Results from two environmental-matrix spikes are likely not representative of all the matrices sampled in this project. On November 23, 2009, a split-spike sample (one portion of the sample was spiked in the field, and the other portion was spiked at the NWQL) was collected to determine possible changes in concentration between the time that the samples were collected in the field and the time they were processed and analyzed at the NWQL. Matrix-spike recovery percentages ranged from 67 percent (5-methyl-1H-benzotriazole) to 109 percent (4-*tert*-octylphenol diethoxylate) for the 18 compounds detected in the environmental sample spiked in the field on November 23, 2009 (table 6). In the environmental samples collected on November 23, 2009, and subsequently spiked at the NWQL, matrix-spike recoveries for the 18 detected compounds ranged from 66 percent (bromoform) to 125 percent (4-*tert*-octylphenol diethoxylate). RPDs for the 32 detected compounds ranged from zero percent (8 compounds) to 33 percent (indole) (table 7). There were 26 compounds with RPDs between zero and 10. There were six compounds with RPDs greater than 10; however, RPDs greater than 10 likely were caused by low compound concentrations and variability inherent in the method rather than a change in measured spike concentrations in the environmental sample between the field and the laboratory. For example, the RPD for indole was 33 with measured concentrations in the field- and laboratory-spiked environmental samples differing by only 0.04 µg/L (0.10 and 0.14 µg/L, respectively). There were 17 compounds with spike recoveries ranging from 21 percent (3-beta-coprostanol) to 104 percent (menthol) in the environmental field-spiked sample collected March 26, 2013 (table 6).

Surrogate Data

The NWQL adds surrogates to each environmental sample after filtration or to internal laboratory samples before sample processing to monitor sample-specific method performance. Each surrogate is closely related to a target compound but is not expected to be present in any environmental sample. There were 26 surrogates used in the study: 2 surrogates added to schedule SH2080; 4 added to schedule LCAB; 14 added to schedule SH2434; 3 added to schedule SH1433; and 3 added to schedule SH5433 (table 8). Surrogate recoveries for the antibiotics schedule (LCAB) were grouped together and not separated by sample type (Michael Meyer, USGS, written commun., 2015). Not all surrogates were added to each sample, and some surrogates were substituted during the project.

Generally, compounds in the SH2080 schedule had poor surrogate recoveries; however, the median recoveries for all compounds analyzed by the NWQL, including those in schedule SH2080, ranged from 44 (decafluorobiphenyl) to 101 percent (4-androstene-3,17-*dione*-2,2,4,6,6,16,16-*d*7) for PWTP samples (table 8). Median recoveries for compounds in the SH2434 and SH1433 schedules collected at WWTPs ranged from 47 percent (decafluorobiphenyl) to 115 percent (estriol-2,4,17-*d*3). Median recoveries for samples collected at sites on the Trinity River, including compounds in schedule SH5433, were generally lower than the other two site types (PWTPs and WWTPs) and ranged from 23 percent (decafluorobiphenyl) to 90 percent (Caffeine-13*C*). Median recoveries for the four surrogates in the LCAB schedule ranged from 83 percent (meclocycline) to 140 percent (naxidilic acid).

Statistical Analyses

SigmaPlot version 13 was used for statistical analyses (Systat Software, Inc., 2018). The Wilcoxon rank-sum test (Helsel and Hirsch, 2002) was used to compare concentrations of compounds detected in raw- and finished-water samples from PWTPs. At WWTPs, collection times for untreated-influent and treated-effluent water samples were based on estimated residence times to sample the same volume of water, which allows for statistical analyses of paired samples. Therefore, the Wilcoxon signed-rank test (Helsel and Hirsch, 2002) was used to compare concentrations of compounds detected in both untreated-influent and treated-effluent water samples from WWTPs. One-way analysis of variance (ANOVA) on ranks (Helsel and Hirsch, 2002) was used to compare detection frequencies and compound concentrations for samples collected at Trinity River main-stem sites. Dunn's multiple comparison tests (Systat Software, Inc., 2018) were used if ANOVA results were statistically significant. Results were considered statistically significant if the probability value (*p*-value) was less than or equal to 0.05 (Helsel and Hirsch, 2002).

Table 6. Spike report for treated-effluent water samples collected at water-quality study sites in or near Dallas, Texas, 2009–13.

[µg/L, micrograms per liter; NWQL, U.S. Geological Survey National Water Quality Laboratory; <, less than; --, no data/not determined; E, estimated]

Parameter code	Compound	November 23, 2009 @ 2221 (spiked in field)			November 23, 2009 @ 2220 (spiked at NWQL)			March 26, 2013 @ 2215 (spiked in field)		
		Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)	Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)	Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)
34572	1,4-Dichlorobenzene	0.78	0.13	76	0.73	0.13	68	0.47	0.1	43
62054	1-Methylnaphthalene	0.32	<0.022	--	0.33	<0.022	--	0.25	<0.022	--
62055	2,6-Dimethylnaphthalene	0.28	<0.06	--	0.28	<0.06	--	0.23	<0.06	--
62056	2-Methylnaphthalene	0.3	<0.036	--	0.3	<0.036	--	0.24	<0.036	--
62057	3-β-Coprostanol	E 2.6	<1.8	--	E 3.2	<1.8	--	E 1.3	E 0.58	21
62058	3-Methyl-1H-indole (Skatole)	0.33	<0.036	--	0.36	<0.036	--	0.22	<0.036	--
62060	4-Cumylphenol	0.43	<0.06	--	0.44	<0.06	--	0.26	<0.06	--
62061	4-n-Octylphenol	E 0.15	<0.16	--	E 0.16	<0.16	--	E 0.074	<0.06	--
62085	4-Nonylphenol (all isomers)	E 3.2	<2.0	--	E 3.4	<2.0	--	E 1.6	<2.0	45
62083	4-Nonylphenol diethoxylate	E 7.7	E 0.93	98	E 8.4	0.93	106	E 4.7	E 0.93	53
61705	4-*tert*-Octylphenol diethoxylate	E 0.58	E 0.11	109	E 0.66	0.11	125	E 0.28	<1.0	--
61706	4-*tert*-Octylphenol monoethoxylate	E 1.4	E 0.025	89	E 1.5	0.025	95	E 0.78	<1.0	--
62062	4-*tert*-Octylphenol	E 0.2	<0.14	--	E 0.21	<0.14	--	E 0.14	E 0.011	60
62063	5-Methyl-1H-benzotriazole	2.8	E 0.49	67	2.9	0.49	69	1.7	<1.2	--
62066	9, 10-Anthraquinone	E 0.44	<0.16	--	E 0.46	<0.16	--	E 0.38	<0.16	--
62064	Acetophenone	0.88	<0.4	--	0.94	<0.4	--	0.84	<0.4	--
62065	Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)	0.58	0.15	100	0.6	0.15	103	0.42	0.12	70
34421	Anthracene	0.18	<0.028	--	0.2	<0.028	--	0.11	<0.01	--
34248	Benzo[*a*]pyrene	0.094	<0.05	--	0.15	<0.05	--	0.081	<0.06	--
62067	Benzophenone	0.96	0.12	98	1.0	0.12	100	0.88	0.17	82
62068	*β*-Sitosterol	E 4.7	<4.0	--	E 5.8	<4.0	--	E 1.7	<4.0	--
62086	*β*-Stigmastanol	E 1.4	<2.6	--	1.8	<2.6	--	E 1.4	<2.6	--
40290	Bromacil	1.7	<0.36	--	1.9	<0.36	--	--	--	--
50305	Caffeine	0.87	<0.06	--	0.94	<0.06	--	0.8	<0.06	--
62070	Camphor	0.81	<0.044	--	0.86	<0.044	--	0.77	<0.044	--
62071	Carbazole	0.2	<0.03	--	0.22	<0.03	--	0.17	<0.03	--
38933	Chlorpyrifos	2.8	<0.16	--	3.0	<0.16	--	2.6	<0.16	--
62072	Cholesterol	E 2.5	<2.0	--	E 3	<2.0	--	E 2.1	<2.0	--
62005	Cotinine	E 0.71	<0.038	--	E 0.84	<0.038	--	E 0.4	<0.038	--

Table 6. Spike report for treated-effluent water samples collected at water-quality study sites in or near Dallas, Texas, 2009–13.—Continued

[µg/L, micrograms per liter; NWQL, U.S. Geological Survey National Water Quality Laboratory; <, less than; --, no data/not determined; E, estimated]

Parameter code	Compound	November 23, 2009 @ 2221 (spiked in field)			November 23, 2009 @ 2220 (spiked at NWQL)			March 26, 2013 @ 2215 (spiked in field)		
		Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)	Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)	Spike (µg/L)	Environmental sample concentration (µg/L)	Recovery (percent)
39572	Diazinon	2.6	<0.16	--	3.5	<0.16	--	1.2	<0.16	--
62073	*d*-Limonene	1	<0.08	--	1.1	<0.08	--	0.46	<0.08	--
34377	Fluoranthene	0.2	<0.024	--	0.21	<0.024	--	0.16	<0.024	--
62075	Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	1.8	1.40	93	1.8	1.4	91	1.4	1.4	93
62076	Indole	0.1	<0.08	--	0.14	<0.08	--	0.048	<0.08	--
62077	Isoborneol	0.85	<0.18	--	0.91	<0.18	--	E 0.96	<0.18	--
34409	Isophorone	0.48	0.1	88	0.52	0.1	96	0.5	0.15	81
62078	Isopropylbenzene	0.31	<0.3	--	0.31	<0.3	--	0.13	<0.3	--
62079	Isoquinoline	0.36	<0.046	--	0.43	<0.046	--	0.22	<0.046	--
62080	Menthol	3.3	E 0.098	93	3.5	0.098	97	E 3.6	E 0.098	104
50359	Metalaxyl	1.8	<0.12	--	2.0	<0.12	--	1.5	<0.12	--
62081	Methyl salicylate	0.84	E 0.01	96	0.9	0.01	101	0.76	E 0.01	--
39415	Metolachlor	0.43	<0.08	--	0.45	<0.08	--	0.36	<0.028	--
62082	*N*,*N*-diethyl-*meta*-toluamide (DEET)	0.5	0.09	95	0.52	0.09	98	0.39	0.09	76
34443	Naphthalene	0.18	<0.04	--	0.18	<0.04	--	0.13	<0.04	--
62084	*p*-Cresol	0.85	<0.08	--	0.88	<0.08	--	0.64	<0.08	--
34462	Phenanthrene	0.19	<0.032	--	0.2	<0.032	--	0.17	<0.016	--
34466	Phenol	0.85	<0.16	--	0.93	<0.16	--	0.78	<0.16	--
40370	Prometon	1.7	<0.12	--	1.9	<0.12	--	--	--	--
34470	Pyrene	0.2	<0.042	--	0.21	<0.042	--	0.16	<0.042	--
34476	Tetrachloroethylene	E 0.54	<0.12	--	E 0.57	<0.12	--	E 0.38	<0.12	--
34288	Tribromomethane (Bromoform)	2	0.84	67	2	0.84	66	1.7	0.89	47
62089	Tributyl phosphate	0.56	E 0.15	95	0.60	E 0.15	103	0.45	E 0.15	85
62090	Triclosan	3.4	<0.2	--	3.4	<0.2	--	2.4	<0.2	--
62091	Triethyl citrate	0.59	E 0.14	105	0.63	E 0.14	112	0.54	E 0.14	70
62092	Triphenyl phosphate	0.84	E 0.059	91	0.9	E 0.059	96	0.8	E 0.059	81
62093	Tris(2-butoxyethyl)phosphate	6.3	<0.8	--	6.9	<0.8	--	5.5	<0.8	--
62087	Tris(2-chloroethyl)phosphate	1.8	0.21	92	2.0	0.21	102	1.7	0.21	84
62088	Tris(dichloroisopropyl)phosphate	3.9	0.37	103	4.2	0.37	109	3.6	0.37	86

Table 7. Relative percent differences for field and laboratory spikes of treated-effluent water samples collected November 23, 2009, at WWTP-2 near Dallas, Texas.

[µg/L, micrograms per liter; RPD, relative percent difference; E, estimated value]

Compound	Field spike value (µg/L)	Laboratory spike value (µg/L)	RPD
1-Methylnaphthalene	0.32	0.33	3
2,6-Dimethylnaphthalene	0.28	0.28	0
2-Methylnaphthalene	0.3	0.3	0
3-*β*-Coprostanol	E 2.6	E 3.2	21
4-Cumylphenol	0.43	0.44	2
4-Nonylphenol (sum of all isomers)	E 3	E 3	0
4-Nonylphenol diethoxylate	E 7.7	E 8.4	9
β-Stigmastanol	E 1.4	E 1.8	25
Camphor	0.81	0.86	6
Carbaryl	E 0.64	E 0.65	2
Carbazole	0.2	0.22	10
Chlorpyrifos	2.8	3.0	7
Cholesterol	E 2.5	E 3	18
Diazinon	2.6	3.5	30
d-Limonene	1	1	10
Fluoranthene	0.2	0.2	5
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	1.8	1.8	0
Indole	0.1	0.14	33
Isoborneol	0.85	0.91	7
Isophorone	0.48	0.52	8
Isopropylbenzene	0.31	0.31	0
Isoquinoline	0.40	0.40	0
Menthol	3.3	3.5	6
Metalaxyl	1.8	2.0	11
Metolachlor	0.43	0.45	5
N,*N*-diethyl-*meta*-toluamide (DEET)	0.5	0.52	4
Naphthalene	0.18	0.18	0
p-Cresol	0.85	0.88	3
Tetrachloroethylene	E 0.54	E 0.57	5
Triclosan	3.4	3.4	0
Triethyl citrate	0.59	0.63	7
Tris(dichloroisopropyl)phosphate	3.9	4.2	7

Table 8. Surrogate percent recoveries for environmental samples collected at study sites in or near Dallas, Texas, 2009–13.

[pcode, parameter code; SH, U.S. Geological Survey (USGS) National Water Quality Laboratory schedule; LCAB, USGS Organic Geochemistry Research Laboratory liquid chromatography/mass spectrometry antibiotics schedule; --, no data; N/A, parameter code not assigned for surrogate standard; α, alpha; β, beta]

Surrogate (isotope dilution standard)	pcode	Potable water treatment plants					Wastewater treatment plants					Trinity River main-stem study sites				
		Number	Minimum (percent)	Mean (percent)	Median (percent)	Maximum (percent)	Number	Minimum (percent)	Mean (percent)	Median (percent)	Maximum (percent)	Number	Minimum (percent)	Mean (percent)	Median (percent)	Maximum (percent)
Human health and pharmaceuticals (SH2080)																
Carbamazepine-*d*10	90797	42	22	52	53	70	28	9	21	19	39	35	27	38	39	50
Ethyl nicotinate-*d*4	99571	42	38	69	59	93	28	21	40	40	62	35	44	61	62	72
Antibiotics (LCAB)																
Combined dataset																
Meclocycline	62680	137	2	83	83	204										
Naxidilic acid	N/A	149	32	220	140	1,300										
Oleandomycin	62964	92	52	120	120	260										
13*C*6-sulfamethazine	N/A	139	4	130	130	270										
Steroidal hormones and sterols (SH2434)																
16-Epiestriol-2,4-*d*2	91676	20	63	76	76	87	19	41	76	80	99	25	24	65	72	89
17-*α*-Ethynylestradiol-2,4,16,16-*d*4	90813	42	69	84	82	109	28	51	81	84	104	35	32	80	83	90
17-β-Estradiol-13,14,15,16,17,18-13*C*6	91753	30	69	79	78	94	24	51	79	83	94	30	31	78	78	96
17-β-Estradiol-2,4,16,16-*d*4	90777	12	78	86	85	94	4	70	82	84	91	5	80	82	82	84
4-Androstene-3,17-dione-2,2,4,6,6,16,16-*d*7	90815	12	58	99	101	128	4	55	96	102	122	5	49	76	77	92
Bisphenol A-*d*16	67308	42	65	91	92	123	28	60	95	100	108	35	26	82	86	103
Cholesterol-25,26,26,26,27,27,27-*d*7	90778	42	62	75	75	90	28	49	72	73	88	35	15	73	74	91
Dihydrotestosterone-1,2,4,5*a*-*d*4	90823	12	81	91	90	107	4	59	83	85	105	5	80	85	84	92
Estriol-2,4,17-*d*3	90819	12	74	95	94	118	4	114	116	115	120	5	76	84	86	88
Estrone-2,4,16,16-*d*4	90820	12	64	89	94	118	4	60	87	88	110	5	68	79	81	84
Mestranol-2,4,16,16-*d*4	90821	42	70	80	80	92	28	55	79	82	98	35	30	81	83	92
Progesterone-2,2,4,6,6,17*a*,21,21,21-*d*9	90822	12	6	63	72	126	4	22	89	102	128	5	31	42	41	55
Testosterone-2,2,4,6,6-*d*5	90824	12	70	92	94	109	4	77	99	104	111	5	58	81	83	92
trans-Diethyl-1,1,1',1'-*d*4-stilbestrol-3,3',5,5'-*d*4	90817	42	30	61	61	90	28	57	80	81	100	35	19	52	50	80
Wastewater compounds in the water column (SH1433)																
Caffeine-13*C*	99584	42	76	91	93	104	28	56	95	94	104	35	75	90	90	112
Fluoranthene-*d*10	99586	42	74	84	85	98	28	48	87	88	123	35	66	82	82	103
Decafluorobiphenyl	99585	42	24	44	44	70	28	16	48	47	69	35	30	45	45	67
Wastewater compounds in bed sediments (SH5433)																
Fluoranthene-*d*10	90738	--	--	--	--	--	--	--	--	--	--	35	48	75	72	101
Bisphenol A-*d*3	90735	--	--	--	--	--	--	--	--	--	--	35	2	21	46	45
Decafluorobiphenyl	90737	--	--	--	--	--	--	--	--	--	--	35	5	21	23	36

*Data in LCAB section combined from potable water treatment plant, wastewater treatment plant, and Trinity River sample sites.

Detections, Concentrations, and Distributions of Compounds of Emerging Concern

Analytical results were sorted by study site type (PWTPs, WWTPs, and Trinity River sites) and are presented in Null and others (2019). Results are grouped primarily by compound type and subdivided on the basis of the laboratory schedules used for analysis; compounds are listed in the following order: human-health pharmaceuticals (schedule SH2080); antibiotics (schedule LCAB); steroidal hormones, stanols, and sterols (schedule SH2434); wastewater compounds in water (schedule SH1433); and wastewater compounds in bed sediment at Trinity River sites (schedule SH5433).

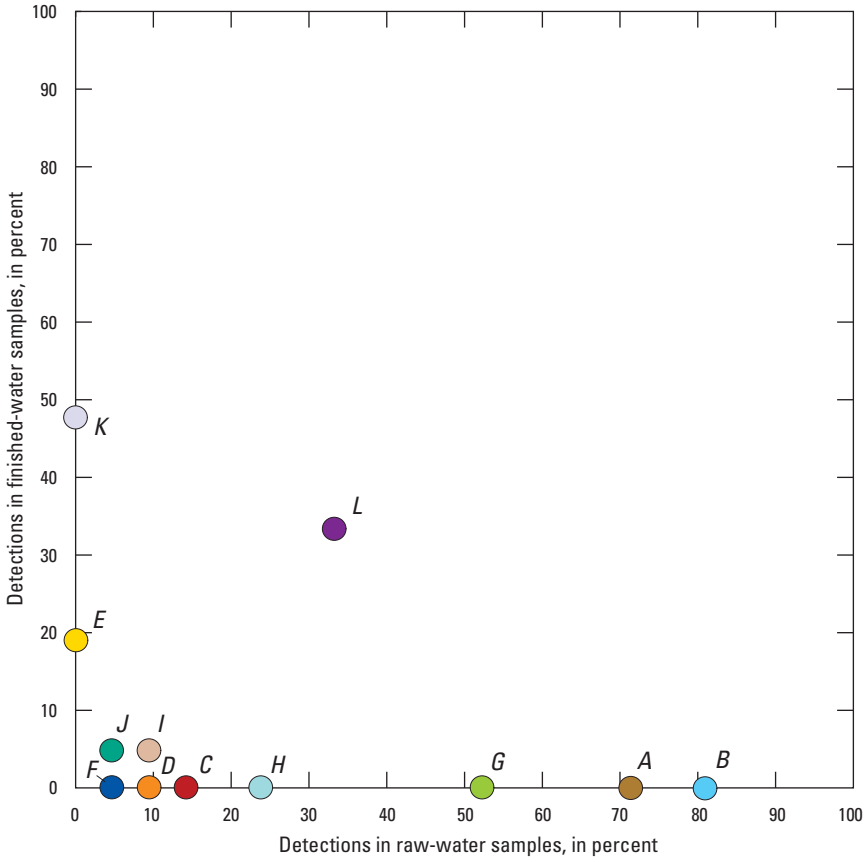
Compounds of Emerging Concern in Water Samples Collected at Potable Water Treatment Plants

Compound Detections

Of the 120 targeted compounds, 16 were detected in samples collected at PWTPs; 14 compounds were detected in raw-water samples, and 5 compounds were detected in finished-water samples (tribromomethane, 4-androstene-3,17-*dione*, benzophenone, methyl salicylate, and tris(dichloroisopropyl)phosphate) (fig. 2). Eleven CECs were detected exclusively in raw-water samples, indicating that they were removed or degraded to compounds that were not analyzed. Of those 11 CECs, 3 were human-health pharmaceutical compounds (carbamazepine, sulfamethoxazole, and caffeine); 3 were steroidal hormones/stanols/sterols (estrone, 3-beta-coprostanol, and cholesterol); a detergent (4-*tert*-octylphenol); a flavor/fragrance (hexahydro-hexamethyl-cyclopenta-benzopyran [HHCB]); an industrial wastewater compound (*p*-cresol); a PAH (pyrene), and a flame retardant/plasticizer (tributyl phosphate). Two of the five CECs detected in finished-water samples (the androgen, 4-androstene-3,17-*dione*, and tribromomethane, which is a byproduct of water treatment processes) were detected exclusively in finished-water samples. Tris(dichloroisopropyl)phosphate, benzophenone, and methyl salicylate were detected in raw-water and finished-water samples; however, benzophenone and methyl salicylate were detected in only one finished-water sample. Twelve CECs were detected less frequently in finished-water samples than in raw-water samples. Tris(dichloroisopropyl) phosphate, a persistent plasticizer/flame retardant (Hudec and others, 1981) and EDC (Wang and others, 2013), was detected seven times in each sample type (33 percent of samples). Stackelberg and others (2004) detected methyl salicylate and tris(dichloroisopropyl)phosphate in finished-water samples collected at a PWTP. Carbamazepine, sulfamethoxazole, and tris(2-butoxyethyl)phosphate were detected in finished-water samples by Stackelberg and others (2004) but were not detected in finished-water samples in this study. Tribromomethane and 4-androstene-3,17-*dione* were detected in finished-water samples but not in raw-water samples (fig. 2). Tribromomethane, also known as bromoform, is a trihalomethane disinfection byproduct formed during the water treatment process by the reaction between disinfectants (chlorine or ozone) and organic matter and bromide (U.S. Department of Health and Human Services, 2005). Tribromomethane was detected in 48 percent of finished-water samples and in at least one finished-water sample from each of the three PWTPs. Three compounds were detected in more than 50 percent of the raw-water samples: sulfamethoxazole (81 percent of samples), carbamazepine (71 percent of samples), and cholesterol (52 percent of samples). Reif and others (2012) found sulfamethoxazole, carbamazepine, and caffeine to be the most frequently detected pharmaceutical compounds in samples collected at surface-water sites within 5 miles of PWTP raw-water intakes in Pennsylvania. In a nationwide study, Valder and others (2014) detected cholesterol in 19 percent of samples. The most common personal-use products detected in the Valder and others study (2014) was the fragrance compound HHCB, which was detected in 30 percent of samples. In this study, HHCB was detected in 24 percent of raw-water samples but was not detected in any finished-water sample. The study by Valder and others (2014) and this study used a similar censoring level for HHCB (table 4).

Compound Concentrations

Concentrations of CECs detected in finished-water samples at PWTPs were generally very low and less than concentrations of CECs detected in raw-water samples. In some cases, detected concentrations were in the range of the variable LRLs. The treatment processes used by the PWTPs could have reduced concentrations (and detections) to levels less than the LRL or selected censoring level. In addition, CECs could have been transformed during water-treatment processes into degradates that were not analyzed. For example, the three CECs most prevalent in raw-water samples, sulfamethoxazole, carbamazepine, and cholesterol, were not detected in finished-water samples (fig. 2). Maximum concentrations of sulfamethoxazole, carbamazepine, and cholesterol in raw-water samples were 0.22, 0.02, and 0.46 µg/L, respectively. In a similar study of surface waters in the San Antonio River Basin near San Antonio, Texas, maximum concentrations of 0.27 and 1.74 µg/L were reported for sulfamethoxazole and cholesterol, respectively (Opsahl and Lambert, 2013). Eight CECs that were detected in less than 25 percent of raw-water samples were not detected in any finished-water samples (fig. 2). Although five compounds were detected in finished-water samples, only three of these five compounds were detected in more than 10 percent of finished-water samples: tribromomethane



EXPLANATION

A		**Carbamazepine**
B		**Sulfamethoxazole**
C		**Caffeine**
D		**Estrone**
E		**4-Androstene-3,17-*dione***
F		**3-beta-Coprostanol**
G		**Cholesterol**
F		**4-*tert*-Octylphenol**
H		**Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)**
I		**Benzophenone**
J		**Methyl salicylate**
F		***p*-Cresol**
K		**Tribromomethane (Bromoform)**
F		**Pyrene**
C		**Tributyl phosphate**
L		**Tris(dichloroisopropyl)phosphate**

Note: One symbol might represent more than one compound

Figure 2. Detection frequencies of selected compounds in raw- and finished-water samples collected at potable water treatment plants in or near Dallas, Texas, 2009–13.

(48 percent of samples), tris(dichloroisopropyl)phosphate (33 percent of samples), and 4-androstene-3,17-*dione* (19 percent of samples) (Null and others, 2019). The maximum concentration of tribromomethane was 0.50 µg/L, which is similar to the maximum concentration (0.67 µg/L) reported by Opsahl and Lambert (2013) from river sites downstream from treated-effluent discharges from WWTPs. The maximum concentration of tris(dichloroisopropyl) phosphate was 0.35 µg/L. The maximum concentration of the male steroidal hormone, 4-androstene-3,17-*dione*, was 0.003 µg/L. The maximum concentration for this compound was also 0.003 µg/L in the San Antonio study (Opsahl and Lambert, 2013). Three compounds were detected in raw- and finished-water samples: tris(dichloroisopropyl)phosphate, benzophenone, and methyl salicylate. The paired statistical analyses of these compounds between raw- and finished-water samples were not statistically significant (table 9) likely because of the high number of values that were less than the LRLs.

Compounds of Emerging Concern in Water Samples Collected at Wastewater Treatment Plants

Generally, detection frequencies for CECs in treated-effluent samples from WWTPs were substantially lower compared to detection frequencies for CECs in untreated-influent samples from WWTPs. Of the 120 targeted CECs, 74 were detected in water samples collected at WWTPs (Null and others, 2019). Seventy-three CECs were detected in untreated-influent samples, and 31 CECs were detected in all 14 untreated-influent samples. Forty-seven compounds were detected in at least 70 percent of the untreated-influent samples. A total of 30 compounds were detected in treated-effluent samples, 5 of which were detected in all treated-effluent samples: carbamazepine, AHTN, HHCB, 1,4-dichlorobenzene, and tris(dichloroisopropyl)phosphate. Forty-four CECs were detected exclusively in untreated-influent samples, indicating that they were removed or degraded to compounds that were not analyzed. Twenty-nine CECs (including carbamazepine, sulfamethoxazole, 4-androstene-3,17-*dione*, 3-beta-coprostanol, AHTN, HHCB, 1,4-dichlorobenzene, tribromomethane, benzophenone, and tris(dichloroisopropyl)phosphate) were detected in untreated and treated water, indicating that treatment processes likely did not remove or degrade these compounds. Wilcoxon signed-rank test results indicated concentrations decreased significantly during wastewater treatment processes for 22 CECs detected in untreated-influent and treated-effluent samples collected at WWTPs (8 human-health pharmaceutical and antibiotic compounds, 3 steroidal hormones, 2 stanol/sterol compounds, 2 detergent compounds, and various CECs from other classes of compounds, such as HHCB, DEET, 1,4-dichlorobenzene, *p*-cresol, benzophenone, triethyl citrate, and tributyl phosphate). Concentrations of carbamazepine and tribromomethane increased significantly between samples types at WWTPs. Tribromomethane was the only compound detected substantially more frequently in treated-effluent samples (93-percent detection frequency) than untreated-influent samples (29-percent detection frequency). The estrogen compound equilin was the only compound detected in a treated-effluent sample that was not also detected in at least one untreated-influent sample. Equilin was detected in one treated-effluent sample, which corresponds to a 7-percent detection frequency.

Human-Health Pharmaceuticals and Antibiotics

Eighteen of the 44 targeted human-health pharmaceuticals and antibiotic compounds were detected in untreated-influent samples (fig. 3). Five pharmaceutical compounds (carbamazepine, 1,7-dimethylxanthine, acetaminophen, caffeine, and cotinine) were detected in all untreated-influent samples with maximum concentrations of 0.29, 8.8, 56, 64, and 0.32 µg/L, respectively (fig. 3; Null and others, 2019). One pharmaceutical compound (sulfamethoxazole) and four antibiotic compounds (ciprofloxacin, ofloxacin, anhydroerythromycin, and trimethoprim) were detected in untreated-influent samples at detection frequencies ranging from 78 to 93 percent. The maximum concentration of sulfamethoxazole was 2.75 µg/L. Ibuprofen, azithromycin, tetracycline, and lincomycin were detected in 57 to 72 percent of untreated-influent samples (fig. 3; Null and others, 2019).

Table 9. Results of Wilcoxon rank-sum tests for selected compounds detected in water samples collected at potable water treatment plants in or near Dallas, Texas, 2009–13.

[µg/L, micrograms per liter; ≤, less than or equal to; <, less than]

Compound	Parameter code	Raw-water median (µg/L)	Finished-water median (µg/L)	Statistically significant at p≤0.05	*p*-value
Tris(dichloroisopropyl)phosphate	62088	<0.10	<0.10	No	0.945
Benzophenone	62067	<0.08	<0.08	No	0.750
Methyl salicylate	62081	<0.044	<0.044	No	1

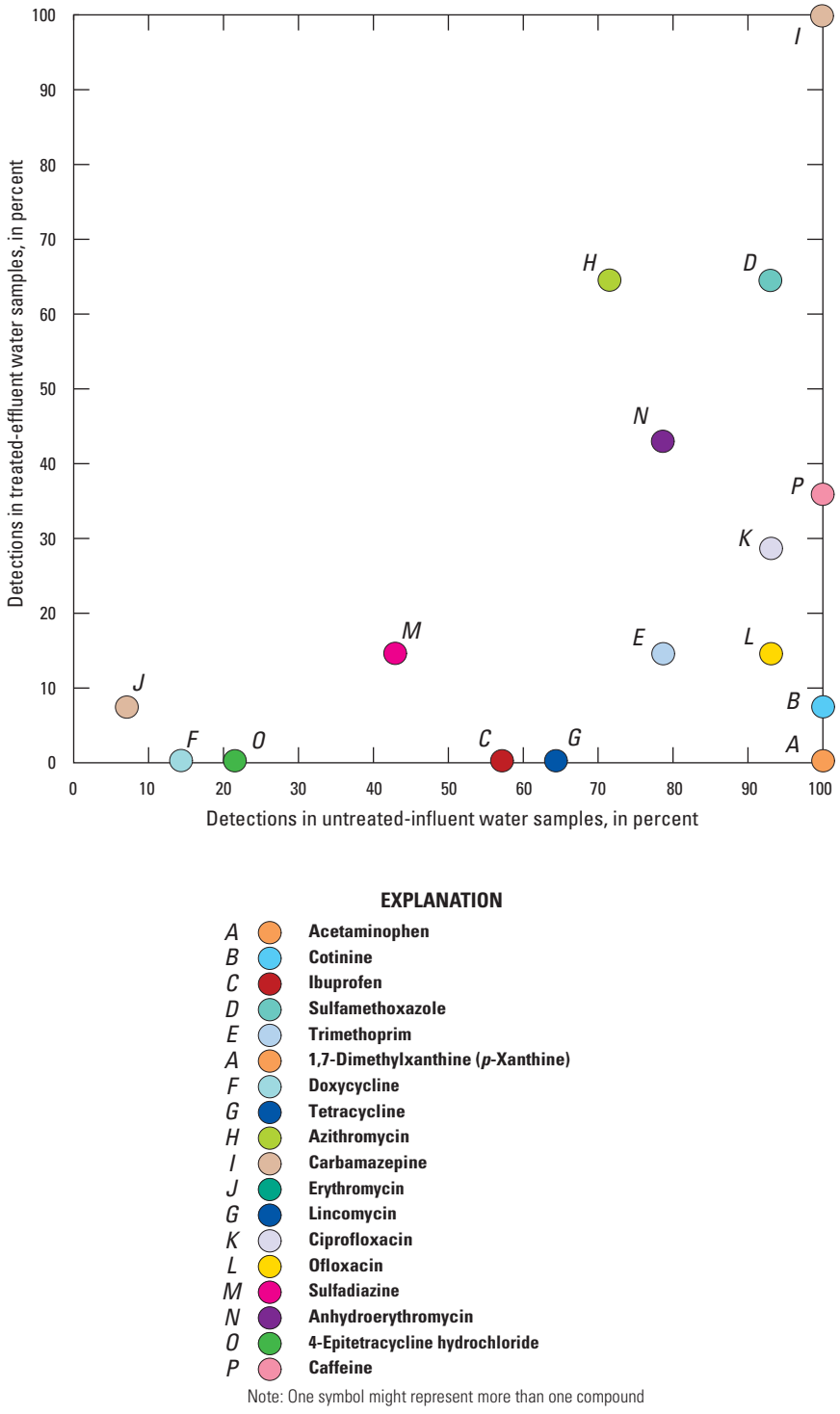


Figure 3. Detection frequencies of human-health pharmaceuticals and antibiotic compounds in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

Of the 44 targeted CECs in the human-health pharmaceuticals and antibiotics group, 11 were detected in treated-effluent samples. Sulfamethoxazole and azithromycin were detected in 64 percent of samples. Carbamazepine was the only CEC in the human-health pharmaceuticals and antibiotic compounds group that was detected in all untreated-influent and treated-effluent samples. The occurrence of sulfamethoxazole and carbamazepine in untreated-influent and treated-effluent samples is consistent with low amount of removals reported by Kasprzyk-Hordern and others (2009). The maximum concentration of carbamazepine was 0.38 µg/L, which was measured in a treated-effluent sample (Null and others, 2019). In contrast to the maximum carbamazepine concentration of 0.38 µg/L measured in this study, Opsahl and Lambert (2013) reported a maximum concentration of 0.12 µg/L for carbamazepine in stream-water samples in the San Antonio River Basin.

Nine CECs were detected in at least 70 percent of untreated-influent samples (fig. 3). Eight of these nine CECs that were detected in at least 70 percent of untreated-influent samples were also detected in more than 10 percent of treated-effluent samples (fig. 4). Cotinine is not shown on figure 4 because although it was detected in all untreated-influent samples, it was detected in less than 10 percent of treated-effluent samples. Median concentrations for the nine compounds detected in at least 70 percent of untreated-influent samples were significantly lower in treated-effluent samples than in untreated-influent samples (*p*-values ranged from less than 0.001 to 0.042; table 10). The only exception was the difference in carbamazepine concentrations measured in untreated-influent and treated-effluent samples. The median concentration of carbamazepine was significantly higher in treated-effluent samples (0.20 µg/L) than in untreated-influent samples (0.18 µg/L) (*p*-value equals 0.011) (Null and others, 2019). Increases in concentrations of carbamazepine after wastewater treatment processes could be caused by hydrolysis of compound conjugates and were also reported by Kasprzyk-Hordern and others (2009). The largest difference between median concentrations in untreated influent and treated effluent was observed for caffeine. A median concentration of 30.0 µg/L was measured for caffeine in untreated-influent samples compared to a median concentration of less than 0.08 µg/L in treated-effluent samples.

Acetaminophen and 1,7-dimethylxanthine (*p*-xanthine) were detected in all untreated-influent samples but were not detected in treated-effluent samples. Cotinine was also detected in all untreated-influent samples but was only detected in one treated-effluent sample. It is likely acetaminophen, 1,7-dimethylxanthine (*p*-xanthine), and cotinine were removed or degraded by the wastewater treatment processes. The effectiveness of a WWTP in removing or degrading CECs in untreated influent arriving

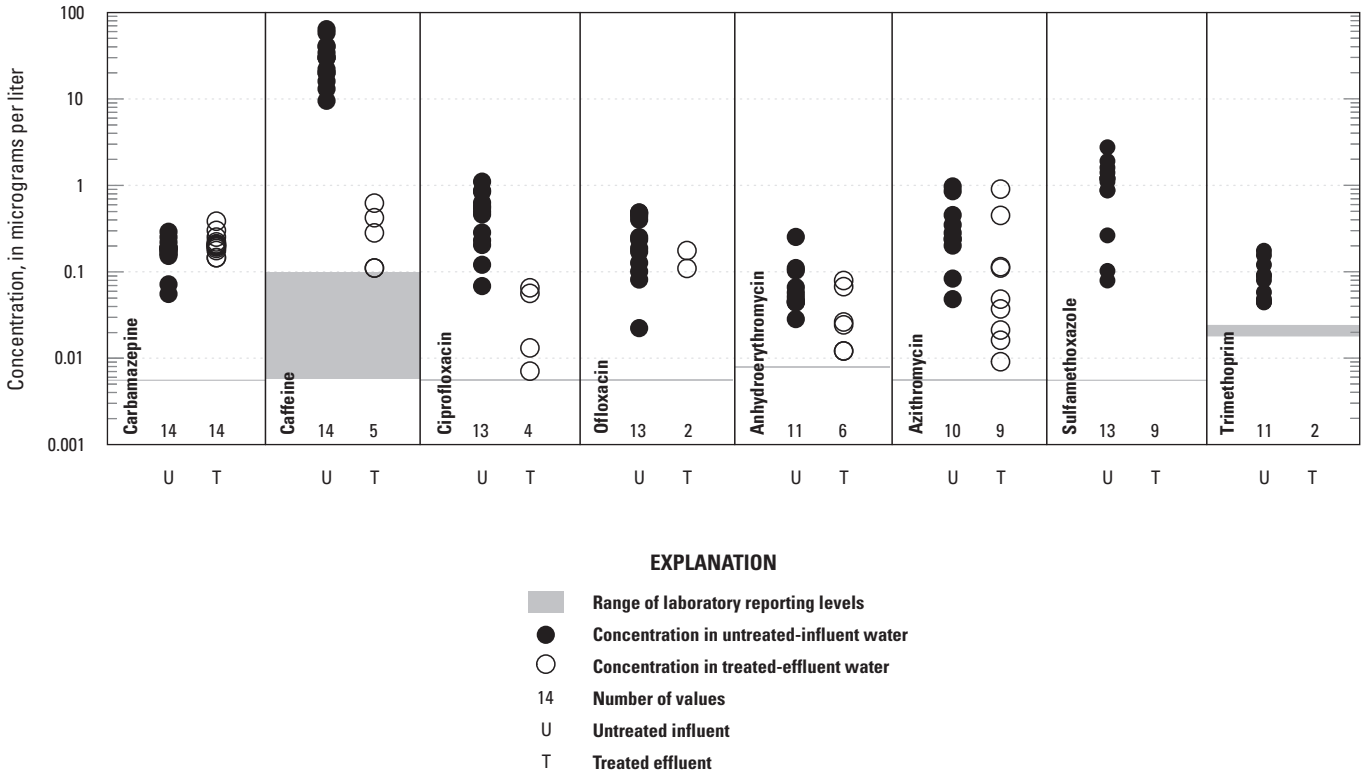


Figure 4. Concentrations of selected human-health pharmaceuticals and antibiotics in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

Table 10. Results of Wilcoxon signed-rank tests for selected human-health pharmaceuticals and antibiotic compounds detected in water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

[µg/L, micrograms per liter; ≤, less than or equal to; <, less than]

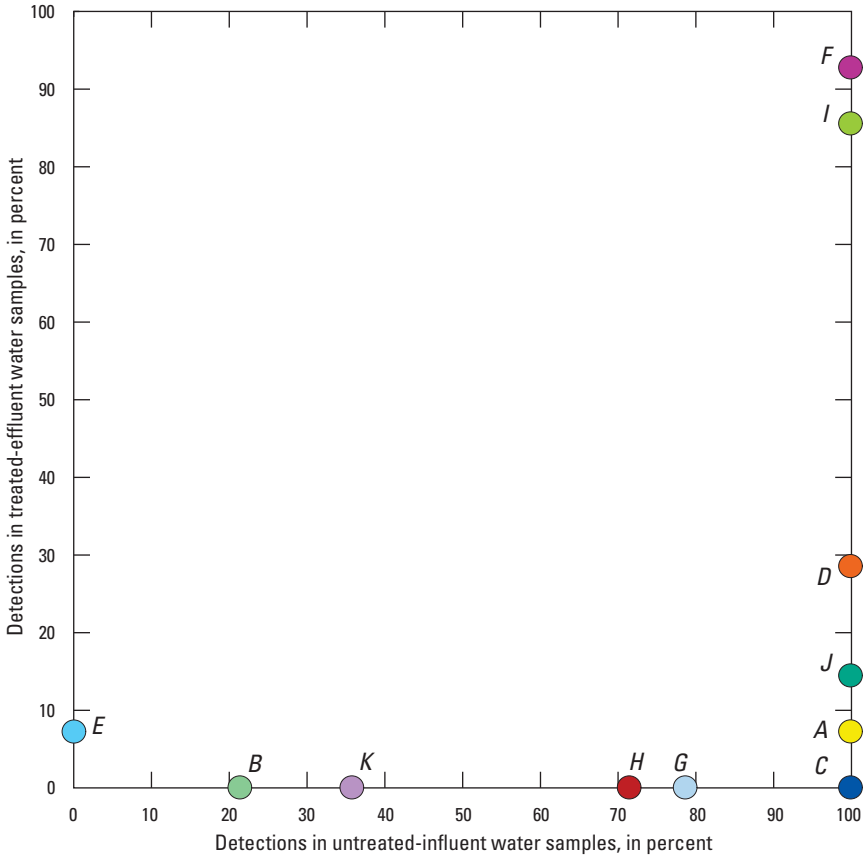
Compound	Parameter code	Untreated-influent median (µg/L)	Treated-effluent median (µg/L)	Increase or decrease from untreated- to treated-water	Statistically significant at p≤0.05	*p*-value
Carbamazepine	62793	0.183	0.201	Increase	Yes	0.011
Sulfamethoxazole	62775	1.15	0.011	Decrease	Yes	<0.001
Caffeine	50305	30.0	<0.08	Decrease	Yes	<0.001
Cotinine	62005	0.211	<0.038	Decrease	Yes	<0.001
Ciprofloxacin	62898	0.494	<0.005	Decrease	Yes	<0.001
Ofloxacin	62899	0.234	<0.005	Decrease	Yes	0.002
Anhydroerythromycin	63674	0.046	<0.008	Decrease	Yes	0.003
Azithromycin	62792	0.236	0.019	Decrease	Yes	0.042
Trimethoprim	62023	0.053	<0.034	Decrease	Yes	0.010

at the plant depends on several factors including which compounds are present in the source water, type and mode of operation of each treatment process, and physiochemical characteristics of the compounds themselves, some of which change over time (Stackelberg and others, 2007).

Steroidal Hormones, Stanols, and Sterols

Fifteen of the 21 targeted steroidal hormone/stanol/sterol compounds were detected in untreated-influent samples (fig. 5). Thirteen of these 15 detected compounds were in more than 70 percent of the untreated-influent samples. Two steroidal hormone compounds were detected in more than 10 percent of the treated-effluent samples—the androgen compound 4-androstene-3,17-*dione* (detected in 93 percent of the samples) and the estrogen compound estrone (detected in 29 percent of the samples). Maximum concentrations for these androgen and estrogen compounds were 0.10 and 0.08 µg/L in untreated-influent samples and 0.003 and 0.03 µg/L in treated-effluent samples, respectively (Null and others, 2019). The naturally occurring steroidal hormone compound 4-androstene-3-17-*dione* has been identified as the androgen most commonly present in treated wastewater effluent samples (Phillips and others, 2012). Opsahl and Lambert (2013) reported maximum concentrations of 0.003 and 0.005 µg/L for these androgen and estrogen compounds, respectively, in stream-water samples from locations downstream from WWTP discharges. Concentrations reported by Opsahl and Lambert (2013) were likely lower than those reported in this study because samples were collected at the point of treated-effluent discharge in this study rather than at a downstream stream-water site.

Fewer steroidal hormone compounds were detected in treated-effluent samples (4 detections) than in untreated-influent samples (11 detections), which indicates that the wastewater treatment processes effectively removed most steroidal hormone compounds or transformed them into degradates that were not analyzed. Three of the 11 CECs detected in untreated-influent water were also detected in treated-effluent water, indicating that 73 percent of detected compounds in this group were removed or degraded by wastewater treatment processes. Statistical comparisons of steroidal hormone compound concentrations for untreated-influent and treated-effluent samples show a significant decrease in concentrations between sample types for three steroidal hormones (17-beta-estradiol, estrone, and 4-androstene-3,17-*dione*) (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11). Median concentrations of 4-androstene-3,17-*dione* in untreated-influent and treated-effluent water samples were 0.07 and 0.002 µg/L, respectively. Median concentrations of estrone in untreated-influent and treated-effluent water samples were 0.04 and less than 0.0008 µg/L, respectively. Concentrations of 4-androstene-3,17-*dione* and estrone in untreated-influent samples were generally 10 or more times higher than those in the treated-effluent samples, and median concentrations of both steroidal hormones and of the estrogen compound 17-beta-estradiol decreased significantly after the wastewater was treated (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11).



EXPLANATION

A		**17-beta-Estradiol**
B		**Equilenin**
C		**Estriol**
D		**Estrone**
E		**Equilin**
F		**4-Androstene-3,17-*dione***
C		***cis*-Androsterone**
G		**Epitestosterone**
C		**Testosterone**
C		**11-Ketotestosterone**
C		**Dihydrotestosterone**
H		**Progesterone**
I		**3-beta-Coprostanol**
J		**Cholesterol**
K		**beta-Stigmastanol**
H		**beta-Sitosterol**

Note: One symbol might represent more than one compound

Figure 5. Detection frequencies of steroidal hormones, stanols, sterols, and other wastewater compounds measured in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

Table 11. Results of Wilcoxon signed-rank tests for selected hormones, stanols, sterols, and wastewater compounds detected in water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

[µg/L, micrograms per liter; ≤, less than or equal to; <, less than; β, beta]

Compound	Parameter code	Untreated-influent median		Treated-effluent median		Increase or decrease from untreated to treated water	Statistically significant at p≤0.05	*p*-value
Steroidal hormones								
17-β-Estradiol	64510	0.01	µg/L	<0.0008	µg/L	Decrease	Yes	<0.001
Estrone	64521	0.04	µg/L	<0.0008	µg/L	Decrease	Yes	<0.001
4-Androstene-3,17-*dione*	64513	0.07	µg/L	0.002	µg/L	Decrease	Yes	<0.001
Stanols and sterols								
3-*β*-Coprostanol	64512	42	µg/L	0.70	µg/L	Decrease	Yes	<0.001
Cholesterol	64514	77	µg/L	<2.0	µg/L	Decrease	Yes	<0.001
Detergents and detergent metabolites								
4-Nonylphenol (sum of all isomers)	62085	5.0	µg/L	<2.0	µg/L	Decrease	Yes	<0.001
4-*tert*-Octylphenol monoethoxylate	61706	1.25	µg/L	<1.0	µg/L	Decrease	Yes	0.002
Personal-use products (flavors and fragrances)								
Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)	62065	0.18	µg/L	0.14	µg/L	Decrease	No	0.094
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	62075	1.90	µg/L	1.25	µg/L	Decrease	Yes	0.005
Pesticides and repellents								
N,*N*-diethyl-*meta*-toluamide (DEET)	62082	0.93	µg/L	<0.70	µg/L	Decrease	Yes	0.008
1,4-Dichlorobenzene	34572	0.90	µg/L	0.12	µg/L	Decrease	Yes	<0.001
Industrial wastewater compounds								
p-Cresol	62084	27.0	µg/L	<0.08	µg/L	Decrease	Yes	<0.001
Tribromomethane (Bromoform)	34288	<0.10	µg/L	0.86	µg/L	Increase	Yes	<0.001
Benzophenone	62067	0.95	µg/L	0.16	µg/L	Decrease	Yes	<0.001
Triethyl citrate (ethyl citrate)	62091	0.76	µg/L	0.25	µg/L	Decrease	Yes	<0.001
Flame retardants and plasticizers								
Tris(dichloroisopropyl)phosphate	62088	0.44	µg/L	0.49	µg/L	Increase	No	0.626
Tributyl phosphate	62089	0.28	µg/L	0.15	µg/L	Decrease	Yes	<0.001

Two of the four stanol/sterol compounds (3-beta-coprostanol and cholesterol) were detected in all untreated-influent samples (fig. 5). The compounds 3-beta-coprostanol and cholesterol were also detected frequently in a nationwide study where they were found in 31 and 77 percent of stream-water samples, respectively (Valder and others, 2014). In this study, 3-beta-coprostanol and cholesterol were detected in 86 and 14 percent of treated-effluent samples with maximum concentrations of 7.94 and 9.41 µg/L, respectively (Null and others, 2019). Maximum concentrations of 3-beta-coprostanol and cholesterol in the San Antonio River Basin were 0.42 and 1.74 µg/L, respectively (Opsahl and Lambert, 2013). Treatment processes of the WWTPs could have had a pronounced effect on concentrations of the four stanols/sterols. The other two targeted stanols/sterols, beta-sitosterol and beta-stigmastanol, were detected in 71 and 36 percent of untreated-influent samples, respectively, and were not detected in treated-effluent samples. Median concentrations of 3-beta-coprostanol in untreated-influent and treated-effluent samples were 42 and 0.70 µg/L, respectively (fig. 6; table 11). Median concentrations of cholesterol in untreated-influent and treated-effluent samples were 77 µg/L and less than 2.0 µg/L, respectively (table 11). Differences in median concentrations of 3-beta-coprostanol and cholesterol in untreated-influent samples compared to the median concentrations of these compounds in treated-effluent samples were statistically significant (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11).

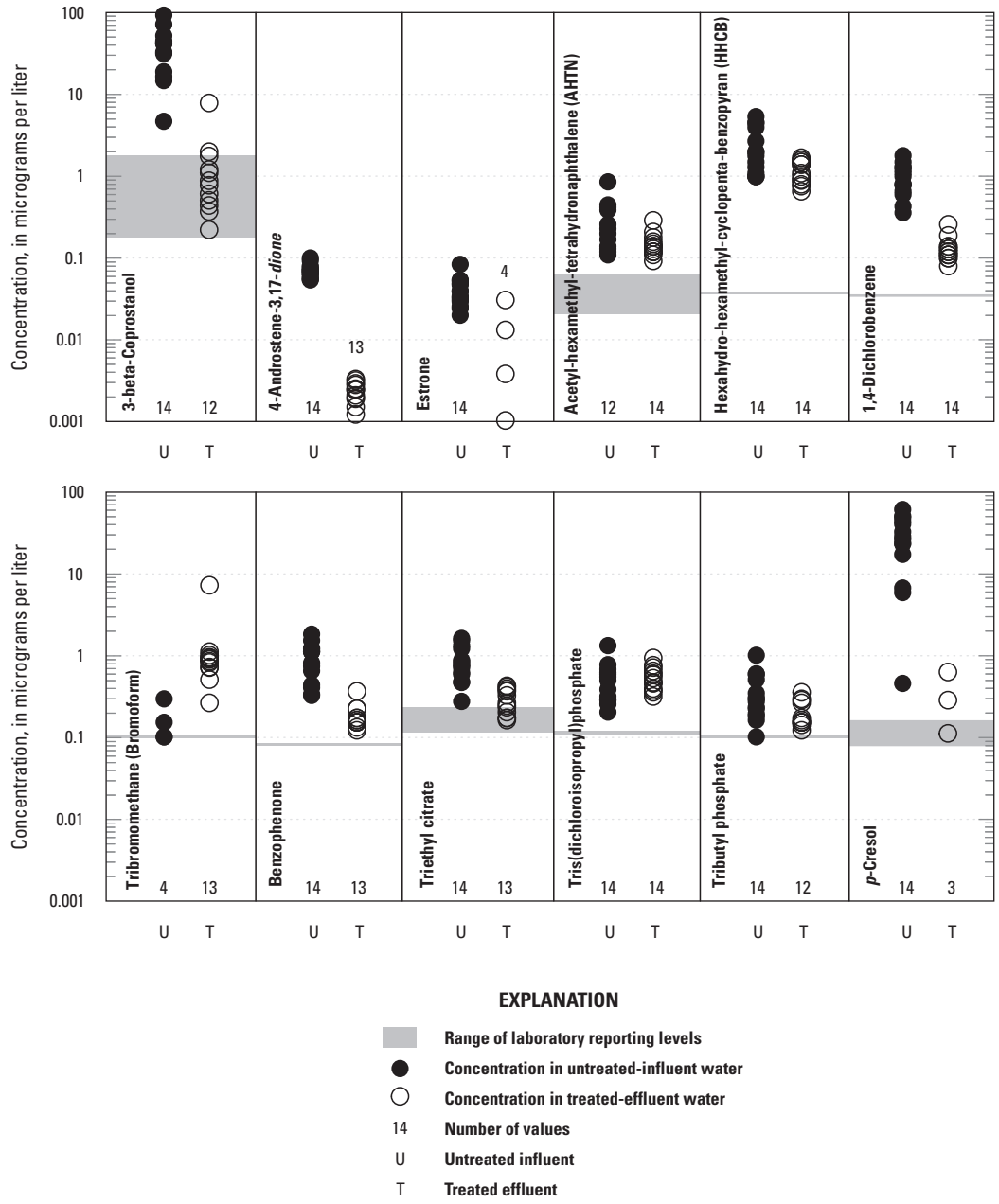


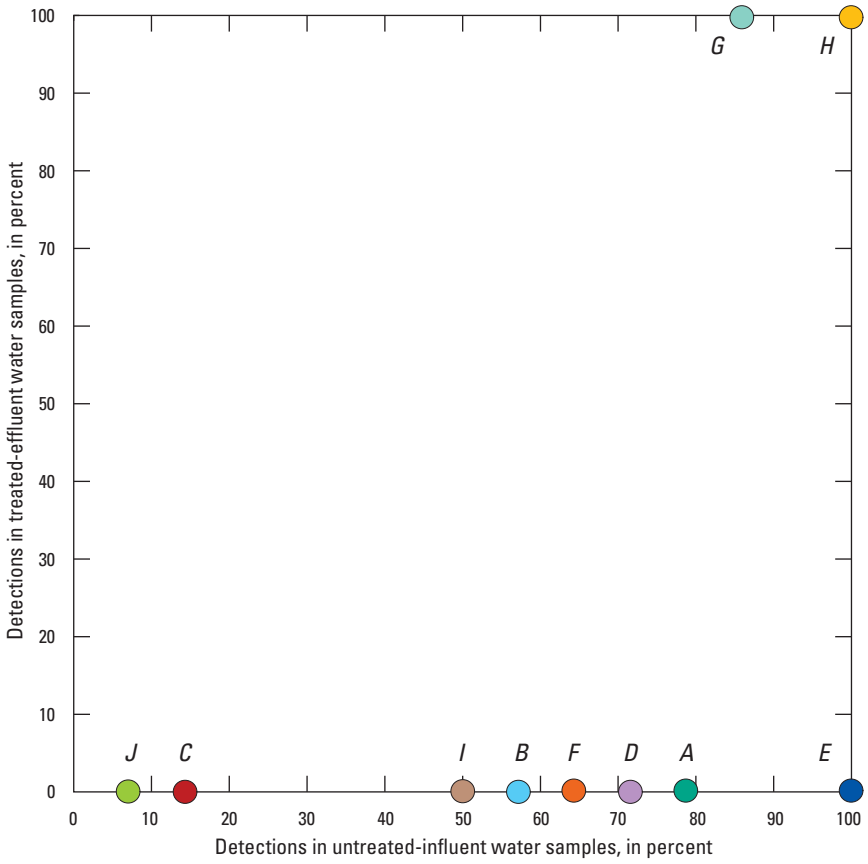
Figure 6. Concentrations of steroidal hormones, stanols, sterols, and wastewater compounds detected in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

Detergents, Personal-Use Products, and Pesticides

Eighteen of the 27 detergents, personal-use products (flavors and fragrances), and pesticide compounds were detected at least once in an untreated-influent sample (fig. 7). Of these 18 compounds, 11 were detected in at least 70 percent of samples. HHCB, AHTN, and 1,4-dichlorobenzene were detected in all treated-effluent samples, with maximum concentrations of 1.7, 0.29, and 0.26 µg/L, respectively (Null and others, 2019). Opsahl and Lambert (2013) reported a maximum concentration for HHCB in the San Antonio River Basin of 1.9 µg/L. Five of the seven compounds in the detergents group were detected in untreated-influent samples (fig. 7). Three compounds from the detergents group (4-*tert*-octylphenol monoethoxylate, 4-nonylphenol (sum of all isomers), and 4-*tert*-octylphenol) were detected in at least 70 percent of the untreated-influent samples; 4-*tert*-octylphenol, a non-ionic detergent metabolite (Morace, 2012), was detected in each untreated-influent sample. No compounds from the detergents group were detected in samples from treated-effluent discharge, indicating that they were removed or degraded by the wastewater treatment processes.

Nine of the 10 compounds categorized as personal-use products were detected in at least 1 untreated-influent sample (fig. 7). Eight of the nine detected personal-use compounds were detected in at least 64 percent of the untreated-influent samples. The compounds HHCB, menthol, 3-methyl-1H-indole (skatole), camphor, *d*-limonene, and isoborneol were detected in each untreated-influent sample. Only two of the nine compounds detected in untreated-influent samples (HHCB and AHTN) were also detected in treated-effluent samples, indicating that 78 percent of the personal-use products were removed or degraded. AHTN was detected in 86 percent of untreated-influent samples and in 100 percent of treated-effluent samples. The higher number of detections in treated-effluent samples could be an artifact of laboratory uncertainty associated with the analyses and could be attributed to the similar concentrations of this compound between the two sample types. Median concentrations of AHTN were similar between untreated-influent and treated-effluent samples (0.18 and 0.14 µg/L, respectively; fig. 6; table 11), indicating that the wastewater treatment process likely does not affect this compound (*p*-value equals 0.094, Wilcoxon signed-rank test; table 11). This pattern is consistent with findings by Phillips and others (2012). Median concentrations of HHCB in untreated-influent and treated-effluent samples were 1.90 and 1.25 µg/L, respectively. The reduction of median concentrations of HHCB between untreated-influent and treated-effluent water samples was statistically significant (*p*-value equals 0.005, Wilcoxon signed-rank test; table 11); however, another study (Stackelberg and others, 2004) found consistent HHCB concentrations throughout the treatment process, indicating little or no removal of HHCB was likely through conventional water treatment processes.

Four of the 10 targeted pesticide/repellent compounds (prometon, bromacil, DEET, and 1,4-dichlorobenzene) were detected in untreated-influent samples (fig. 7). 1,4-Dichlorobenzene was detected in all untreated-influent and treated-effluent samples—1,4-dichlorobenzene was the only pesticide compound detected in at least 60 percent of untreated-influent samples (maximum concentration of 1.8 µg/L) and was the only pesticide compound detected in treated-effluent samples (75 percent of pesticide compounds removed/degraded) (Null and others, 2019). DEET, a common insect repellent, has been found in many other studies at high frequencies and was detected in 57 percent of the untreated-influent water samples at concentrations greater than or equal to 0.70 µg/L. Although the LRL for DEET in this study was 0.06 µg/L, concentrations of less than 0.700 µg/L were censored (reported as not detected) based on DEET concentrations measured in blank samples (table 4). The maximum concentration of DEET in untreated-influent samples was 5.7 µg/L (Null and others, 2019). DEET was not detected at concentrations greater than or equal to 0.70 µg/L in any of the treated-effluent samples (fig. 7). Although 1,4-dichlorobenzene was detected in all untreated-influent and treated-effluent samples, the median concentration of 0.12 µg/L in treated-effluent samples was significantly less than the median concentration of 0.90 µg/L in untreated-influent samples (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11; fig. 6). The herbicides prometon and bromacil were detected in 14 and 7 percent of untreated-influent samples, respectively, but were not detected in treated-effluent samples (fig. 7).



EXPLANATION

Detergents and detergent metabolites

- A — 4-Nonylphenol (sum of all isomers)
- B — 4-Nonylphenol diethoxylate
- C — 4-*tert*-Octylphenol diethoxylate
- D — 4-*tert*-Octylphenol monoethoxylate
- E — 4-*tert*-Octylphenol

Personal-use products (flavors and fragrances)

- E — Camphor
- E — 3-Methyl-1H-indole (Skatole)
- F — Acetophenone
- G — Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)
- H — Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)
- I — Indole
- E — Isoborneol
- E — *d*-Limonene
- E — Menthol

Pesticides and repellents

- B — *N,N*-diethyl-*meta*-toluamide (DEET)
- H — 1,4-Dichlorobenzene
- J — Bromacil
- C — Prometon

Note: One symbol might represent more than one compound

Figure 7. Detection frequencies of detergents and detergent metabolites, personal-use products (flavors and fragrances), and pesticides and repellents in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

Industrial Wastewater and Disinfection Compounds and Polycyclic Aromatic Hydrocarbons

Sixteen of the 22 industrial wastewater, disinfection, and PAHs compounds were detected in untreated-influent samples (fig. 8). Of these 16 compounds, 8 were detected in at least 75 percent of the untreated-influent samples. Nine of the 10 industrial wastewater compounds were detected in untreated-influent samples. Four of these nine compounds—benzophenone, triethyl citrate, *p*-cresol, and methyl salicylate—were detected in all untreated-influent samples. Three compounds (tribromomethane, benzophenone, and triethyl citrate) were detected in 93 percent of treated-effluent samples. A statistically significant decrease in the median concentration of benzophenone from 0.95 µg/L in untreated-influent samples to 0.16 µg/L in treated-effluent samples was observed (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11). A statistically significant decrease in the median concentration of triethyl citrate was also observed, from 0.76 µg/L in untreated-influent samples to 0.25 µg/L in treated-effluent samples (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11). The median concentration of the disinfection byproduct tribromomethane increased in a statistically significant manner from less than 0.10 µg/L in untreated-influent samples to 0.86 µg/L in treated-effluent samples (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11; fig. 6). Detection frequencies of tribromomethane also increased between untreated-influent samples and treated-effluent samples. Increases in detection frequencies and concentrations of tribromomethane are likely a result of wastewater treatment processes, specifically, chlorination.

The disinfection compounds triclosan and phenol were detected in 100 and 93 percent of untreated-influent samples, respectively (fig. 8). Neither disinfection compound was detected in any of the treated-effluent samples, indicating that the compounds were removed or degraded. In all samples, concentrations of triclosan and phenol ranged from less than 0.20 to 5.6 µg/L and less than 0.16 to 19 µg/L, respectively (Null and others, 2019).

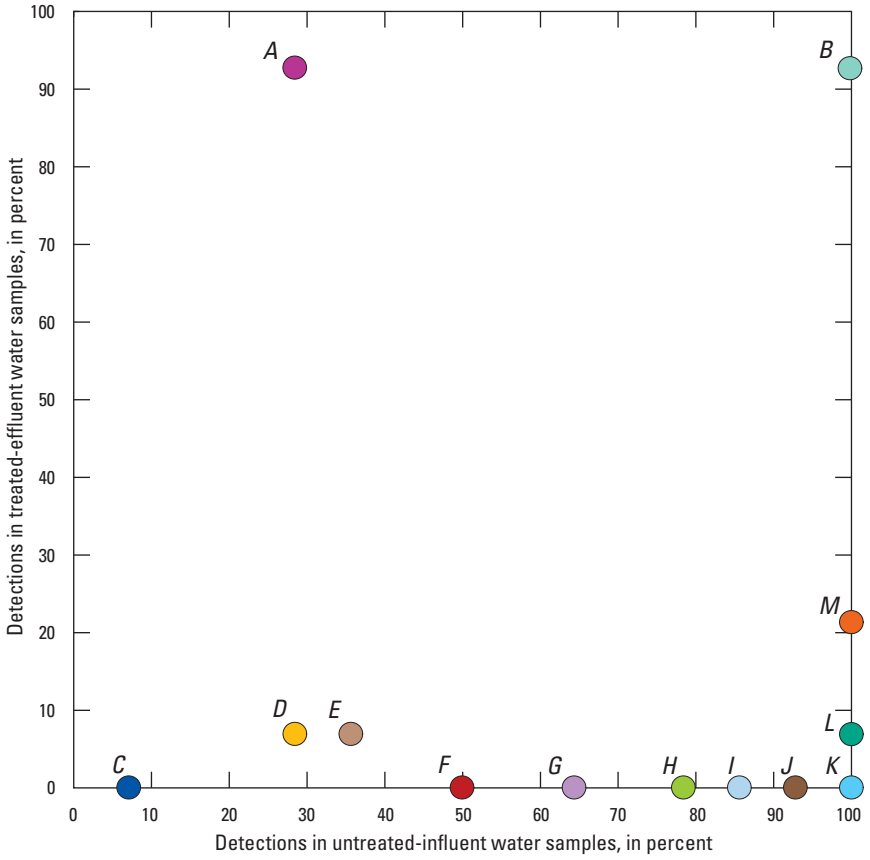
Five of the 10 PAHs were detected in untreated-influent samples (fig. 8). Two PAHs, naphthalene and 1-methylnaphthalene, were detected in at least 75 percent of untreated-influent samples, with maximum concentrations of 1.00 and 0.16 µg/L, respectively (Null and others, 2019). There were no detections of PAHs in treated-effluent samples. The wastewater treatment process possibly degraded or removed these compounds.

Flame Retardants and Plasticizers

All six flame retardant and plasticizer compounds were detected in untreated-influent samples (fig. 9), and three of these compounds—the flame retardants tris(2-butoxyethyl) phosphate and tris(dichloroisopropyl)phosphate and the plasticizer tributyl phosphate—were detected in 100 percent of untreated-influent samples (50 percent of all flame retardants and plasticizers removed/degraded). Maximum concentrations of tributyl phosphate, tris(2-butoxyethyl)phosphate, and tris(dichloroisopropyl)phosphate in untreated-influent samples were 0.99, 51.0, and 1.3 µg/L, respectively (Null and others, 2019). BPA, a common plasticizer (Ferrell, 2009), was detected in 93 percent of untreated-influent samples but was not detected in treated-effluent samples. Maximum concentrations of BPA were 1.01 and less than 0.20 µg/L (less than the LRL), in untreated-influent and treated-effluent samples, respectively (Null and others, 2019). Kasprzyk-Hordern and others (2009) detected BPA in all untreated-influent samples and 75 percent of treated-effluent samples. They reported that mean concentrations of BPA decreased 79 percent between the two sample types. Tris(2-butoxyethyl) phosphate was detected in 1 of 15 treated-effluent samples, a detection frequency of about 7 percent. Two compounds, tributyl phosphate and tris(dichloroisopropyl)phosphate, were detected in more than 87 percent of treated-effluent samples, with detections of tris(dichloroisopropyl)phosphate observed in all treated-effluent samples. Median concentrations of tributyl phosphate decreased significantly between untreated-influent (0.28 µg/L) and treated-effluent (0.15 µg/L) samples (*p*-value less than 0.001, Wilcoxon signed-rank test; table 11), which is similar to the pattern observed by Phillips and others (2012). The wastewater treatment process did not appear to affect the concentrations of tris(dichloroisopropyl)phosphate appreciably; the median concentration for this compound increased slightly from 0.44 µg/L in untreated-influent samples to 0.49 µg/L in treated-effluent samples, an increase that was not statistically significant (*p*-value equals 0.626, Wilcoxon signed-rank test; table 11).

Observations Regarding Compounds of Emerging Concern Detected in Water Samples Collected at Potable Water and Wastewater Treatment Plants

The analytical results for water samples collected at PWTPs and WWTPs indicate a decrease in the frequency of detections of most CECs in treated samples compared to untreated samples. At PWTPs, 11 CECs were reduced to concentrations less than their respective LRLs by the treatment process or were transformed into degradates that were not analyzed. Compounds most commonly detected in raw-water samples at PWTPs include sulfamethoxazole, carbamazepine, cholesterol, tris(dichloroisopropyl) phosphate, and HHCB (fig. 2). Of these compounds, only tris(dichloroisopropyl)phosphate was detected in finished-water samples. At WWTPs, 44 CECs were removed or degraded during water-treatment processes—acetaminophen, testosterone, isoborneol, and triclosan are a few of the compounds that were each detected in every untreated-influent sample but were not detected in any treated-effluent



EXPLANATION

Industrial wastewater compounds

- *A* Tribromomethane (Bromoform)
- *B* Triethyl citrate (ethyl citrate)
- *B* Benzophenone
- *C* Isopropylbenzene
- *D* 5-Methyl-1H-benzotriazole
- *E* Isophorone
- *F* Tetrachloroethene
- *L* Methyl salicylate
- *M* *p*-Cresol

Disinfection compounds

- *J* Phenol
- *K* Triclosan

Polycyclic aromatic hydrocarbons

- *C* Phenanthrene
- *C* 2,6-Dimethylnaphthalene
- *G* 2-Methylnaphthalene
- *H* 1-Methylnaphthalene
- *I* Naphthalene

Note: One symbol might represent more than one compound

Figure 8. Detection frequencies of industrial wastewater and disinfection compounds and polycyclic aromatic hydrocarbons in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

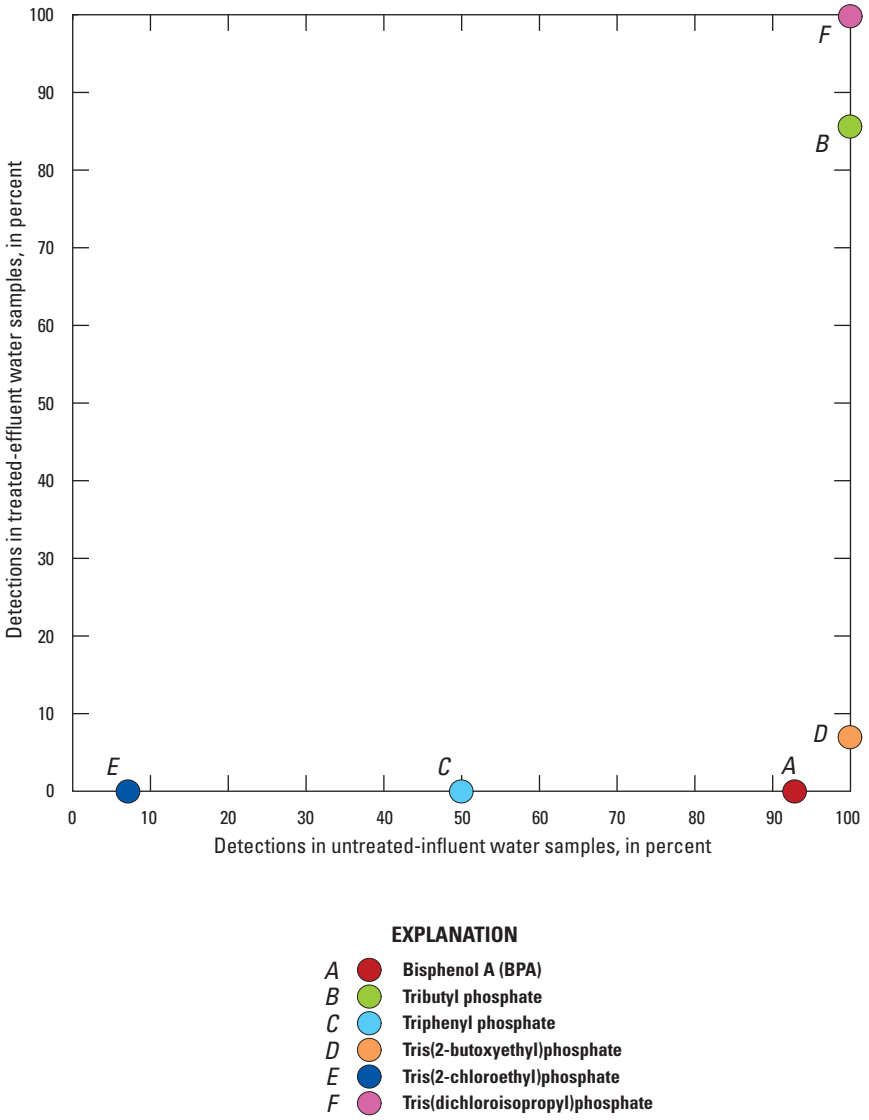


Figure 9. Detection frequencies of flame retardants and plasticizers in untreated-influent and treated-effluent water samples collected at wastewater treatment plants in or near Dallas, Texas, 2009–13.

samples. Some compounds, however, were not affected by water treatment processes. For example, methyl salicylate (PWTPs) (fig. 2) and erythromycin, carbamazepine, HHCB, and 1,4-dichlorobenzene (WWTPs) were detected at the same frequency in untreated samples as they were in treated samples (figs. 3 and 7). At a given type of treatment plant, the detection frequency in untreated and treated samples of the flame retardant/plasticizer tris(dichloroisopropyl) phosphate was similar; tris(dichloroisopropyl)phosphate was detected in 100 percent of the untreated-influent and treated-effluent samples at WWTPs, and in about 30 percent of the raw-water and finished-water samples collected at PWTPs (Null and others, 2019). At PWTPs, 4-androstene-3,17-*dione* and tribromomethane were detected more frequently in finished-water samples than in raw-water samples; at WWTPs, equilin, AHTN, and tribromomethane were detected more frequently in treated-effluent samples than in untreated-influent samples. Of these four compounds (4-androstene-3,17-*dione*, tribromomethane, equilin, and AHTN), only tribromomethane was detected in substantially more treated-effluent samples than in untreated-influent samples, likely because it is a byproduct of the treatment process at each type of water treatment plant.

Statistically significant differences were not observed in the median concentrations of CECs measured in raw-water and finished-water samples collected at PWTPs (table 9). A statistically significant decrease in the median concentration was measured for 22 compounds in treated-effluent

samples compared to the median concentration measured in untreated-influent samples collected at WWTPs (tables 10 and 11). Median concentrations of carbamazepine and tribromomethane increased significantly between untreated-influent and treated-effluent samples (tables 10 and 11).

Compounds of Emerging Concern in Stream-Water and Bed-Sediment Samples Collected at Study Sites on the Trinity River

Results for stream-water samples and bed-sediment samples collected at the five USGS water-quality monitoring stations on the Trinity River are presented in Null and others (2019). Comparisons of compounds detected in stream-water samples to compounds detected in bed-sediment samples were limited to selected stanols/sterols and wastewater compounds listed in laboratory schedules SH1433 and SH5433 (Null and others, 2019).

Compounds of Emerging Concern in Stream-Water Samples

Twenty-three of the 120 targeted CECs were detected in stream-water samples collected at the 5 study sites on the main stem of the Trinity River (Null and others, 2019). Ten of the 23 CECs were detected in samples collected at all 5 sites—carbamazepine, sulfamethoxazole, caffeine, 3-beta-coprostanol, cholesterol, HHCB, benzophenone, triethyl citrate, tributyl phosphate, and tris(dichloroisopropyl) phosphate—indicating their persistence in the environment. Tris(dichloroisopropyl)phosphate, a flame retardant (Stackelberg and others, 2004), was the only compound detected in all stream-water samples. Carbamazepine and sulfamethoxazole were detected in 97 percent of stream-water samples, and HHCB was detected in 94 percent of stream-water samples. Nine compounds were detected in more than half of the stream-water samples. In general, detection frequencies decreased with distance downstream; however, the decrease was not statistically significant (*p*-value equals 0.314, one-way ANOVA on ranks). Median concentrations of nine compounds detected at all five Trinity River sites decreased between the most upstream main-stem site closest to where WWTP effluent is discharged and the farthest downstream site on the Trinity River, Trinity River Trinidad; the concentration of tributyl phosphate was similar at the most upstream and farthest downstream main-stem sites.

Nine of the 44 human-health pharmaceutical and antibiotic compounds were detected in stream-water samples (fig. 10). All nine of the human-health pharmaceutical and antibiotic compounds detected in stream-water samples collected at Trinity River sites were also detected in treated-effluent samples at WWTPs, and four of these nine compounds were detected in more than 40 percent of treated-effluent samples at WWTPs (fig. 3). Carbamazepine, caffeine, and sulfamethoxazole were among the most frequently detected human-health pharmaceutical and antibiotic compounds in samples collected at main-stem sites on the Trinity River (fig. 10). In a similar study by Reif and others (2012), these three compounds were also detected frequently. Median concentrations of these three compounds did not change significantly between Trinity River main-stem sites (*p*-value greater than 0.596 for each compound, one-way ANOVA on ranks; Null and others, 2019). Carbamazepine and sulfamethoxazole, which were frequently detected in raw-water samples at PWTPs and in both sample types at WWTPs, were each detected in 34 of 35 stream-water samples (97 percent of samples). Caffeine was detected in 25 of 35 stream-water samples (71 percent of samples) and was detected in at least 4 of 7 samples at each study site. Five antibiotic compounds (ciprofloxacin, ofloxacin, anhydroerythromycin, azithromycin, and sulfamethoxazole) were detected in samples collected at the upstream site on the Trinity River (Trinity River Highway 310 site). In addition to sulfamethoxazole, anhydroerythromycin was the only other antibiotic compound detected in more than 15 percent of all stream-water samples (Null and others, 2019).

Two of the 17 steroidal hormone CECs were detected in stream-water samples—estrone and 4-androstene-3,17-*dione* (fig. 10), and these two compounds were detected in less than 15 percent of all stream-water samples that were collected (Null and others, 2019). Estrone and 4-androstene-3,17-*dione* were detected in all untreated-influent samples and in 28 percent or more of treated-effluent samples collected at WWTPs (fig. 5). Estrone was detected in 5 of the 35 stream-water samples (about 14 percent of samples); all stream-water samples in which estrone was detected were collected at the three most upstream sites on the Trinity River. 4-Androstene-3,17-*dione*, which was detected in 93 percent of treated-effluent samples at WWTPs, was not detected in samples collected at the two most upstream sites (Trinity River Highway 310 and Trinity River Dallas) or in samples collected at the most downstream site (Trinity River Trinidad) but was detected in samples collected at the Trinity River Wilmer and Trinity River Rosser sites. Detected concentrations, 0.001 and 0.0008 µg/L, respectively, were slightly greater than or equal to the LRL of 0.0008 µg/L used for most analyses of the compound.

Of the four stanol/sterol compounds analyzed, two compounds (3-beta-coprostanol and cholesterol) were detected in stream-water samples collected at each main-stem site on the Trinity River. The frequency at which 3-beta-coprostanol was detected (31 percent of stream-water samples) was lower than the frequency at which cholesterol was detected (77 percent of the stream-water samples). 3-beta-Coprostanol and cholesterol were detected in all untreated-influent samples at WWTPs (fig. 5) and were detected in 86 and 14percent of treated-effluent samples, respectively. Generally, concentrations of 3-beta-coprostanol and cholesterol measured in samples collected at the main-stem sites on the Trinity River decreased from upstream to downstream (Null and others, 2019); however, the decreases were not statistically significant (*p*-value greater than 0.370 for each compound, one-way ANOVA on ranks).

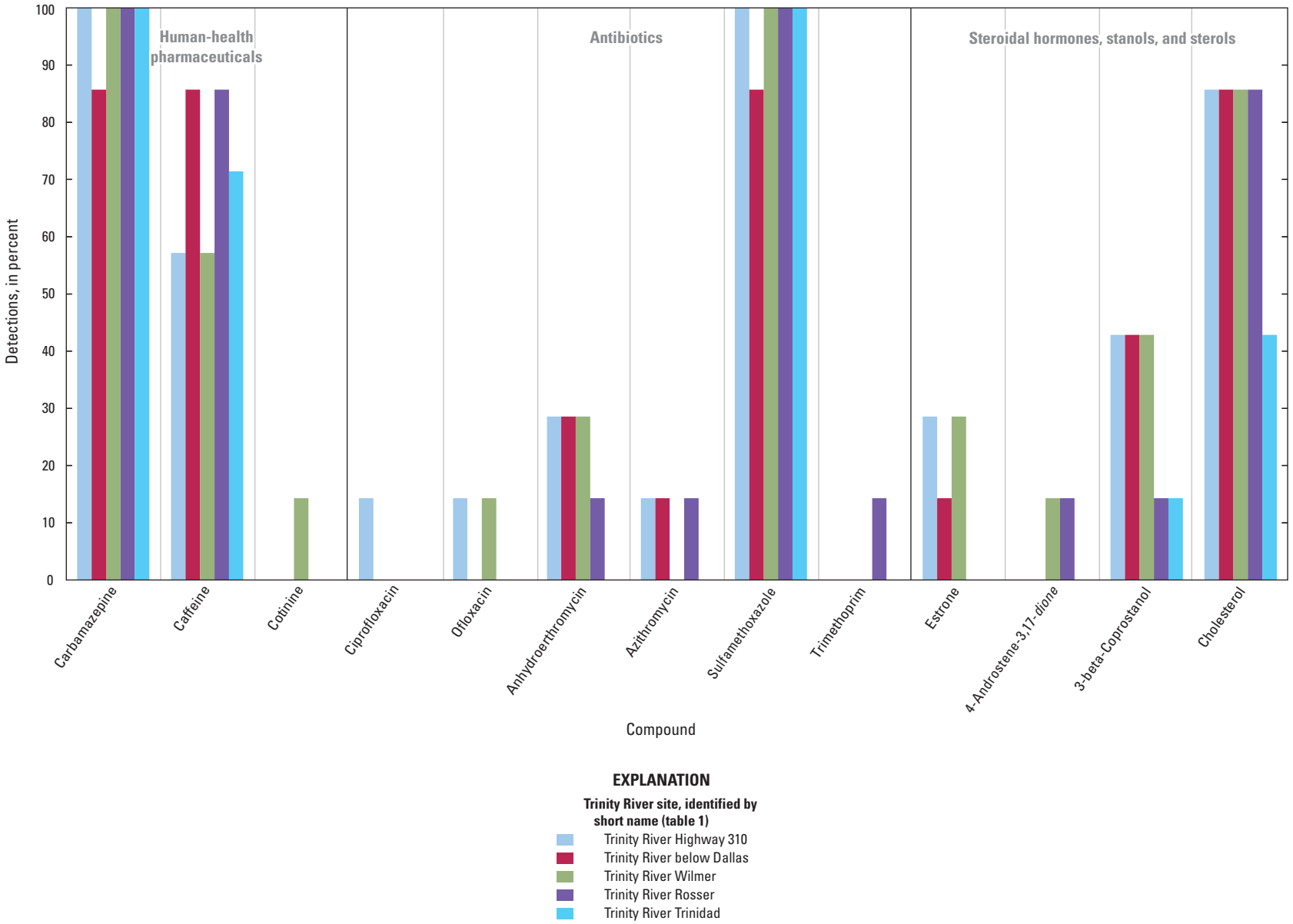


Figure 10. Detection frequencies of human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, and sterols in seven stream-water samples collected at study sites on the Trinity River in or near Dallas, Texas, 2009–13.

In stream-water samples collected at main-stem sites on the Trinity River, there were no detected concentrations of detergent compounds that were equal to or greater than the LRL. The lack of detections of detergent compounds in samples collected at main-stem river sites is consistent with results from WWTPs; some of these compounds were detected in untreated-influent samples, but none was detected in treated-effluent samples, representative of discharge to the Trinity River (fig. 7).

Two personal-use product compounds (the fragrances AHTN and HHCB) were detected in stream-water samples collected at main-stem sites on the Trinity River; AHTN was detected in samples collected at two of the five main-stem sites, whereas HHCB was detected at all five sites (fig. 11). AHTN and HHCB were detected in each treated-effluent sample collected at the WWTPs (fig. 7). Compared to the frequency of detection of AHTN in the WWTP samples, the frequency of detection of AHTN in samples collected at the main-stem site closest to where a WWTP discharges (Trinity River Highway 310) was substantially less (14 percent of samples). AHTN has been reported at greater detection frequencies in samples collected at stream sites in other studies. For example, AHTN was detected in all samples analyzed in a study by Stackelberg and others (2004). For the analyses in this report, HHCB was detected in every sample collected at the four most upstream sites on the Trinity River and in about 94 percent of all stream-water samples (fig. 11; Null and others, 2019). Stackelberg and others (2004) reported a similar detection frequency for HHCB; HHCB was detected in 92 percent of the samples that represented streamflow and raw water for municipal supply. Median concentrations of HHCB in samples collected at main-stem Trinity River sites were greatest at the Trinity River Wilmer site (0.44 µg/L), and the median concentration decreased substantially at the downstream Trinity River Rosser and Trinity River Trinidad sites (0.22 and 0.17 µg/L, respectively; Null and others, 2019). HHCB was the only compound for which a statistically significant change in concentrations was measured between upstream and downstream main-stem sites. Median concentrations of HHCB were statistically significantly lower at the most distant main-stem site, the Trinity River Trinidad site, than at the other sites (*p*-value equals 0.001, one-way ANOVA on ranks with Dunn's multiple comparison test).

Two of the 10 pesticide compounds (1,4-dichlorobenzene and metolachlor) were detected in stream-water samples collected at main-stem Trinity River sites (fig. 11). The compound 1,4-dichlorobenzene was detected in 86 percent of samples at the two most upstream main-stem sites (Null and others, 2019). At the three remaining main-stem sites, detection frequencies decreased with distance downstream to 57, 14, and zero percent, respectively. Median concentrations of 1,4-dichlorobenzene also decreased with distance downstream, but the change was not statistically significant (*p*-value greater than 0.05, one-way ANOVA on ranks). Metolachlor was detected in only one sample collected at one of the five main-stem sites (the Trinity River Rosser site). Although the insect repellent DEET was detected in concentrations equal to or greater than the LRL (0.06 µg/L) at all five main-stem sites, none of the concentrations was equal to or greater than the censoring level of 0.700 µg/L used for DEET (table 4).

Three of the 10 industrial wastewater compounds were detected in water samples collected at the main-stem sites on the Trinity River (fig. 11). Benzophenone, tribromomethane, and triethyl citrate were detected in samples collected at each main-stem site, except for tribromomethane, which was not detected at the farthest downstream site, Trinity River Trinidad. All three of these compounds were detected in at least 93 percent of treated-effluent samples at WWTPs (fig. 8). Benzophenone was detected in 86 percent of water samples collected at each main-stem Trinity River site. Tribromomethane was detected in each water sample collected at the three upstream sites on the Trinity River. Detection frequency of values greater than or equal to the LRL decreased to 43 percent at the Trinity River Rosser site, and there were no detections at the Trinity River Trinidad site. Median concentrations of benzophenone decreased with distance downstream, but the changes were slightly outside the range considered statistically significant at the 95-percent confidence level (*p*-value equals 0.059, one-way ANOVA on ranks). Median concentrations of triethyl citrate also decreased with distance downstream, but the changes were not statistically significant (*p*-value equals 0.323, one-way ANOVA on ranks). Median concentrations of tribromomethane were greatest at the three upstream sites Trinity River Highway 310, Trinity River Dallas, and Trinity River Wilmer (0.40, 0.41, and 0.43 µg/L, respectively) then decreased substantially to 0.17 µg/L at the Trinity River Rosser site (Null and others, 2019).

The two disinfection compounds phenol and triclosan were not detected in concentrations equal to or greater than the LRL in stream-water samples collected at the main-stem Trinity River sites. This agrees with results from the WWTPs, where these compounds were detected in untreated-influent samples but not in treated-effluent samples (fig. 8).

The 10 PAH compounds were not detected in concentrations equal to or greater than the LRL in stream-water samples collected at main-stem Trinity River sites. This agrees with results from the WWTPs, where these compounds were not detected in the treated-effluent samples (fig. 8).

Three of the six flame retardant and plasticizer compounds (tributyl phosphate, tris(2-butoxyethyl) phosphate, and tris(dichloroisopropyl)phosphate) were detected in stream-water samples collected at main-stem sites (fig. 11). Two of these compounds, tributyl phosphate and tris(dichloroisopropyl)phosphate, were detected in at least 86 percent of treated-effluent samples at WWTPs (fig. 9), and both were detected at all main-stem sites (fig. 11). Tris(dichloroisopropyl)phosphate was the only compound detected in all 35 main-stem samples in this study. Median concentrations of tributyl phosphate and tris(dichloroisopropyl)phosphate did not

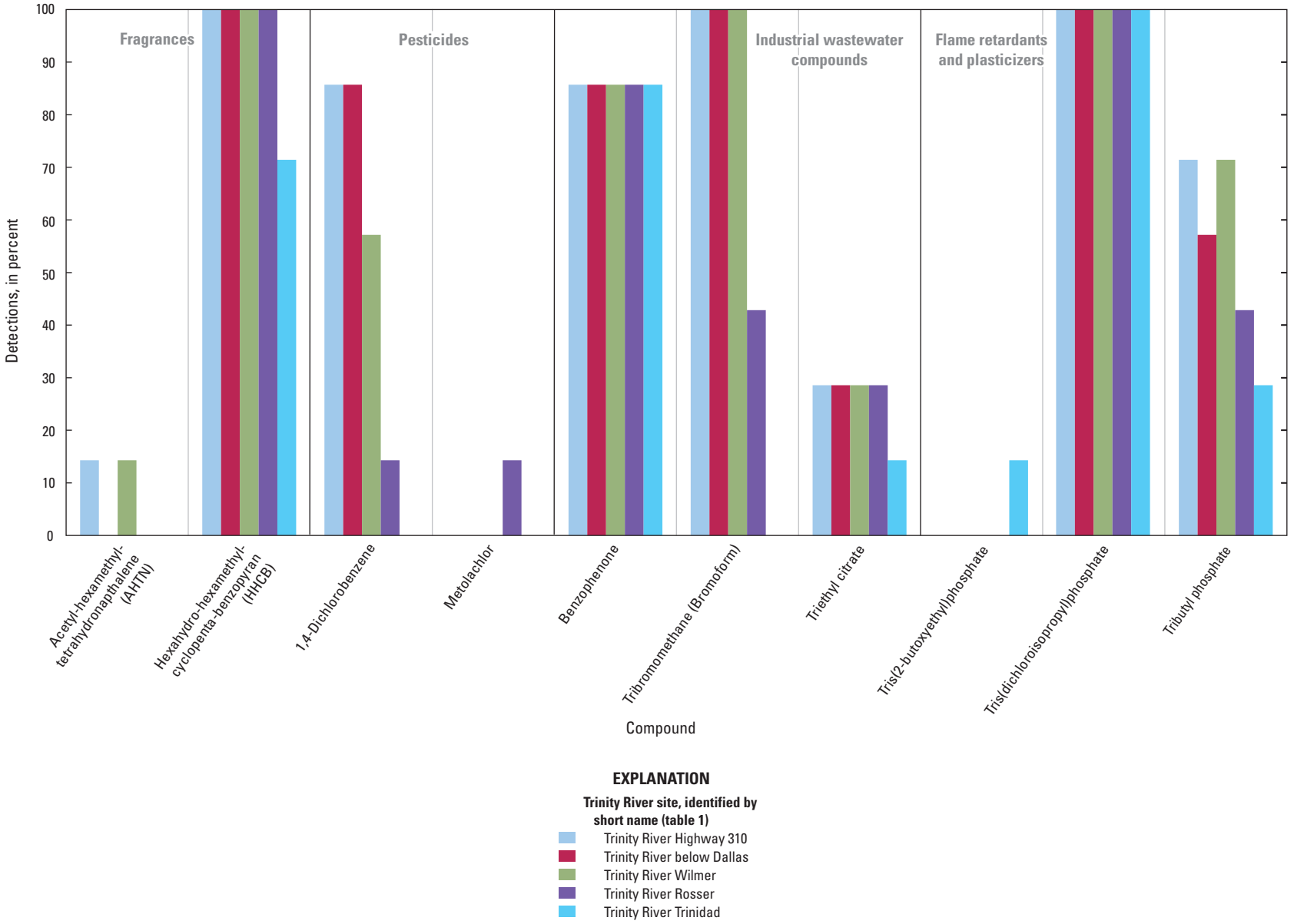


Figure 11. Detection frequencies of personal-use products (fragrances), pesticides, industrial wastewater compounds, and flame retardants and plasticizers in seven stream-water samples collected at study sites on the Trinity River in or near Dallas, Texas, 2009–13.

change significantly between sites, possibly because of their resistance to degradation in the environment (*p*-values equal 0.355 and 0.570, respectively, one-way ANOVA on ranks). Median concentrations of tributyl phosphate and tris(dichloroisopropyl)phosphate measured in stream-water samples collected at the five main-stem sites ranged from 0.14 to 0.18 µg/L and 0.51 to 0.63 µg/L, respectively.

Compounds of Emerging Concern in Bed-Sediment Samples

Eleven of the 57 CECs analyzed in bed-sediment samples were detected (fig. 12). Two compounds (beta-sitosterol and cholesterol) were detected in bed-sediment samples collected at all five main-stem sites. Nine other CECs were detected in bed-sediment samples, including 3-beta-coprostanol (also detected in stream-water samples at all five main-stem sites), phenol, fluoranthene, pyrene, and bis(2-ethylhexyl) phthalate. Of the 11 CECs detected in bed-sediment samples, 9 were not detected in any stream-water sample (cholesterol and 3-beta-coprostanol were the exceptions) likely because of the strong hydrophobic characteristics of these compounds. At the two farthest downstream sites on the Trinity River, the detection of compounds other than beta-sitosterol or cholesterol was rare—phenol was detected in one bed-sediment sample collected at the Rosser site, and bis(2-ethylhexyl) phthalate was detected in one bed-sediment sample collected at the Trinity River Trinidad site (fig. 12; Null and others, 2019). This indicates that most compounds analyzed were not subjected to substantial downstream transport. Overall, detection frequencies of CECs decreased with distance downstream; however, the decrease was not statistically significant (*p*-value equals 0.205, one-way ANOVA on ranks). Concentrations of five CECs in bed-sediment samples were highest at the most distant downstream site at which they were detected: cholesterol, phenol, fluoranthene, pyrene, and bis(2-ethylhexyl) phthalate, indicating that these CECs are resistant to degradation during downstream transport. It is important to note that variable LRLs for some compounds complicate data interpretation.

All four stanol/sterol compounds were detected in bed-sediment samples (fig. 12). Two of these compounds, beta-sitosterol and cholesterol, were detected in 54 and 57 percent of bed-sediment samples, respectively (Null and others, 2019). These two compounds were the most frequently detected CECs in bed-sediment samples in this study. Cholesterol and 3-beta-coprostanol were the only two CECs detected in stream-water and bed-sediment samples (figs. 10 and 12). Cholesterol and 3-beta-coprostanol were detected in every untreated-influent sample collected at WWTPs and in 14 and 86 percent of treated-effluent samples collected at WWTPs, respectively (fig. 5). At the farthest upstream site (Trinity River Highway 310), 3-beta-coprostanol and beta-stigmastanol were each detected in one of the seven samples collected, which corresponds to a 14-percent detection frequency (fig. 12); 3-beta-coprostanol was also detected in one sample collected at the second farthest upstream site on the Trinity River (Trinity River Dallas). Neither 3-beta-coprostanol nor beta-stigmastanol was detected in any samples collected at the three most downstream sites on the Trinity River. Beta-sitosterol was not detected in treated-effluent samples at WWTPs or in stream-water samples collected at any of the main-stem sites on the Trinity River. The maximum concentrations for beta-sitosterol (4,310 micrograms per kilogram [µg/kg]) and cholesterol (3,550 µg/kg), however, were measured in samples collected at the most upstream site on the Trinity River (Trinity River Highway 310) (Null and others, 2019). Downstream from the Trinity River Highway 310 site, beta-sitosterol and cholesterol concentrations decreased in bed-sediment samples collected at the Trinity River Dallas and Trinity River Wilmer sites, and then increased at the two sites farthest downstream (Trinity River Rosser and Trinity River Trinidad). Concentrations were not significantly different between sites for either beta-sitosterol or cholesterol (*p*-values equal 0.896 and 0.226, respectively, one-way ANOVA on ranks).

Two of the 10 compounds in the personal-use product group were detected in bed-sediment samples collected at main-stem sites (fig. 12). Indole was detected in one sample collected at the Trinity River Highway 310 site, and *d*-limonene was detected in one sample collected at the Trinity River Dallas site. The compound *d*-limonene was detected in every untreated-influent sample collected at the WWTPs, whereas indole was detected in only 50 percent of untreated-influent samples (fig. 7). Neither indole nor *d*-limonene was detected in any treated-effluent samples collected at the WWTP sites or in stream-water samples collected at the main-stem Trinity River sites (Null and others, 2019).

Phenol was the only disinfection compound detected in bed-sediment samples (fig. 12). Phenol was detected in one sample collected at the Trinity River Highway 310 site and in one sample collected at the Trinity River Rosser site. Phenol was not detected in treated-effluent samples collected at the WWTPs (fig. 8) or in main-stem Trinity River stream-water samples (Null and others, 2019).

Three of the 10 PAH compounds were detected in bed-sediment samples (benzo[a]pyrene, fluoranthene, and pyrene); however, none of these three compounds was detected in more than 50 percent of the samples collected at a given site (fig. 12). PAHs were not detected in treated-effluent samples at WWTPs (fig. 8) or stream-water samples (Null and others, 2019), likely because of their strong hydrophobic characteristics (Abdel-Shafy and Mansour, 2015). Fluoranthene and pyrene were detected in bed-sediment samples collected at the three sites farthest upstream on the Trinity River. Overall, the detection frequency for fluoranthene and pyrene was low at the five Trinity River main-stem sites (detected in 23 percent of all bed-sediment samples) and was very low for benzo[a]pyrene (detected in 3 percent of all bed-sediment samples); however, each of these three PAHs was detected in at least one bed-sediment sample collected at the Trinity River Wilmer site (fig. 12).

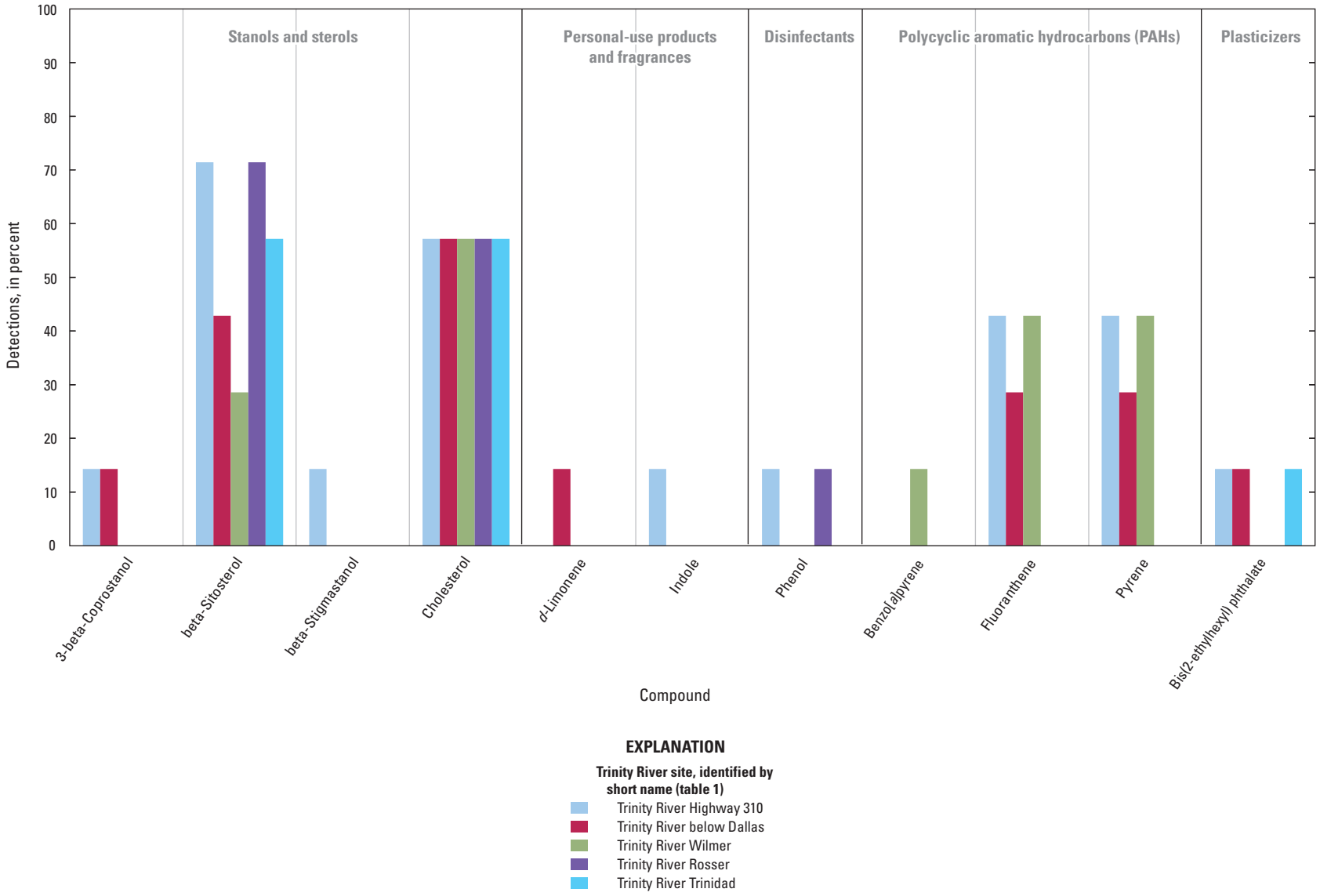


Figure 12. Detection frequencies of stanols and sterols, personal-use products and fragrances, disinfectants, polycyclic aromatic hydrocarbons, and plasticizers in seven bed-sediment samples collected at study sites on the Trinity River in or near Dallas, Texas, 2009–13.

Concentrations of fluoranthene and pyrene increased with distance downstream and were greatest at the Trinity River Wilmer site (116 and 112 µg/kg, respectively; Null and others, 2019). None of the PAHs was detected at the two most downstream sites on the Trinity River.

One of the nine flame retardant and plasticizer compounds, bis(2-ethylhexyl) phthalate, was detected in bed-sediment samples (fig. 12). The plasticizer compound bis(2-ethylhexyl) phthalate was detected in one sample collected at each of three different sites. The maximum concentration of bis(2-ethylhexyl) phthalate (1,030 µg/kg) was detected in a sample collected at the most downstream site, Trinity River Trinidad (Null and others, 2019). Because of its strong hydrophobic characteristics (U.S. Department of Health and Human Services, 2002), this compound was not targeted in water samples collected at PWTP, WWTP, or Trinity River main-stem sites.

Summary

The U.S. Geological Survey, in cooperation with the City of Dallas, Dallas Water Utilities, evaluated the occurrence and concentrations of selected compounds of emerging concern (CECs) in samples collected at potable water treatment plants (PWTPs) and wastewater treatment plants (WWTPs) in Dallas, and downstream in the Trinity River, Texas, from August 2009 to December 2013. CECs are synthetic or naturally occurring chemicals that are not commonly monitored in the environment but can enter the environment and cause known or suspected adverse ecological or human health effects. Targeted CECs (120 total) included human-health pharmaceuticals (prescription and nonprescription), antibiotics, steroidal hormones, stanols, sterols, detergents and detergent metabolites (hereinafter referred to as "detergents"), personal-use products (flavors and fragrances), pesticides and repellents, industrial wastewater compounds, disinfection compounds, polycyclic aromatic hydrocarbons (PAHs), flame retardants, and plasticizers. Water samples were collected at three PWTPs, two WWTPs, and five main-stem sites on the Trinity River in and near Dallas. Additionally, bed-sediment samples were collected at each of the Trinity River sites. Bed-sediment samples were analyzed only for stanols, sterols, and organic compounds typically found in domestic and industrial wastewater (57 compounds). Quality-control samples collected during the study period included equipment blanks, replicates, matrix spikes, and surrogates. During the study, 129 samples were analyzed; of these 129 samples, 105 were environmental samples, and 24 were quality-control samples.

Generally, CECs were detected more frequently in samples collected at WWTPs than in samples collected at PWTPs. Water treatment processes at PWTPs and WWTPs were effective at reducing concentrations of most CECs to undetectable levels or transforming the compounds into degradates that were not analyzed.

At PWTPs, 16 out of 120 targeted CECs were detected. Only four compounds were detected in more than 30 percent of raw-water samples: sulfamethoxazole (81 percent of samples), carbamazepine (71 percent of samples), cholesterol (52 percent of samples), and tris(dichloroisopropyl) phosphate (33 percent of samples). Only two compounds were detected in more than 20 percent of finished-water samples: tribromomethane (48 percent of samples) and tris(dichloroisopropyl)phosphate (33 percent of samples). Eleven compounds were detected in raw-water samples but were not detected in finished-water samples (that is, compounds were removed or degraded to compounds that were not analyzed): three human-health pharmaceutical compounds (carbamazepine, sulfamethoxazole, and caffeine), three steroidal hormones/stanols/sterols (estrone, 3-beta-coprostanol, and cholesterol), a detergent (4-*tert*-octylphenol), a flavor/fragrance (hexahydro-hexamethyl-cyclopenta-benzopyran [HHCB]), an industrial wastewater compound (*p*-cresol), a PAH (pyrene), and a flame retardant/plasticizer (tributyl phosphate). Only three compounds were detected in raw water and finished water, indicating that treatment processes likely did not remove or degrade these compounds: the industrial wastewater compounds benzophenone and methyl salicylate and the flame retardant/plasticizer tris(dichloroisopropyl)phosphate, which is a suspected endocrine disrupting compound. Benzophenone and methyl salicylate, however, were detected in only one finished-water sample. Tris(dichloroisopropyl)phosphate, a persistent plasticizer/flame retardant, was detected at the same frequency (33 percent of samples) in raw and finished water. Two compounds that were detected in finished water but not in raw water were an androgen (4-androstene-3,17-*dione*) and tribromomethane, which is a byproduct of water treatment processes.

At WWTPs, 74 of 120 targeted CECs were detected. Of these 74 CECs, 73 were detected in untreated-influent samples. Thirty-one CECs were detected in all untreated-influent samples; however, only 5 CECs were detected in all treated-effluent samples: carbamazepine, acetyl-hexamethyl-tetrahydronaphthalene (AHTN), HHCB, 1,4-dichlorobenzene, and tris(dichloroisopropyl)phosphate. Forty-four CECs were detected in untreated-influent samples but not in treated-effluent samples, indicating removal or degradation of these compounds. Twenty-nine compounds were detected in untreated-influent water and treated-effluent water, indicating that treatment processes likely did not remove or degrade these compounds, which included carbamazepine, sulfamethoxazole, caffeine, anhydroerythromycin, azithromycin, trimethoprim, 4-androstene-3,17-*dione*, 3-beta-coprostanol, AHTN, HHCB, benzophenone, tribromomethane, triethyl citrate, tributyl phosphate, and tris(dichloroisopropyl) phosphate. Equilin, an estrogen, was the only compound that was detected in treated-effluent water but not in untreated-influent water; however, it was detected in only one treated-effluent sample.

Detection frequencies decreased substantially for most groups of CECs between untreated-influent samples at WWTPs and treated-effluent samples at WWTPs. For example, 11 steroidal hormones were detected in untreated-influent water, and, of these, only 3 were detected in treated-effluent water, indicating that 73 percent of detected compounds in this group were removed or degraded by wastewater treatment processes. All detergents (five compounds), disinfectants (two compounds), and PAHs (five compounds) were removed or degraded. Of the nine flavor/fragrance compounds detected in untreated-influent water, only two (AHTN and HHCB) were detected in treated-effluent water, indicating that 78 percent of the detected compounds in this group were removed or degraded. Four pesticide compounds were detected in untreated-influent water, and only one of these (1,4-dichlorobenzene) was detected in treated-effluent water, indicating that 75 percent of pesticide compounds were removed or degraded. In addition, of the six flame retardant/plasticizer compounds detected in untreated-influent water, three were detected in treated-effluent water, indicating a 50-percent removal or degradation of these types of compounds.

Wilcoxon signed-rank test results indicated concentrations decreased significantly during wastewater treatment processes for 22 CECs detected in untreated-influent and treated-effluent samples collected at WWTPs (8 human-health pharmaceutical and antibiotic compounds, 3 steroidal hormones, 2 stanol/sterol compounds, 2 detergent compounds, and various CECs from other classes of compounds, such as HHCB, *N*,*N*-diethyl-*m*-toluamide (DEET), 1,4-dichlorobenzene, *p*-cresol, benzophenone, triethyl citrate, and tributyl phosphate. However, concentrations of carbamazepine and tribromomethane increased significantly between samples types at WWTPs.

Twenty-three CECs were detected in stream-water samples collected at the 5 Trinity River main-stem sites. Ten of these 23 CECs were detected in samples collected at all 5 sites—carbamazepine, sulfamethoxazole, caffeine, 3-beta-coprostanol, cholesterol, HHCB, benzophenone, triethyl citrate, tributyl phosphate, and tris(dichloroisopropyl) phosphate—indicating their persistence in the environment. Median concentrations of nine of these persistent compounds decreased between the most upstream main-stem site closest to where WWTP effluent is discharged and the farthest downstream site on the Trinity River, Trinity River Trinidad. The concentration of tributyl phosphate was similar at the most upstream and farthest downstream main-stem sites. HHCB was the only compound with a statistically significant change in concentration; the median concentration of HHCB was statistically significantly lower at the most distant main-stem site compared with the median HHCB concentration measured at the other four main-stem sites.

Eleven of the 57 targeted compounds were detected in bed-sediment samples. Of these 11 compounds, only beta-sitosterol and cholesterol were detected in bed-sediment samples at all 5 study sites on the Trinity River, an indication that these 2 compounds were ubiquitous throughout the study reach. Nine other CECs were detected in bed-sediment samples, including 3-beta-coprostanol (also detected in stream-water samples at all five main-stem sites), phenol, fluoranthene, pyrene, and bis(2-ethylhexyl) phthalate. Concentrations of five CECs in bed-sediment samples were highest at the most distant downstream site at which they were detected—cholesterol, phenol, fluoranthene, pyrene, and bis(2-ethylhexyl) phthalate—indicating that these CECs are resistant to degradation during downstream transport. Of the 11 CECs detected in bed-sediment samples, 9 were not detected in any stream-water sample, likely because of the strong hydrophobic characteristics of these compounds; cholesterol and 3-beta-coprostanol were the exceptions.

Results from water treatment plants indicate that the water treatment process is effective at removing or degrading most CECs. Water treatment processes, however, are less effective for compounds that are engineered to be resistant to degradation (for example, flame retardants). Results from Trinity River main-stem sites, including bed-sediment data, indicate that some compounds are naturally attenuated during transport, but a few are persistent throughout the study reach. For example, tris(dichloroisopropyl)phosphate, a flame retardant and plasticizer, was resistant to water treatment processes at PWTPs and WWTPs and was ubiquitous in receiving waters of the Trinity River. Many CECs are hydrophobic and were only detected in bed sediment, indicating multiple pathways through which CECs can persist in the environment. CECs in highly urbanized areas could negatively affect the health of aquatic organisms; however, more research is needed to determine specific effects of CECs on aquatic and terrestrial organisms.

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Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.

[CAS, Chemical Abstracts Service; LRL, laboratory reporting level; USGS, U.S. Geological Survey; SH, USGS National Water Quality Laboratory schedule; µg/L, micrograms per liter; LCAB, USGS Organic Geochemistry Research Laboratory liquid chromatography/mass spectrometry antibiotics schedule; N/A, not available or not applicable; pct, percent; µg/kg, micrograms per kilogram; α, alpha; β, beta; UV, ultraviolet]

Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Human-health prescription pharmaceuticals (SH2080, except as noted)					
Carbamazepine†	62793	298-46-4	0.005 µg/L	Anticonvulsant and antimanic (preferred values by LCAB).	1, 2, 3
Carbamazepine	62793	298-46-4	0.060 µg/L	Anticonvulsant and antimanic.	1, 2, 3
Diltiazem	62008	42399-41-7	0.04 µg/L	Antihypertensive.	2, 3
Diphenhydramine	62796	147-24-0	0.036	Antihistamine, antiemetic (anti-nausea), sleep aid, and sedative.	1
Sulfamethoxazole	62021	723-46-6	0.1 µg/L	Human antibiotic used to treat urinary tract infections, often used in combination with trimethoprim.	2, 3
Sulfamethoxazole†	62775	723-46-6	0.005 µg/L	Human antibiotic used to treat urinary tract infections, often used in combination with trimethoprim (preferred values by LCAB method).	1, 2, 3
Thiabendazole	62801	148-79-8	0.060 µg/L	Anthelmintics (used to treat worm infections).	1, 2, 3
Warfarin	62024	81-81-2	0.080–0.100 µg/L	Anticoagulant. In large concentrations used as a rodenticide for controlling rats and house mice in and around homes, animal and agricultural premises, and commercial and industrial sites.	1, 2, 3
Human-health nonprescription pharmaceuticals (SH2080, except as noted)					
1,7-Dimethylxanthine (*p*-Xanthine)	62030	611-59-6	0.100–0.120 µg/L	Caffeine metabolite.	1, 2, 3
Acetaminophen	62000	103-90-2	0.080–0.120 µg/L	Analgesic.	2, 3
Albuterol (Salbutamol)	62020	18559-94-9	0.060–0.080 µg/L	Antiasthmatic.	1, 2, 3
Caffeine	50305	58-08-2	0.060–0.200 µg/L	Stimulant.	1
Caffeine†	50305	58-08-2	0.060–0.100 µg/L	Stimulant (preferred values by SH1433).	1
Codeine	62003	76-57-3	0.040–0.046 µg/L	Analgesic.	1, 2, 3
Cotinine	62005	486-56-6	0.4–0.8 µg/L	Nicotine metabolite (SH1433).	6
Cotinine†	62005	486-56-6	0.026–0.038 µg/L	Nicotine metabolite (preferred values by SH2080).	6
Dehydronifedipine	62004	67035-22-7	0.080–0.160 µg/L	Antianginal.	1
Human-health nonprescription pharmaceuticals (LCAB)					
Ibuprofen	62014	15687-27-1	0.05 µg/L	Nonsteroidal anti-inflammatory drug.	1
Antibiotics - Quinolones (LCAB)					
Ciprofloxacin	62898	85721-33-1	0.005 µg/L	Broad spectrum antimicrobial agent, treats urinary tract infections and gastrointestinal and abdominal infections.	2, 3
Enrofloxacin	66495	93106-60-6	0.005 µg/L	Used to treat pets and domestic animals.	2, 3
Lomefloxacin	62900	98079-51-7	0.005 µg/L	Used to treat bacterial infections, bronchitis, and urinary tract infections.	2, 3
Norfloxacin	62757	70458-96-7	0.005 µg/L	Chemotherapeutic antibacterial agent, occasionally used to treat urinary tract infections.	2, 3
Ofloxacin	62899	82419-36-1	0.005 µg/L	Used in eye and ear drops.	2, 3
Sarafloxacin	62771	98105-99-8	0.005 µg/L	Used to treat poultry and fish.	2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Antibiotics - Macrolides (LCAB)					
Anhydroerythromycin	63674	114–07–8	0.008 µg/L	Macrolide antibiotic used in erythromycin degradate.	11
Azithromycin	62792	117772-70-0	0.005 µg/L	Used to treat bacterial infections, bronchitis, pneumonia, sexually transmitted diseases, infections in the ears, lungs, sinuses, skin, throat, and reproductive organs.	2, 3
Erythromycin	62797	114-07-8	0.008 µg/L	Used to treat infections, bronchitis, diptheria, Legionnaires disease, pertussis, pneumonia, rheumatic fever, venereal disease, ear, intestine, lung, and urinary tract infections.	2, 3
Roxithromycin	62895	80214-83-1	0.005 µg/L	Used to treat respiratory tract, urinary, and soft tissue infections.	2, 3
Tylosin	62896	1401-69-0	0.005 µg/L	Bacteriostatic food additive used in veterinary medicine.	2, 3
Virginiamycin	62897	11006-76-1	0.005 µg/L	Used in the fuel ethanol industry to prevent microbial contamination.	2, 3
Antibiotics - Sulfonamides (LCAB)					
Sulfathiazole	62778	72-14-0	0.005–0.050 µg/L	Oral and topical antimicrobial agent.	2, 3
Sulfachloropyridazine	62774	80-32-0	0.005 µg/L	Used to treat acute urinary tract infections in pediatric patients.	2, 3
Sulfadiazine	62963	68-35-9	0.005–0.100 µg/L	Used to treat urinary tract infections.	2, 3
Sulfadimethoxine	62776	122-11-2	0.005 µg/L	Used to treat respiratory, urinary tract, enteric, and soft tissue infections.	2, 3
Sulfamethazine	61762	57-68-1	0.005 µg/L	Antibiotic commonly added to animal feed (swine and poultry) in subtherapeutic doses as growth-promoting agents.	2, 3
Antibiotics - Tetracyclines (LCAB)					
4-Epichlortetracycline hydrochloride	63731	14297-93-9	0.010 µg/L	Degradation product of chlorotetracycline.	2, 3
4-Epioxytetracycline	63729	35259-39-3	0.010 µg/L	Degradation product of oxytetracycline.	2, 3
4-Epitetracycline hydrochloride	63727	79-85-6	0.010 µg/L	Degradation product of oxytetracycline.	2, 3
Chlortetracycline	61744	64-72-2	0.010 µg/L	Broad-spectrum antibiotic commonly given to poultry, swine, and livestock. May lead to the development of antibiotic resistance.	2, 3
Doxycycline	62694	564-25-0	0.010 µg/L	Used to treat pneumonia, respiratory tract infections, Lyme disease, acne, infections of the skin and urinary systems, and anthrax. Also used to help prevent malaria.	2, 3
iso-Chlortetracycline	64175	514-53-4	0.010 µg/L	Degradation product of tetracycline.	2, 3
epi-iso-Chlortetracycline	64047	N/A	0.010 µg/L	Degradation product of chlorotetracycline.	2, 3
Oxytetracycline	61759	6153-64-6	0.010 µg/L	Broad-spectrum antibiotic, used to treat infections caused by chlamydia, acne, and chronic bronchitis.	2, 3
Tetracycline	62781	60-54-8	0.010 µg/L	Used to treat urinary tract infections, chlamydia, gonorrhea, acne, and chronic bronchitis.	2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Antibiotics - other (LCAB)					
Chloramphenicol	65194	56-75-7	0.100 µg/L	Human antibiotic.	1
Lincomycin	62894	154-21-2	0.005 µg/L	Human antibiotic.	11
Ormetoprim	62962	6981-18-6	0.005 µg/L	Veterinary antibiotic.	1
Trimethoprim†	62023	738-70-5	0.020–0.034 µg/L	Human antibiotic often used in combination with sulfamethoxazole (preferred values by SH2080).	1, 2, 3
Trimethoprim	62023	738-70-5	0.005 µg/L	Human antibiotic often used in combination with sulfamethoxazole.	1, 2, 3
Steroidal hormones, stanols, and sterols (SH2434)					
Natural estrogens (SH2434)					
17-α-Estradiol	64508	57-91-0	0.0008 µg/L	Low occurrence in humans, common in other species.	1, 2, 3
17-β-Estradiol	64510	50-28-2	0.0008 µg/L	Principal estrogen in humans, strong estrogen.	1
Equilenin	64518	517-09-9	0.002 µg/L	Equine estrogen used in hormone replacement therapy.	1, 2, 3
Estriol	64520	50-27-1	0.002 µg/L	Metabolite of 17-β-Estradiol.	1, 2, 3
Estrone	64521	53-16-7	0.0008 µg/L	Metabolite of 17-β-Estradiol.	1, 2, 3
Synthetic estrogens (SH2434)					
17-α-Ethynyl estradiol	64509	57-63-6	0.0008 µg/L	Used in oral contraceptives, very strong estrogen.	2, 3
Equilin	64519	474-86-2	0.004–0.008 µg/L	Equine estrogen used in hormone replacement therapy.	1, 2, 3
Mestranol	64522	72-33-3	0.0008 µg/L	Used in oral contraceptives, metabolized to 17-α-ethynyl estradiol prior to excretion.	1, 2, 3
trans-Diethylstilbestrol	64516	56-53-1	0.0008 µg/L	Used in pharmaceuticals.	1, 2, 3
Natural androgen (SH2434)					
4-Androstene-3,17-*dione*	64513	63-05-8	0.0008 µg/L	Testosterone precursor.	1, 2, 3
cis-Androsterone	64515	53-41-8	0.0008 µg/L	Testosterone metabolite, commonly used in deer repellent.	1
Epitestosterone	64517	481-30-1	0.002–0.004 µg/L	Human androgen.	1, 2, 3
Testosterone	64525	58-22-0	0.0008–0.0016 µg/L	Principal human androgen, strong androgen.	1, 2, 3
Synthetic androgen (SH2434)					
11-Ketotestosterone	64507	564-35-2	0.002 µg/L	Very strong androgen.	1, 2, 3
Dihydrotestosterone (DHT)	64524	521-18-6	0.0008 µg/L	Also known as Stanolone. Testosterone metabolites; very strong androgen.	1, 2, 3
Natural progestin (SH2434)					
Progesterone	64523	57-83-0	0.0008–0.0016 µg/L	Principal human progestational hormone.	1, 2, 3
Synthetic progestin (SH2434)					
Norethindrone	64511	68-22-4	0.0008 µg/L	Used in oral contraceptives.	1

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Stanols and sterols (animal) (SH2434, except as noted)					
3-β-Coprostanol	62057	360-68-9	1.8–2.0 µg/L	Animal fecal indicator, useful sewage tracer (values by SH1433).	1
3-β-Coprostanol†	64512	360-68-9	0.20–2.0 µg/L	Animal fecal indicator, useful sewage tracer (preferred values by SH2434).	1, 2, 3
Cholesterol	62072	57-88-5	2.0 µg/L	An important structural molecule of animal cell membranes, precursor for steroid hormones, bile acids, and vitamin D (by SH1433).	1
Cholesterol†	64514	57-88-5	0.20–2.0 µg/L	An important structural molecule of animal cell membranes, precursor for steroid hormones, bile acids, and vitamin D (preferred values by SH2434).	2, 3
Stanols and sterols (plant) (SH1433)					
β-Stigmastanol	62086	19466-47-8	2.0–2.6 µg/L	Herbivore fecal indicator (digestion of sitosterol).	1
β-Sitosterol	62068	83-46-5	4 µg/L	Plant sterol.	1
Wastewater compounds (SH1433)					
Detergents and detergent metabolites (SH1433)					
4-Cumylphenol	62060	599-64-4	0.060–0.100 µg/L	Nonionic detergent metabolite.	1
4-*n*-Octylphenol	62061	1806-26-4	0.06–0.16 µg/L	Nonionic detergent metabolite.	1
4-Nonylphenol (sum of all isomers)	62085	84852-15-3	2 µg/L	Nonionic detergent.	10
4-Nonylphenol diethoxylate, (sum of all isomers) also known as NP2EO	62083	N/A	5 µg/L	Nonionic detergent metabolite; surfactant metabolite.	9
4-*tert*-Octylphenol	62062	140-66-9	0.14–1.40 µg/L	Nonionic detergent metabolite.	5
4-*tert*-Octylphenol diethoxylate, (sum of all isomers) also known as OP2EO	61705	N/A	1 µg/L	Nonionic detergent.	9
4-*tert*-Octylphenol monoethoxylate, (sum of all isomers) also known as OP1EO	61706	N/A	1 µg/L	Nonionic detergent or metabolite.	9
Personal-use products (flavors and fragrances) (SH1433)					
3-Methyl-1H-indole (Skatole)	62058	83-34-1	0.036–0.040 µg/L	Fragrance, stench in feces and coal tar.	1
Acetophenone	62064	98-86-2	0.4 µg/L	Fragrance in detergent and tobacco, flavor in beverages.	1
Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)	62065	21145-77-7	0.028–0.075 µg/L	Used in cosmetics (except oral products).	1
Camphor	62070	76-22-2	0.044–0.060 µg/L	Flavor, odorant, ointments.	1
d-Limonene	62073	5989-27-5	0.08–0.14 µg/L	Fragrance in aerosols, antimicrobial, antiviral, and fungicide.	1, 2, 3

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Personal-use products (flavors and fragrances) (SH1433)—Continued					
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	62075	1222-05-5	0.052 µg/L	Musk fragrance, persistent and widespread, in groundwater.	1, 2, 3
Indole	62076	120-72-9	0.08 µg/L	Inert pesticide ingredient, fragrance, in coffee.	1, 2, 3
Isoborneol	62077	124-76-5	0.08–0.18 µg/L	Fragrance in perfumery, in disinfectants.	1, 2, 3
Isoquinoline	62079	119-65-3	0.046–0.400 µg/L	Flavors and fragrances.	1, 2, 3
Menthol	62080	89-78-1	0.32–0.40 µg/L	Cigarettes, cough drops, liniment, mouthwash.	1, 2, 3
Pesticides (SH1433)					
1,4-Dichlorobenzene	34572	106-46-7	0.04 µg/L	Used as an ingredient in urinal cakes, deodorant, disinfectant and chemical intermediate in addition to uses as a general insecticide, moth repellent, fumigant, and germicide.	1
Bromacil	04029	314-40-9	0.36–1.00 µg/L	Uracil compound used as a herbicide for brush control on noncropland areas. Greater than 80 percent noncrop usage on grass.	4
Carbaryl	82680	63-25-2	0.16–1.00 µg/L	Broad-spectrum carbamate insecticide used to control over 100 species of insects on citrus trees, fruit trees, nut trees, cotton, vegetables, forests, lawns, ornamentals, shade trees, and other crops, as well as poultry, livestock, and pets.	8
Carbazole	62071	86-74-8	0.030–0.040 µg/L	Insecticide, manufacturing of dyes, explosives, and lubricants.	2, 3
Chlorpyrifos	38933	2921-88-2	0.12–0.16 µg/L	Broad-spectrum, chlorinated organophosphate (OP) insecticide, acaricide, and nematicide used on grain, cotton, field, fruit, nut, and vegetable crops, as well as on lawns, ornamental plants, and golf course turf. Also registered for direct use on sheep and turkeys, for horse site treatment, dog kennels, domestic dwellings, farm buildings, storage bins, and commercial establishments as well as nonstructural wood treatments including processed wood products, fenceposts, and utility poles. Can lead to neurotoxicity.	2, 3
Diazinon	39572	333-41-5	0.08–0.16 µg/L	Nonsystemic organophosphate Restricted Use Pesticide (RUP) for professional pest control operator use only. Greater than 40 percent nonagricultural usage on a wide variety of trees, fruit, row crops, and vegetables. No longer used on golf courses/sod farms because of die-offs of birds that often congregated in these areas. Classified as moderately toxic.	1, 2, 3

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Pesticides (SH1433)—Continued					
Metalaxyl	50359	57837-19-1	0.12 µg/L	Systemic benzenoid compound used as a pesticide, herbicide, and fungicide. Applied as either a foliar spray, soil treatment, or a seed treatment to control downy mildews. Used on many different food crops, including tobacco, ornamentals, conifer, and golf course/turf applications.	1, 2, 3
Metolachlor	39415	51218-45-2	0.028–0.080 µg/L	Pre-emergent herbicide used to control certain broadleaf and annual grassy weeds in row crops, fruit and nut trees, highway rights-of-way and woody ornamentals. General-use pesticide that is an indicator of agricultural drainage.	1, 2, 3
N,*N*-diethyl-*meta*-toluamide (DEET)	62082	134-62-3	0.06 µg/L	Insecticide, urban uses, mosquito repellent.	1, 2, 3
Prometon	04037	1610-18-0	0.12–0.20 µg/L	Herbicide, noncrop only, applied prior to blacktop.	1, 2, 3
Industrial wastewater compounds (SH1433)					
3-*tert*-Butyl-4-hydroxyanisole (BHA)	62059	25013-16-5	0.60–8.0 µg/L	Used as an antioxidant and preservative in food, animal feed, and rubber.	1
5-Methyl-1H-benzotriazole	62063	136-85-6	0.08 µg/L	Antioxidant in antifreeze and deicers.	1
Benzophenone	62067	119-61-9	0.08 µg/L	Fixative for perfumes and soap.	1
Isophorone	34409	78-59-1	0.032–0.11 µg/L	Used as solvent for paints, tin coatings, agricultural chemicals, and synthetic resins; excellent solvent for vinyl resins, cellulose esters, and ethers, pesticides, storing lacquers; pesticide manufacturing. Solvent for lacquer, plastic, oil, silicon, resin.	1, 2, 3
Isopropylbenzene	62078	98-82-8	0.20–0.30 µg/L	Manufacturing phenol/acetone, fuels and paint thinner.	1, 2, 3
Methyl salicylate	62081	119-36-8	0.044–0.100 µg/L	Liniment, food, beverage, UV-absorbing lotion.	1, 2, 3
p-Cresol	62084	106-44-5	0.08–0.18 µg/L	Wood preservative.	1, 2, 3
Tetrachloroethylene	34476	127-18-4	0.12 µg/L	Used in dry cleaning of fabrics.	1, 2, 3
Tribromomethane (Bromoform)	34288	75-25-2	0.10 µg/L	Was used as a solvent, sedative, and flame retardant, today mainly used as a laboratory reagent.	7
Triethyl citrate (ethyl citrate)	62091	77-93-0	0.16–0.40 µg/L	Cosmetics, pharmaceuticals.	1, 2, 3
Disinfection compounds (SH1433)					
Phenol	34466	108-95-2	0.16–1.40 µg/L	Antiseptic and disinfectant. Used in pharmaceuticals, germicidal paints, dyes, indicators, slimicide, laboratory reagents; phenolic resins; epoxy resins (Bisphenol A [BPA]), nylon-6, 2,4-D. Also used as a solvent for refining lubricating oils, preparation of acids and other compounds.	1, 2, 3
Triclosan	62090	3380-34-5	0.20 µg/L	Disinfectants, antimicrobial (concern for aquired microbial resistance).	1, 2, 3

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Polycyclic aromatic hydrocarbons (SH1433)					
1-Methylnaphthalene	62054	90-12-0	0.022–0.040 µg/L	2–5 percent of gasoline, diesel fuel, or crude oil.	1
2,6-Dimethylnaphthalene	62055	581-42-0	0.06–0.12 µg/L	Present in diesel/kerosene (trace in gasoline).	1
2-Methylnaphthalene	62056	91-57-6	0.036–0.040 µg/L	2–5 percent of gasoline, diesel fuel, and crude oil. Used in organic synthesis and insecticides.	1
9,10-Anthraquinone	62066	84-65-1	0.16 µg/L	Used to make dyes, used to bleach pulp for paper making.	4
Anthracene	34221	120-12-7	0.010–0.040 µg/L	Wood preservative, component of coal tar pitch volatiles, diesel, or crude oil; used in dyes, preparation of phenanthrene, carbazole, anthraquinone, and insecticides, organic semiconductor research.	1
Benzo[*a*]pyrene	34248	50-32-8	0.05–0.08 µg/L	Regulated PAH and combustion byproduct, component of coal tar pitch volatiles, and used in cancer research.	4
Fluoranthene	34377	206-44-0	0.024–0.040 µg/L	Component of coal tar and asphalt (only traces in gasoline or diesel fuels); used as a research chemical. Combustion product.	2, 3
Naphthalene	34443	91-20-3	0.040 µg/L	PAH also known as ‘camphor tar’ and derived from coal tar or crude oil. Used as a fumigant and moth repellent, in preparation of pesticides, fungicides, dyes, detergents, wetting agents, synthetic resins, celluloids, preservatives, and lubricants. Major component (about 10 percent) of gasoline.	1, 2, 3
Phenanthrene	34462	85-01-8	0.016–0.040 µg/L	Used in explosives, dyes, biochemical research, synthesis of drugs, and organic synthesis; combustion product.	1, 2, 3
Pyrene	34470	129-00-0	0.040–0.042 µg/L	Research chemical derived from industrial and experimental coal gasification operations. Component of coal tar and asphalt (only traces in gasoline or diesel fuel).	1, 2, 3
Flame retardants and plasticizers (SH1433)					
Bisphenol A (BPA)	67304	80-05-7	0.10–0.20 µg/L	Plasticizer.	2, 3
Tributyl phosphate	62089	126-73-8	0.16 µg/L	Used as an extractant and a plasticizer.	1, 2, 3
Triphenyl phosphate	62092	115-86-6	0.12 µg/L	Plasticizer, resin, wax, finish, roofing paper.	1, 2, 3
Tris(2-butoxyethyl)-phosphate	62093	78-51-3	0.8 µg/L	Flame retardant.	1, 2, 3
Tris(2-chloroethyl)-phosphate	62087	115-96-8	0.10 µg/L	Plasticizer, flame retardant.	1, 2, 3
Tris(dichloroisopropyl)-phosphate	62088	13674-87-8	0.10–0.16 µg/L	Flame retardant.	2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

[CAS, Chemical Abstracts Service; LRL, laboratory reporting level; USGS, U.S. Geological Survey; SH, USGS National Water Quality Laboratory schedule; µg/L, micrograms per liter; LCAB, USGS Organic Geochemistry Research Laboratory liquid chromatography/mass spectrometry antibiotics schedule; N/A, not available or not applicable; pct, percent; µg/kg, micrograms per kilogram; α, alpha; β, beta; UV, ultraviolet]

Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Wastewater compounds in bed sediments (SH5433)					
Stanols and sterols (animal) in bed sediments (SH5433)					
3-β-Coprostanol	63170	360-68-9	500–2,000 µg/Kg	Compound found in human and carnivorous animal feces.	1
β-Stigmastanol	63186	19466-47-8	500–1,760 µg/Kg	Herbivore fecal indicator (digestion of sitosterol).	1
Cholesterol	63196	57-88-5	250–860 µg/Kg	An important structural molecule of animal cell membranes, precursor for steroid hormones, bile acids, and vitamin D.	1
Sterols (plant) in bed sediments (SH5433)					
β-Sitosterol	63185	83-46-5	500–1,060 µg/Kg	Plant sterol.	1
Detergents and detergent metabolites (SH5433)					
4-Cumylphenol	63173	599-64-4	22–176 µg/Kg	Nonionic detergent metabolite.	1
4-*n*-Octylphenol	63174	1806-26-4	22–176 µg/Kg	Nonionic detergent metabolite.	1
4-Nonylphenol (sum of all isomers)	63175	84852-15-3	350–2,640 µg/Kg	Nonionic detergent.	10
4-Nonylphenol diethoxylate (sum of all isomers) also known as NP2EO	63200	N/A	400–3,500 µg/Kg	Nonionic detergent metabolite; surfactant metabolite.	9
4-Nonylphenol monoethoxylate (sum of isomers)	63221	N/A	220–1,760 µg/Kg	Nonionic detergent or metabolite.	5
4-*tert*-Octylphenol diethoxylate	63201	N/A	22–176 µg/Kg	Nonionic detergent.	9
4-*tert*-Octylphenol monoethoxylate	63206	N/A	120–880 µg/Kg	Nonionic detergent.	9
4-*tert*-Octylphenol	63176	140-66-9	24–1,760 µg/Kg	Nonionic detergent metabolite.	5
Personal-use products (flavors and fragrances) (SH5433)					
Acetophenone	63178	98-86-2	70–530 µg/Kg	Fragrance in detergent and tobacco, flavor in beverages.	1
3-Methyl-1H-indole (Skatole)	63171	83-34-1	36–86 µg/Kg	Fragrance, stench in feces, and coal tar.	1
Acetyl-hexamethyl-tetrahydronaphthalene (AHTN)	63179	21145-77-7	22–171 µg/Kg	Used in cosmetics (except oral products).	1
Benzophenone	63184	119-61-9	22–176 µg/Kg	Fixative for perfumes and soap.	1
Camphor	63192	76-22-2	22–176 µg/Kg	Flavor, odorant, ointments.	1
d-Limonene	63203	5989-27-5	22–176 µg/Kg	Fragrance in aerosols, antimicrobials, antivirals, and fungicides.	1, 2, 3
Hexahydro-hexamethyl-cyclopenta-benzopyran (HHCB)	63209	1222-05-5	46–171 µg/Kg	Musk fragrance, persistent and widespread, in groundwater.	1, 2, 3
Indole	63210	120-72-9	70–170 µg/Kg	Inert pesticide ingredient, fragrance, in coffee.	1, 2, 3
Isoborneol	63211	124-76-5	24–176 µg/Kg	Fragrance in perfumery, in disinfectants.	1, 2, 3
Isoquinoline	63214	119-65-3	70–350 µg/Kg	Flavors and fragrances.	1, 2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Pesticides (SH5433)					
1,4-Dichlorobenzene	63163	106-46-7	24–176 µg/Kg	Used as an ingredient in urinal cakes, deodorant, disinfectant and chemical intermediate in addition to uses as a general insecticide, moth repellent, fumigant, and germicide.	1
Atrazine	63182	1912–24–9	40–350 µg/Kg	Selective triazine herbicide.	5
Bromacil	63189	314-40-9	220–1,760 µg/Kg	Uracil compound used as a herbicide for brush control on noncropland areas. Greater than 80 percent noncrop usage on grass.	4
Carbazole	63194	86-74-8	63–104 µg/Kg	Insecticide, manufacturing of dyes, explosives, and lubricants.	2, 3
Chlorpyrifos	63195	2921-88-2	22–176 µg/Kg	Broad-spectrum, chlorinated organophosphate (OP) insecticide, acaricide, and nematicide used on grain, cotton, field, fruit, nut, and vegetable crops, as well as on lawns, ornamental plants, and golf course turf. Also registered for direct use on sheep and turkeys, for horse site treatment, dog kennels, domestic dwellings, farm buildings, storage bins, and commercial establishments as well as nonstructural wood treatments including processed wood products, fenceposts, and utility poles. Can lead to neurotoxicity.	2, 3
Diazinon	63198	333-41-5	22–176 µg/Kg	Nonsystemic organophosphate Restricted Use Pesticide (RUP) for professional pest control operator use only. Greater than 40 percent nonagricultural usage on a wide variety of trees, fruit, row crops, and vegetables. No longer used on golf courses/sod farms because of die-offs of birds that often congregated in these areas. Classified as moderately toxic.	1, 2, 3
Menthol	63215	57837-19-1	22–176 µg/Kg	Cigarettes, cough drops, liniment, mouthwash.	1, 2, 3
Metolachlor	63218	51218-45-2	22–176 µg/Kg	Pre-emergent herbicide used to control certain broadleaf and annual grassy weeds in row crops, fruit and nut trees, highway rights-of-way and woody ornamentals. General-use pesticide that is an indicator of agricultural drainage.	1, 2, 3
N,N-diethyl-*meta*-toluamide (DEET)	63219	134-62-3	50–350 µg/Kg	Insecticide, urban uses, mosquito repellent.	1, 2, 3
Prometon	63226	1610-18-0	22–176 µg/Kg	Herbicide, noncrop only, applied prior to blacktop.	1, 2, 3
Industrial wastewater compounds (SH5433)					
3-*tert*-Butyl-4-hydroxyanisole (BHA)	63172	25013-16-5	70–510 µg/Kg	Used as an antioxidant and preservative in food, animal feed, and rubber.	1
Isophorone	63212	78-59-1	22–171 µg/Kg	Used as solvent for paints, tin coatings, agricultural chemicals, and synthetic resins; excellent solvent for vinyl resins, cellulose esters, and ethers, pesticides, storing lacquers; pesticide manufacturing. Solvent for lacquer, plastic, oil, silicon, resin.	1, 2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Industrial wastewater compounds (SH5433)—Continued					
Isopropylbenzene	63213	98-82-8	40–350 µg/Kg	Manufacturing phenol/acetone, fuels and paint thinner.	1, 2, 3
p-Cresol	63222	106-44-5	180–860 µg/Kg	Wood preservative.	1, 2, 3
Disinfection compounds (SH5433)					
Phenol	63225	108-95-2	50–313 µg/Kg	Antiseptic and disinfectant. Used in pharmaceuticals, germicidal paints, dyes, indicators, slimicide, laboratory reagents; phenolic resins; epoxy resins (Bisphenol A [BPA]), nylon-6, 2,4-D. Also used as a solvent for refining lubricating oils, preparation of acids and other compounds.	1, 2, 3
Triclosan	63232	3380-34-5	50–176 µg/Kg	Disinfectants, antimicrobial (concern for aquired microbial resistance).	1, 2, 3
Polycyclic aromatic hydrocarbons (SH5433)					
1-Methylnaphthalene	63165	90-12-0	24–176 µg/Kg	2–5 percent of gasoline, diesel fuel, or crude oil.	1
2,6-Dimethylnaphthalene	63167	581-42-0	36–90 µg/Kg	Present in diesel/kerosene (trace in gasoline).	1
2-Methylnaphthalene	63168	91-57-6	24–176 µg/Kg	2–5 percent of gasoline, diesel fuel, and crude oil. Used in organic synthesis and insecticides.	1
9,10-Anthraquinone	63181	84-65-1	34–86 µg/Kg	Used to make dyes, used to bleach pulp for paper making.	4
Anthracene	63180	120-12-7	32–86 µg/Kg	Wood preservative, component of coal tar pitch volatiles, diesel, or crude oil; used in dyes, preparation of phenanthrene, carbazole, anthraquinone, and insecticides, organic semiconductor research.	1
Benzo[*a*]pyrene	63183	50-32-8	32–171 µg/Kg	Regulated PAH and combustion byproduct, component of coal tar pitch volatiles and used in cancer research.	4
Fluoranthene	63208	206-44-0	32–79 µg/Kg	Component of coal tar and asphalt (only traces in gasoline or diesel fuels); used as a research chemical. Combustion product.	2, 3
Naphthalene	63220	91-20-3	24–176 µg/Kg	PAH also known as “camphor tar” and derived from coal tar or crude oil. Used as a fumigant and moth repellent, in preparation of pesticides, fungicides, dyes, detergents, wetting agents, synthetic resins, celluloids, preservatives, and lubricants. Major component (about 10 percent) of gasoline.	1, 2, 3
Phenanthrene	63224	85-01-8	41–171 µg/Kg	Used in explosives, dyes, biochemical research, synthesis of drugs, and organic synthesis; combustion product.	1, 2, 3
Pyrene	63227	129-00-0	50–100 µg/Kg	Research chemical derived from industrial and experimental coal gasification operations. Component of coal tar and asphalt (only traces in gasoline or diesel fuel).	1, 2, 3

Table 2. Human-health pharmaceuticals, antibiotics, steroidal hormones, stanols, sterols, and wastewater compounds analyzed in water and bed-sediment samples collected at study sites in or near Dallas, Texas, 2009–13.—Continued

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Compounds	Parameter code	CAS number	LRL	Description and (or) source	Reference
Flame retardants and platicizers (SH5433)					
2,2',4,4'-Tetrabromodiphenyl ether	63166	5436-43-1	22–176 µg/Kg	Textile and electronic flame retardant.	5
Bis(2-ethylhexyl) phthalate	63187	117-81-7	110–860 µg/Kg	Plasticizer.	5
Bisphenol A (BPA)	63188	80-05-7	22–176 µg/Kg	Plasticizer.	5
Diethyl phthalate	63202	84-66-2	40–350 µg/Kg	Plasticizer for polymers and resins.	5
Tributyl phosphate	63231	126-73-8	22–176 µg/Kg	Used as an extractant and a plasticizer.	1, 2, 3
Triphenyl phosphate	63234	115-86-6	22–176 µg/Kg	Plasticizer, resin, wax, finish roofing paper.	1, 2, 3
Tris(2-butoxyethyl) phosphate	63229	78-51-3	70–530 µg/Kg	Flame retardant.	1, 2, 3
Tris(2-chloroethyl) phosphate	63230	115-96-8	40–350 µg/Kg	Plasticizer, flame retardant.	1, 2, 3
Tris(dichloroisopropyl) phosphate	63235	13674-87-8	40–350 µg/Kg	Flame retardant.	2, 3

†Constituent preferred concentration determined by schedule as noted in description.

[1]Reif and others, 2012.

[2]Foreman and others, 2012.

[3]Furlong and others, 2008.

[4]Zaugg and others, 2007.

[5]Morace, 2012.

[6]Glassmeyer and others, 2005.

[7]Bender and others, 1999.

[8]Kelly and others, 2012.

[9]Jobling and Sumpter, 1993.

[10]Kolpin and others, 2002.

[11]Erickson and others, 2014.

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