

North Carolina Department of Environment and Natural Resources, the City and County of Durham, and the City of Raleigh, North Carolina

## **Water-Quality Characteristics Indicative of Wastewater in Selected Streams in the Upper Neuse River Basin, Durham and Orange Counties, North Carolina, from 2004 to 2013**



Open-File Report 2014–1215

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

**Cover:** Stream channel at the Eno River near Schley, North Carolina. Photograph by Gloria Ferrell.

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By G.M. Ferrell, Matthew S. Yearout, Barbara H. Grimes, Alexandria K. Graves, Sharon A. Fitzgerald, and Michael T. Meyer

North Carolina Department of Environment and Natural Resources, the City and County of Durham, and the City of Raleigh, North Carolina

Open File Report 2014–1215

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**U.S. Geological Survey**

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# Contents

Acknowledgments.....	xi
Abstract.....	1
Introduction.....	1
Purpose and Scope .....	3
Description of Study Area .....	3
Approach.....	5
Selection and Description of Study Sites .....	5
Previous Studies .....	20
Methods of Data Collection and Analysis .....	20
Sample Collection and Analysis .....	20
Quality Assurance and Quality Control of Laboratory Data.....	22
Effects of Centralized and Onsite Wastewater Treatment on Stream Quality.....	27
Quality of Streams and Wastewater Effluent, 2004–2008.....	27
Elemental Quality of Streambed Sediment, 2005.....	43
Water Quality During Stormflow Recession Periods, May 2012–January 2013.....	45
Summary and Conclusions.....	58
References Cited.....	58

## Figures

1. Movement of water through the groundwater system .....	2
2. Location of sampling sites in Durham and Orange Counties, North Carolina .....	4
3. Locations of river reach sampling sites on the Eno River, Orange County, North Carolina .....	6
4. Stream channel at the Eno River near Schley, North Carolina .....	8
5. Study area for Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina .....	9
6. Stream channel at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina.....	10
7. Study area for Nancy Rhodes Creek above Cole Mill Road near Durham, North Carolina .....	11
8. Study area for Black Meadow Run at Argonne Drive near Durham, North Carolina .....	12
9. Rock-lined stream channel upstream from the study site Black Meadow Run at Argonne Drive near Durham, North Carolina .....	13
10. Study area for the Eno River tributary below Clover Hill Place near Durham, North Carolina .....	14
11. Stream channel at Eno River tributary below Clover Hill Place near Durham, North Carolina .....	15
12. Study area for Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina .....	16

13.	Study area for Crooked Creek tributary at Greenbay Drive near Durham, North Carolina .....	17
14.	Crooked Creek tributary at Greenbay Drive near Durham, North Carolina .....	18
15.	Study area for Cabin Branch tributary at Paragon Circle near Durham, North Carolina .....	19
16.	Distributions of concentrations of selected dissolved ions and nutrients in surface-water samples from river reach sites in Orange County, North Carolina, December 2004 to September 2008 .....	28
17.	Temporal variation in concentrations of dissolved ammonia nitrogen, organic nitrogen, and nitrate plus nitrite nitrogen in water samples from the Eno River upstream and downstream from the Town of Hillsborough Wastewater Treatment Facility outfall, and effluent samples from the town of Hillsborough Water Treatment Plant, Hillsborough, North Carolina December 2004 to September 2008 .....	29
18.	Distributions of concentrations of selected dissolved nutrients and ions nitrate plus nitrite, ammonia plus organic nitrogen, sulfate as sulfur, magnesium, and sodium, in streamwater samples from small stream sites in Durham and Orange Counties, North Carolina, December 2004 to October 2008.....	31
19.	Temporal variation in concentrations of dissolved ammonia nitrogen, organic nitrogen, and nitrate plus nitrite nitrogen in water samples from small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to October 2008 .....	31
20.	Elemental concentrations of organic carbon, arsenic, chromium, cobalt, copper, nickel, lead, and vanadium in streambed-sediment samples from sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, June 1, 2005 .....	45
21.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina, May 12–24, 2012.....	47
22.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina, September 16–25, 2012.....	48
23.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Black Meadow Run at Argonne Drive near Durham, North Carolina, May 14–22, 2012.....	49
24.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Black Meadow Run at Argonne Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.....	50
25.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, May 14–21, 2012 .....	51
26.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.....	52
27.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, October 1–21, 2012 .....	53
28.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.....	54

29.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, September 16–25, 2012.....	55
30.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, October 1–23, 2012.....	56
31.	Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, December 21, 2012, to January 5, 2013.....	57

## Tables

1.	Characteristics of study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina .....	7
2.	Characteristics of study sites in the Nancy Rhodes Creek above Cole Mill Road near Durham, North Carolina, catchment .....	12
3.	Characteristics of study sites in the Black Meadow Run at Argonne Drive near Durham, North Carolina, catchment .....	13
4.	Characteristics of study sites in the Eno River tributary below Clover Hill Place near Durham, North Carolina, catchment .....	15
5.	Characteristics of study sites in the Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, catchment .....	18
6.	Characteristics of study sites in the Cabin Branch tributary at Paragon Circle near Durham, North Carolina, catchment .....	20
7.	Analytes measured in surface-water, wastewater-effluent, and streambed-sediment samples collected in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2013.....	Separate Excel file
8.	Organic wastewater compounds analyzed in surface-water and wastewater-effluent samples collected in the upper Neuse River Basin, Orange and Durham Counties, North Carolina, December 2004 to September 2008.....	Separate Excel file
9.	Antibiotics, antibiotic metabolites, and pharmaceutical compounds analyzed in surface-water and wastewater-effluent samples collected in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to September 2008 .....	21
10.	Frequency and concentrations of analytes detected in field and source solution blanks.....	24
11.	Surrogate recoveries for organic wastewater compounds in blank water, surface-water, and wastewater-effluent samples, 2004–2008 .....	24
12.	Variability of replicate analyte determinations.....	25
13.	Quality-control data for analyses of antibiotics, antibiotic metabolites, and pharmaceutical compounds .....	26
14.	Recovery of hormones in samples, May 2012 to January 2013 .....	27
15.	Concentrations of selected dissolved ions, nutrients, and metals in surface-water and wastewater-effluent samples from river reach sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.....	30

16.	Concentrations of selected dissolved ions, nutrients, and metals in surface-water samples from the small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.....	33
17.	Detections of organic wastewater compounds in samples from the river reach study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to June 2005.....	35
18.	Concentrations of antibiotics, antibiotic metabolites, and pharmaceutical compounds detected in surface-water and wastewater-effluent samples from river reach sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.....	36
19.	Detections of organic wastewater compounds in samples from the small stream study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.....	37
20.	Concentrations of antibiotics, antibiotic metabolites, and pharmaceutical compounds detected in surface-water samples from small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.....	38
21.	Values for optical brighteners, methylene-blue active substances, and fecal bacteria at study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, January to June, 2005.....	40
22.	Bacteriological data for samples of surface water and treated effluent at the river reach sites in the upper Neuse River Basin, Orange County, North Carolina, June to September, 2008 .....	40
23.	Bacteriological data for samples of surface water at the small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, April to September, 2008.....	41
24.	Presence of optical brighteners in samples of surface water and wastewater effluent from study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, April to June, 2008 .....	43
25.	Concentrations of selected elements in streambed sediments collected on June 1, 2005, from study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina .....	44



## Appendixes 1–10 *(available at <http://pubs.usgs.gov/of/2014/1215/>)*

1. Description of research method for analysis of hormones in water ..... MS Excel file
2. Description of research method for analysis of optical brighteners  
in water..... MS Excel file
3. Quality assurance data for surface-water and wastewater effluent samples  
collected at sites in the upper Neuse River Basin, Durham and  
Orange Counties, North Carolina, December 2004 to January 2013 ..... MS Excel file
4. Quality assurance data for streambed-sediment samples collected at sites  
in the upper Neuse River Basin, Durham and Orange Counties,  
North Carolina, June 2005..... MS Excel file
5. Water quality data for surface-water and wastewater effluent samples  
collected at sites in the upper Neuse River Basin, Durham and  
Orange Counties, North Carolina, 2004–2008..... MS Excel file
6. Elemental data for streambed-sediment samples collected at sites  
in the upper Neuse River Basin, Durham and Orange Counties,  
North Carolina, June 2005..... MS Excel file
7. Water-quality data for surface-water samples collected at sites in the  
upper Neuse River Basin, Durham and Orange Counties, North Carolina,  
2012–2013 ..... MS Excel file
8. Fluorescence results for sites in the upper Neuse River Basin, Durham  
and Orange Counties, North Carolina, 2012–2013 ..... MS Excel file
- 8a. Fluorescence results at Rhodes Creek tributary above NC Hwy 751 near  
Durham, North Carolina, during May and September 2012..... MS Excel file
- 8b. Fluorescence results at Black Meadow Run at Argonne Drive near Durham,  
North Carolina, and miscellaneous sites in that watershed during  
May and December 2012 and January 2013 ..... MS Excel file
- 8c. Fluorescence results at Sevenmile Creek tributary at Inverness Drive near  
Durham, North Carolina, during May and December 2012 and  
January 2013..... MS Excel file
- 8d. Fluorescence results at Crooked Creek tributary at Greenbay Drive near  
Durham, North Carolina, and at miscellaneous sites in that watershed  
during October 2012 and December 2013 to January 2013..... MS Excel file
- 8e. Fluorescence results at Cabin Branch tributary at Paragon Circle near  
Durham, North Carolina, and miscellaneous sites in that watershed  
during September and October 2012 and  
December 2012 to January 2013 ..... MS Excel file
- 8f. All replicate results for fluorescence from all sites during the  
2012–2013 study period..... MS Excel file
9. Optical brightener data for surface-water samples collected at sites  
in the upper Neuse River Basin, Durham and Orange Counties,  
North Carolina, 2012–2013..... MS Excel file
- 9a. Optical brightener detections at Rhodes Creek tributary above  
NC Hwy 751 near Durham, North Carolina, during 2012 to 2013 ..... MS Excel file
- 9b. Optical brightener detections among analytical replicates at Rhodes Creek  
tributary above NC Hwy 751 near Durham, North Carolina, during  
2012 to 2013..... MS Excel file
- 9c. Optical brightener detections at Black Meadow Run at Argonne Drive near  
Durham, North Carolina, and miscellaneous sites in that watershed  
during 2012 to 2013 ..... MS Excel file

- 9d. Optical brightener detections among analytical and field replicates at Black Meadow Run at Argonne Drive near Durham, North Carolina, and at miscellaneous sites in that watershed during 2012 to 2013..... MS Excel file
- 9e. Optical brightener detections at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, during 2012 to 2013 ..... MS Excel file
- 9f. Optical brightener detections among analytical and field replicates at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, during 2012 to 2013 ..... MS Excel file
- 9g. Optical brightener detections at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, and at miscellaneous sites in that watershed during 2012 to 2013 ..... MS Excel file
- 9h. Optical brightener detections among analytical and field replicates at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, during 2012 to 2013 ..... MS Excel file
- 9i. Optical brightener detections at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, and miscellaneous sites in that watershed during 2012 to 2013 ..... MS Excel file
- 9j. Optical brightener detections in analytical and field replicates at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, during 2012 to 2013 ..... MS Excel file
- 10. Stage data for sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2012–2013..... MS Excel file
- 10a. Stage data for Rhodes Creek tributary above NC Hwy 751 near Durham, North Carolina, May 12–23, 2012..... MS Excel file
- 10b. Stage data for Rhodes Creek tributary above NC Hwy 751 near Durham, North Carolina, September 12–26, 2012 ..... MS Excel file
- 10c. Stage data for Black Meadow Run at Argonne Drive near Durham, North Carolina, May 10–23, 2012..... MS Excel file
- 10d. Stage data for Black Meadow Run at Argonne Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013..... MS Excel file
- 10e. Stage measurements for Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, May 13–24, 2012 ..... MS Excel file
- 10f. Stage measurements for Sevenmile Creek at Inverness Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013..... MS Excel file
- 10g. Stage data for Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, October 1–19, 2012 ..... MS Excel file
- 10h. Stage data for Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013..... MS Excel file
- 10i. Stage data for Cabin Branch tributary at Paragon Circle near Durham, North Carolina, September 12–30, 2012..... MS Excel file
- 10j. Stage data for Cabin Branch tributary at Paragon Circle near Durham, North Carolina, October 1–23, 2012 ..... MS Excel file
- 10k. Stage data for Cabin Branch tributary at Paragon Circle near Durham, North Carolina, December 21, 2012, to January 4, 2013..... MS Excel file

## Conversion Factors, Datums, and Selected Abbreviations

### Inch/Pound to SI

Multiply	By	To obtain
Length		
inch (in.)	2.54	centimeter (cm)
inch (in.)	25.4	millimeter (mm)
foot (ft)	0.3048	meter (m)
mile (mi)	1.609	kilometer (km)
Area		
square mile (mi <sup>2</sup> )	259.0	hectare (ha)
square mile (mi <sup>2</sup> )	2.590	square kilometer (km <sup>2</sup> )
Volume		
gallon (gal)	3.785	liter (L)
gallon (gal)	0.003785	cubic meter (m <sup>3</sup> )
gallon (gal)	3.785	cubic decimeter (dm <sup>3</sup> )
Flow rate		
cubic feet per second (ft <sup>3</sup> /s)	0.028317	cubic meter per second (m <sup>3</sup> /s)
million gallons per day (Mgal/d)	0.04381	cubic meter per second (m <sup>3</sup> /s)

### SI to Inch/Pound

Multiply	By	To obtain
Length		
nanometer (nm)	0.00000003937	inch (in.)
micron (μm)	0.00003937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
centimeter (cm)	0.3937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
Volume		
microliter (μL)	0.00003382	ounce, fluid (fl. oz)
milliliter (mL)	0.03382	ounce, fluid (fl. oz)
liter (L)	33.82	ounce, fluid (fl. oz)
liter (L)	2.113	pint (pt)
liter (L)	1.057	quart (qt)
liter (L)	0.2642	gallon (gal)
liter (L)	61.02	cubic inch (in <sup>3</sup> )
Mass		
nanogram (ng)	3.527 x 10 <sup>-11</sup>	ounce, avoirdupois (oz)
microgram (μg)	3.527 x 10 <sup>-8</sup>	ounce, avoirdupois (oz)
milligram (mg)	0.00003527	ounce, avoirdupois (oz)
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)
Flow rate		
milliliters per minute (mL/min)	0.050853	cubic feet per day (ft <sup>3</sup> /d)

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

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

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

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

## Abbreviations

LC/MS/MS,	liquid chromatography/tandem mass spectrometry
NCDENR,	North Carolina Department of Environment and Natural Resources
NWQL,	National Water Quality Laboratory
OGRL,	Organic Geochemistry Research Laboratory
OWWC,	organic wastewater compound
RDS,	relative standard deviation
RPD,	relative percent difference
USGS,	U.S. Geological Survey
WWTP,	wastewater treatment plant

## Acknowledgments

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# Water-Quality Characteristics Indicative of Wastewater in Selected Streams in the Upper Neuse River Basin, Durham and Orange Counties, North Carolina, from 2004 to 2013

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## Abstract

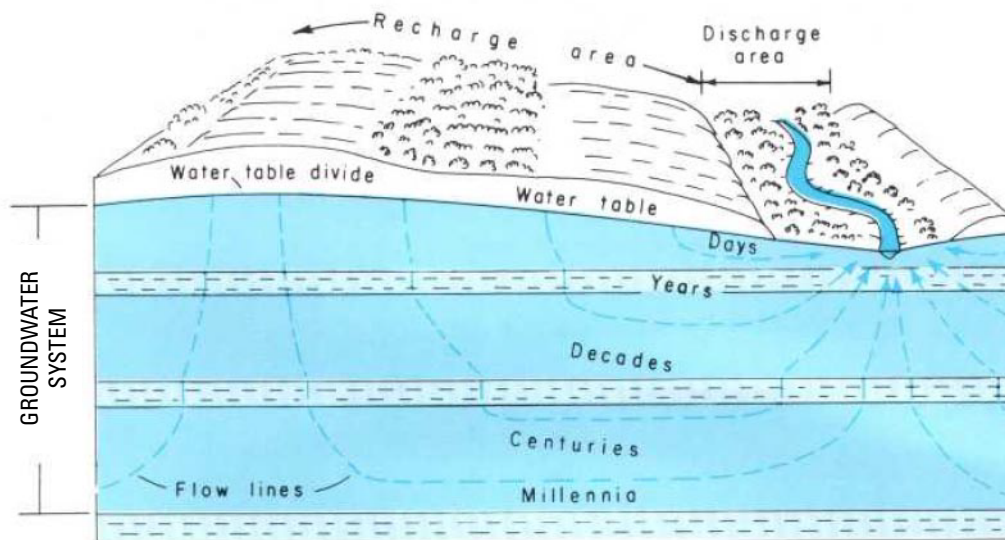
Data were collected during three time periods to assess the effects of wastewater treatment and disposal practices on the occurrence of selected contaminants indicative of wastewater in the upper Neuse River Basin, North Carolina. The first phase of data collection, December 2004 to June 2005, and the second phase, April to October 2008, addressed the effects of point and nonpoint sources of wastewater effluent on stream quality during baseflow conditions. Point-source effects were assessed by sampling a municipal wastewater treatment plant outfall and sites on the Eno River upstream and downstream from the outfall. Water-quality data suggest that the wastewater treatment plant effluent contributed to increases in concentrations of nitrogen and carbamazepine at the downstream site. Nonpoint source effects were assessed by sampling seven small streams that drained an undeveloped area and residential areas served by either centralized or onsite wastewater treatment systems. Samples were analyzed for inorganic constituents, including nutrients, ions, and metals; organic compounds considered indicative of wastewater contamination; antibiotics, optical brighteners, and fecal coliform bacteria. Hypothesized differences in water quality between the sites with primarily centralized and onsite wastewater treatment were not apparent, likely due to the relatively large heterogeneity of the sites within each category.

During the third phase of data collection, May 2012 to January 2013, data were collected to address the suitability of optical brighteners as tracers of wastewater in small streams during streamflow recession. Samples were collected at five small streams following periods of rainfall and analyzed for optical brighteners, specific conductance, nutrients, and selected hormones. Optical brighteners were absent in the undeveloped catchment but were present in the recession period after rainfall events in catchments with centralized though possibly leaky sewage treatment and

areas with onsite treatment. Sand filter systems in areas with onsite treatment appear to change the effluent flow and retention characteristics such that optical brighteners were present both before and after rainfall events. Nitrate plus nitrite, as nitrogen concentrations in samples from this last study phase generally were larger than those collected during baseflow conditions in the previous phases of this study.

## Introduction

Wastewater effluent has the potential to affect stream quality. Centralized wastewater treatment systems frequently discharge treated effluent directly to streams, whereas onsite wastewater treatment systems typically release treated effluent to the subsurface where interactions with soil and vegetation serve to further treat the effluent and reduce the impact of the effluent on groundwater and streams. The effectiveness of these interactions, which include biological transformation and uptake of nutrients and adsorption of inorganic and organic components of effluent, are highly variable and primarily depend upon the geologic and hydrologic setting and soil characteristics. Physical characteristics of a site, including geology, topography, depth to groundwater, and soil type, as well as seasonal and climatic conditions, affect the movement and transformation of treated wastewater from onsite systems into streams. Although effluent from properly functioning septic systems is not directly discharged to streams, treated wastewater discharged to the subsurface moves laterally and vertically into the shallow groundwater system and ultimately is discharged to streams (Heath, 1983; fig. 1). Onsite wastewater systems are designed to remove traditional wastewater contaminants, such as nutrients and microbes; however, their effectiveness in degrading or removing other contaminants, such as pharmaceutical and hormonally active compounds, is not well understood.



**Figure 1.** Movement of water through the groundwater system (from Heath, 1983).

Effluent from municipal wastewater treatment plants (WWTPs) is considered a major source of pharmaceutical and hormonally active compounds in surface waters (Ternes, 1998; Daughton and Ternes, 1999; Heberer, 2002; Kolpin and others, 2002). Little is known about the contribution of domestic wastewater from decentralized, or onsite, wastewater treatment systems. Septic systems are the most common type of onsite wastewater treatment system and are used for the treatment of domestic wastewater by about 30 percent of the U.S. population (U.S. Census Bureau, 2001). Sand-filter treatment systems, although less common than septic systems, are used for onsite wastewater treatment in parts of the study area where soil conditions are unsuitable for septic systems. Sand filter system designs have changed over time and range from unlined systems that do not have a surface discharge to lined, recirculating systems with chlorination units to treat discharging effluent. Onsite wastewater treatment systems are common in rural and low-density suburban areas, whereas centralized wastewater treatment systems are common in urban and high-density suburban areas. Properly functioning septic systems effectively treat domestic wastewater; however, effectiveness decreases over time if systems are not properly maintained (U.S. Environmental Protection Agency, 2002a). Reported estimates of the effective lifespan of septic systems range from 11 to more than 30 years (Siegrist and others, 2001). About half of the septic systems in the United States are more than 30 years old (U.S. Census Bureau, 2001). Failing septic systems in older residences and suburban areas are considered a major threat to urban water quality in North Carolina (Jolley, 2003).

In addition to the effects of wastewater discharged directly to streams, centralized wastewater treatment systems can have detrimental effects on water quality because of leaking or overflowing sewer lines. Nationwide, an estimated 3 to 10 billion gallons of untreated sewage is discharged annually through leaking or overflowing sewer lines (U.S. Environmental Protection Agency, 2002b). Leakage from sewer lines is considered to be a major source of groundwater recharge in urban areas (Barrett and others, 1999; Ellis and Revitt, 2002) and has been linked to groundwater contamination (Wolf and others, 2004; Ellis, 2006). Rates of leakage from gravity flow sewer lines are highly variable, and leaks may be self-sealing as a result of deposition of sediment and other materials transported in sewage (Ellis and others, 2003; Blackwood and others, 2005). Organic wastewater contaminant, nutrient, optical brightener, and bacteria data suggest that sewer line leakage affected water quality in a residential catchment served by centralized wastewater treatment (Ferrel and Grimes, 2014).

The movement of treated wastewater from onsite systems to streams is affected by seasonal and climatic conditions as well as by the physical and geologic setting of the site. The rate at which treated wastewater moves to streams depends on the gradient and the transmissivity of soil and underlying rock. Infiltration of rainfall will facilitate the movement of treated wastewater that has been discharged to the subsurface and will decrease the residence time within the drainfield and soil. Therefore, the greatest effects of wastewater from onsite-treatment systems on stream quality should occur as the stream returns to baseflow conditions immediately following precipitation, when the quantity



of groundwater influx into the stream is large and dilution associated with runoff is minimal. The effects of onsite wastewater treatment systems on stream quality should also be greatest during the winter when temperatures and biological activity are low.

Optical brighteners, which are detergent additives that fluoresce under the visible spectrum to enhance the appearance of white fabrics, have been used as tracers of wastewater in streams (Poiger and others, 1998; Stoll and Giger, 1998; Boving and others, 2004; Hyer, 2007) and as indicators of fecal contamination in stormwater samples (Sankararamakrishnan and Guo, 2005). Because these compounds do not readily degrade in the subsurface, they should be present in groundwater as it discharges to streams.

This study was initiated in December 2004 in cooperation with the North Carolina Department of Environment and Natural Resources (NCDENR), Division of Environmental Health. The study was funded by a grant through the U.S. Environmental Protection Agency's Clean Water Act Section 319, the Nonpoint Source Management Program, and the U.S. Geological Survey (USGS) Cooperative Funding Program to assess the effects of wastewater treatment practices on stream quality. An additional investigation, initiated in 2012, in cooperation with the Durham County Health Department, the City of Durham Stormwater Services Division, the City of Raleigh, and the North Carolina Department of Environment and Natural Resources, Division of Environmental Health, was conducted to characterize the effects of wastewater treatment practices on stream quality during periods of streamflow recession.

In addition to evaluating the contribution of wastewater treatment systems to the distribution of emerging contaminants in streams, the contribution of nitrogen and phosphorus were evaluated. Because nutrient levels in Falls Lake Reservoir, the primary source of drinking water for the City of Raleigh, are conducive to excess algal growth, concern has arisen regarding the nutrient content of inflowing streams. In the 2010 303(d) list (North Carolina Department of Environment and Natural Resources [NCDENR], 2010), water quality in Falls Lake Reservoir was reported as impaired because of excessive levels of chlorophyll *a*. In 2005, the North Carolina General Assembly passed Session Law 2005–190, the Clean Lakes Act, which directed the Environmental Management Commission to study water-supply reservoirs and to develop and implement a nutrient management strategy for reservoirs that are listed as impaired. In response to this legislation, the NCDENR performed a watershed risk assessment (NCDENR, 2009a) and developed a chlorophyll *a* model (NCDENR, 2009b) for the Falls Lake Reservoir. Septic systems were estimated to be the source of about 28 percent of the nitrogen and 12 percent of the phosphorus entering the reservoir from the Eno River Watershed (NCDENR, 2009a), the watershed in which the sites for this study are located. In addition, the present study also evaluated optical brighteners as wastewater indicators,

which has potential application for locating failing septic systems as well as leaking sewer lines.

## Purpose and Scope

The purpose of this report is to present and summarize data collected at 10 study sites in the upper Neuse River Basin in Durham and Orange Counties, North Carolina, from 2004 to 2013. These data were collected to assess the effects of wastewater treatment practices on stream quality and include analyses of nutrients, major ions, bacteria, optical brighteners, antibiotics, hormones, and organic compounds considered indicative of wastewater. An experimental method developed for analysis of optical brighteners in surface water also is presented.

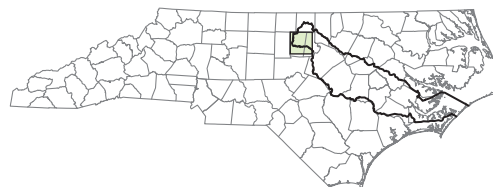
## Description of Study Area

The study area is in the upper Neuse River Basin in Orange and Durham Counties in North Carolina (fig. 2). In 2007, the Neuse River was listed as one of the most endangered rivers in America because of eutrophication attributed to increased waste loads as a result of rapid urban development and agricultural activities (American Rivers Foundation, 2007). Several municipalities, including Hillsborough, Durham, Raleigh, Clayton, Smithfield, Selma, and Goldsboro, obtain drinking-water supplies from streams or reservoirs in the Neuse River Basin.

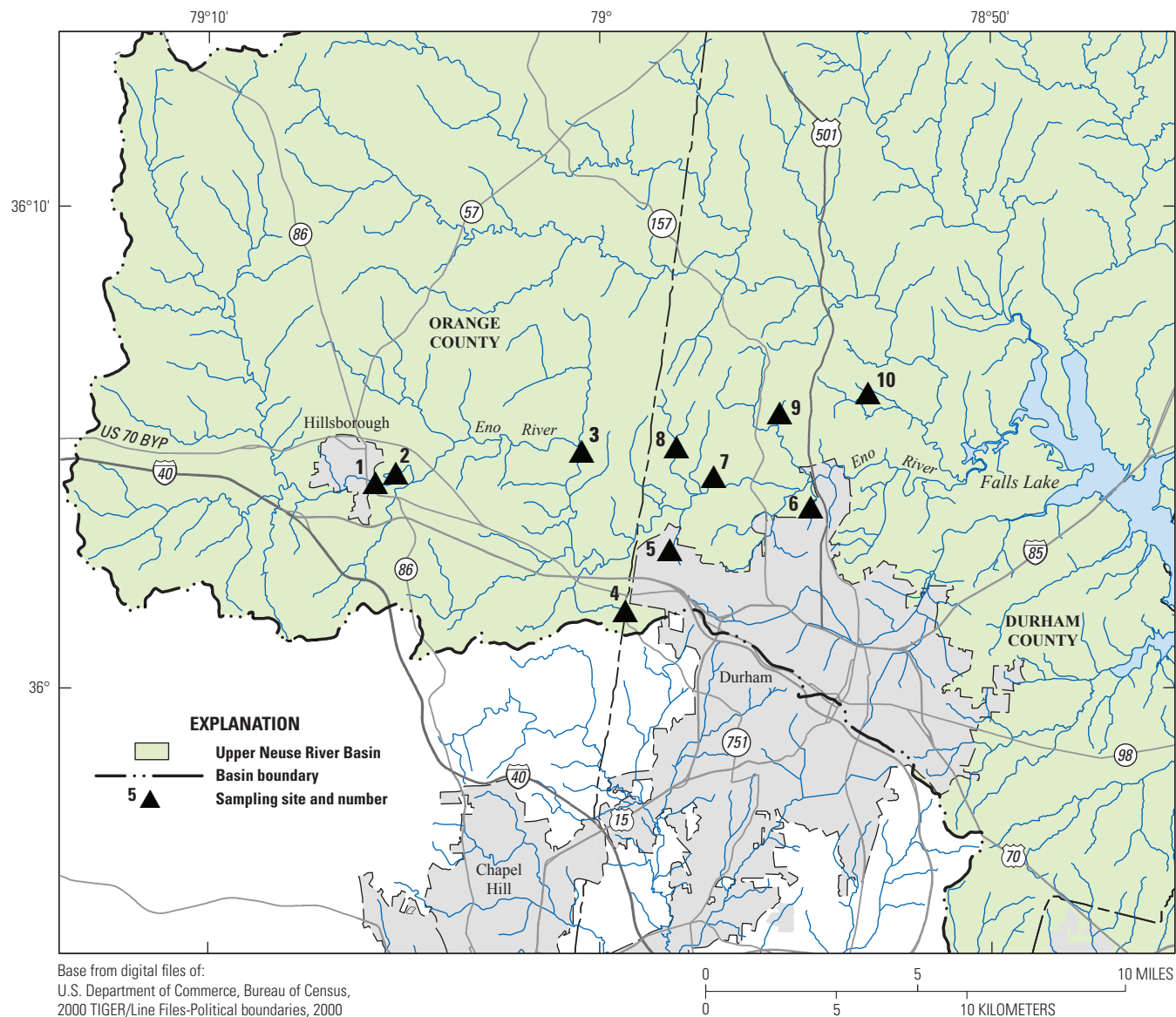
The lower reaches of the Neuse River form the Neuse River Estuary, which flows into Pamlico Sound. The estuary constitutes one of the major fish and shellfish nurseries and foraging areas of the Atlantic coast (Copeland and others, 1991). In an effort to protect these aquatic resources, waters of the Neuse River Basin were designated as nutrient sensitive, and in 1988 the North Carolina General Assembly (15A NCAC 2B.0232) adopted a goal of reducing nitrogen loads to the Neuse River Estuary by 30 percent. Although the effects of pharmaceutical and hormonally active compounds on the Neuse River Estuary are not documented, studies have identified these types of compounds in other marine and estuarine systems (Weigel and others, 2002; Atkinson and others, 2003; Oros and others, 2003; Thomas and Hilton, 2004) and linked their occurrence to adverse effects on marine and estuarine biota (Costanzo and others, 2005; Hoeger and others, 2005; Nunes and others, 2005).

Population growth in the upper Neuse River Basin has resulted in rapid residential expansion into areas lacking the infrastructure required for centralized wastewater treatment systems. As a result, increasing numbers of onsite systems are being installed in the basin. An estimated 30 and 72 percent of the residences in Durham and Orange Counties, respectively, use onsite wastewater treatment (NCDENR, Division of Environmental Health, 2003).





Location of study area in the upper Neuse River Basin in North Carolina



Base from digital files of:  
 U.S. Department of Commerce, Bureau of Census,  
 2000 TIGER/Line Files-Political boundaries, 2000  
 Hydrography from National Dataset USGS (2012) 1:24,000 scale

**Figure 2.** Location of sampling sites in Durham and Orange Counties, North Carolina.

## Approach

Data were collected during three study phases to assess the effects of centralized and onsite wastewater treatment practices on streams in the upper Neuse River Basin in Durham and Orange Counties, North Carolina. The first phase of data collection, December 2004 to June 2005, included collection of samples at sites potentially affected by point and nonpoint sources of wastewater. These samples were analyzed for wastewater indicators including selected nutrients, major ions, metals, and fecal bacteria as well as select trace organic compounds commonly referred to as emerging contaminants. The emerging contaminants analyzed during phase 1 included a suite of organic compounds considered indicative of wastewater such as fragrances, detergents, fuel components, pesticides, solvents, flame retardants, and plasticizers among others; selected antibiotic and pharmaceutical compounds; and optical brighteners. Elemental concentrations of streambed sediments were measured to determine if there were differences in metals concentrations related to the method of wastewater disposal.

The second phase of data collection, from April to October 2008, was a continuation of the first phase with an effort to sample under higher baseflow conditions than existed during phase 1. The sampling network was expanded to include tributaries within the catchments of the streams sampled during the first phase of the study. Also, sample analysis was expanded to include the endogenous hormone 17- $\beta$ -estradiol.

The third phase of data collection evaluated the presence of optical brighteners and selected nutrients during streamflow recession periods in a subset of the tributaries sampled in the second phase to assess the effects of wastewater on streams during periods of groundwater discharge. Samples were collected for 6 to 10 days following precipitation events that were considered likely to produce groundwater discharge as opposed to primarily runoff conditions. Optical brightener and bacteriological data from all phases of this study were used to evaluate the relative effects of wastewater within the study catchments and to locate possible locations of failing onsite wastewater treatment systems and leaking sewer lines.

## Selection and Description of Study Sites

Sites are in areas underlain by metamorphic and igneous rock where topographic relief and shallow depth to groundwater are anticipated to provide a short flowpath to streams (fig. 1). Two networks were established to compare the effects of centralized versus onsite wastewater treatment on stream quality. The first network, referred to as the river reach network (sites 1–3), was designed to evaluate the effects of effluent from the Town of Hillsborough municipal WWTP. Sampling locations included the WWTP outfall and sites upstream and downstream from the outfall (fig. 3; table 1). The Hillsborough WWTP has a treatment capacity of 3 million gallons per day (Mgal/d) and discharged at about 25 percent of capacity (average flow about 0.75 Mgal/d) during July 2004–June 2005

(Town of Hillsborough, North Carolina, 2005, 2008). Wastewater at the WWTP is aerobically treated, filtered, chlorinated, and dechlorinated prior to discharge into the Eno River. Effluent samples were collected at the outlet of the dechlorination chamber (site 2). The upstream river site (site 1) is about 0.25 miles (mi) upstream from the WWTP outfall. The downstream site (site 3, figs. 3, 4) is about 6 mi below the outfall and drains an area about 50 percent larger than site 1 (table 1). Land use in the drainage areas for sites 1 and 3 is similar, and both sites are primarily rural and forested (table 1). Note that for watersheds where the impervious area is relatively small despite the high percentage of development, the development contains large tracts of open space, which can consist of lawns, plantings, parks, and golf courses, for example.

A second sampling network, referred to as the small stream network (sites 4–10) was established to compare the effects of centralized and onsite wastewater treatment on stream quality in small catchments. This network includes seven sites with drainage areas ranging from 0.11 to 0.62 square miles (mi<sup>2</sup>; table 1). One of the sites is in an undeveloped, forested watershed; the remainder are in residential areas where the majority of the residences were constructed from 1960 to 1985. Sites on small streams were selected to characterize water-quality conditions representative of background or undeveloped conditions, residential with centralized wastewater treatment conditions, and residential with onsite wastewater treatment. Because of their more urban setting, the catchments of sites in areas of centralized wastewater treatment generally had higher residential densities and a higher percentage of impervious area than the sites in areas of onsite wastewater treatment (table 1). At some of the sites, additional sampling locations were established to assess sources of wastewater indicators. Soils at all of the study sites are characterized as having slow percolation (table 1; Kirby, 1976).

Site 4, on a tributary of Rhodes Creek draining a wooded area in Duke Forest, was selected to represent undeveloped stream-quality conditions (table 1, fig. 5). The area is managed for timber production and is primarily pine forest. With the exception of roadways, the catchment is undeveloped and the area adjacent to the stream channel is heavily wooded (fig. 6).

Three residential sites, sites 5–7, located in areas served by the City of Durham municipal sewer system, were selected to characterize small streams in areas of centralized wastewater treatment (City of Durham, North Carolina, 2005). Catchments for these sites are highly developed and the forested land comprises less than 10 percent of the total area. Site 5 (fig. 7) is on Nancy Rhodes Creek in the western part of Durham. Site 5 drains an area that is more than 95 percent developed (table 1). Most of the undeveloped land is along the lower reaches of Nancy Rhodes Creek, immediately upstream from site 5. Three additional sampling locations, sites 5A, 5B, and 5C, were established within the catchment of site 5 (table 2).

Site 6 is on Black Meadow Run in the northern part of Durham (fig. 8). This site has the highest residential density and has the greatest percentage of impervious surface of any of the study sites. Eight additional sampling locations were established

within the catchment of site 6 (table 3). These additional locations include points along drainage ditches and on Black Meadow Run. The stream channel has been stabilized with rock (fig. 9) along the reach between sites 6 and 6E (fig. 8; table 3) to reduce erosion associated with stormwater runoff.

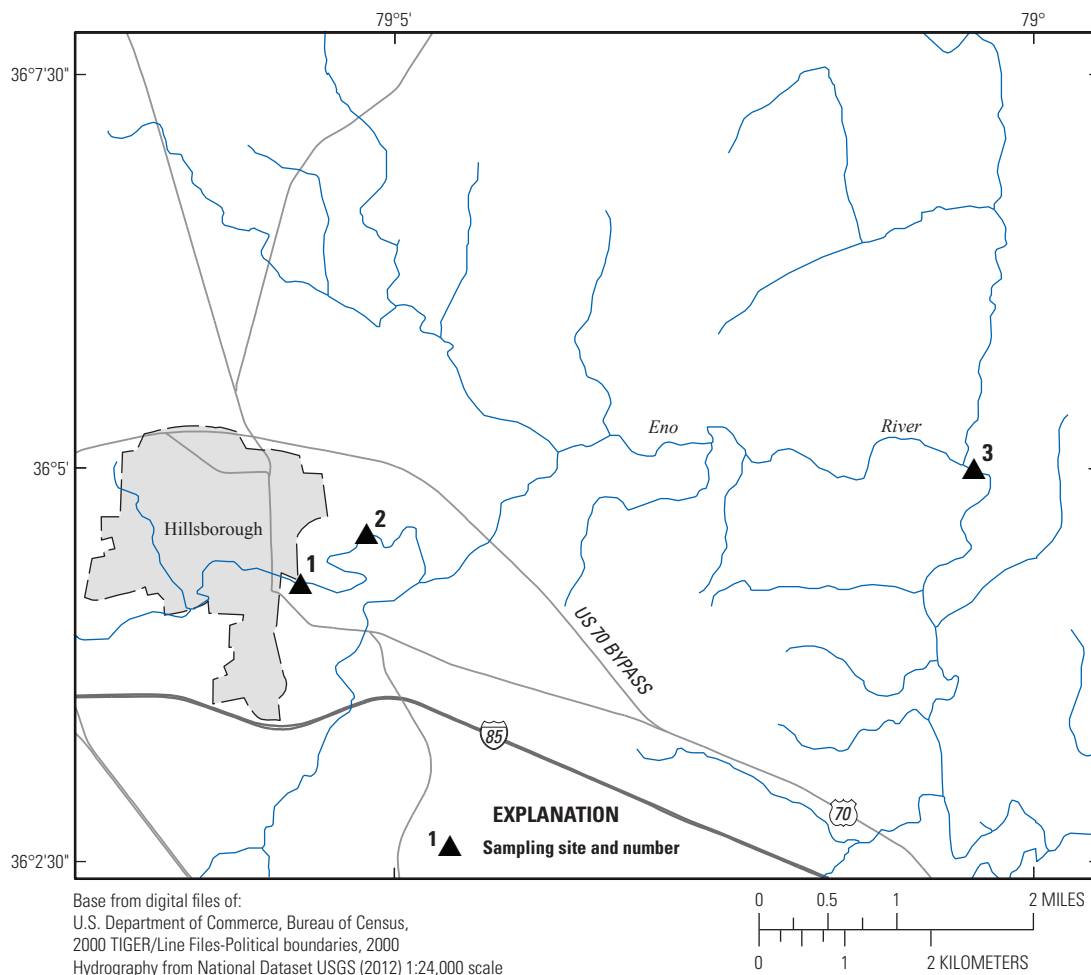
Site 7 is located on a tributary of the Eno River below Clover Hill Place in northwestern Durham (fig. 10). Streambanks in the vicinity of site 7 are eroded and undercut (fig. 11), and lawns are adjacent to the stream channel along much of its reach. Several of the residences in the northwestern part of this catchment use onsite wastewater treatment systems. One additional sampling location, site 7A, was established in this catchment (fig. 10; table 4). This site has the lowest percentage of impervious surface and the lowest residential density of the centralized wastewater treatment sites.

Three residential sites, sites 8–10, located in areas outside the limits of the municipal sewer system, were selected to characterize water-quality conditions associated with small streams in areas of onsite wastewater treatment. In comparison to the sites in areas of centralized wastewater treatment, the catchments of the sites in areas of onsite wastewater treatment are less developed, more forested, and have less impervious surface (table 1).

Site 8 is on a tributary to Sevenmile Creek (fig. 12). Forest comprises about 65 percent of the land in this catchment. Land adjacent to much of the stream channel is heavily wooded and little erosion of the streambanks and channel has occurred. The catchment for site 8 is the least developed, the most forested, and has the lowest amount of impervious surface of the residential sites.

Site 9 is on a tributary of Crooked Creek (figs. 13 and 14) north of Durham. In 2009, a new residential development was constructed in the southeastern part of the catchment. Homes in this development are served by the City of Durham municipal sewer system (City of Durham, North Carolina, 2005). Although the area is primarily residential, some cultivated land is within the catchment (table 1). Four additional sampling locations were established in the catchment of site 9 (table 5; fig. 13).

Site 10 is on a tributary to Cabin Branch (fig. 15). One additional sampling location, site 10A, was established in this catchment (table 6; fig. 15). Land in the site 10 catchment is more developed and has less forested area than the other catchments in areas of onsite wastewater treatment. The area adjacent to the stream channel is primarily forested



**Figure 3.** Locations of river reach sampling sites on the Eno River, Orange County, North Carolina.

**Table 1.** Characteristics of study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina.[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile; WWTP, wastewater-treatment plant; %, percent; --, not determined; NA, not applicable]

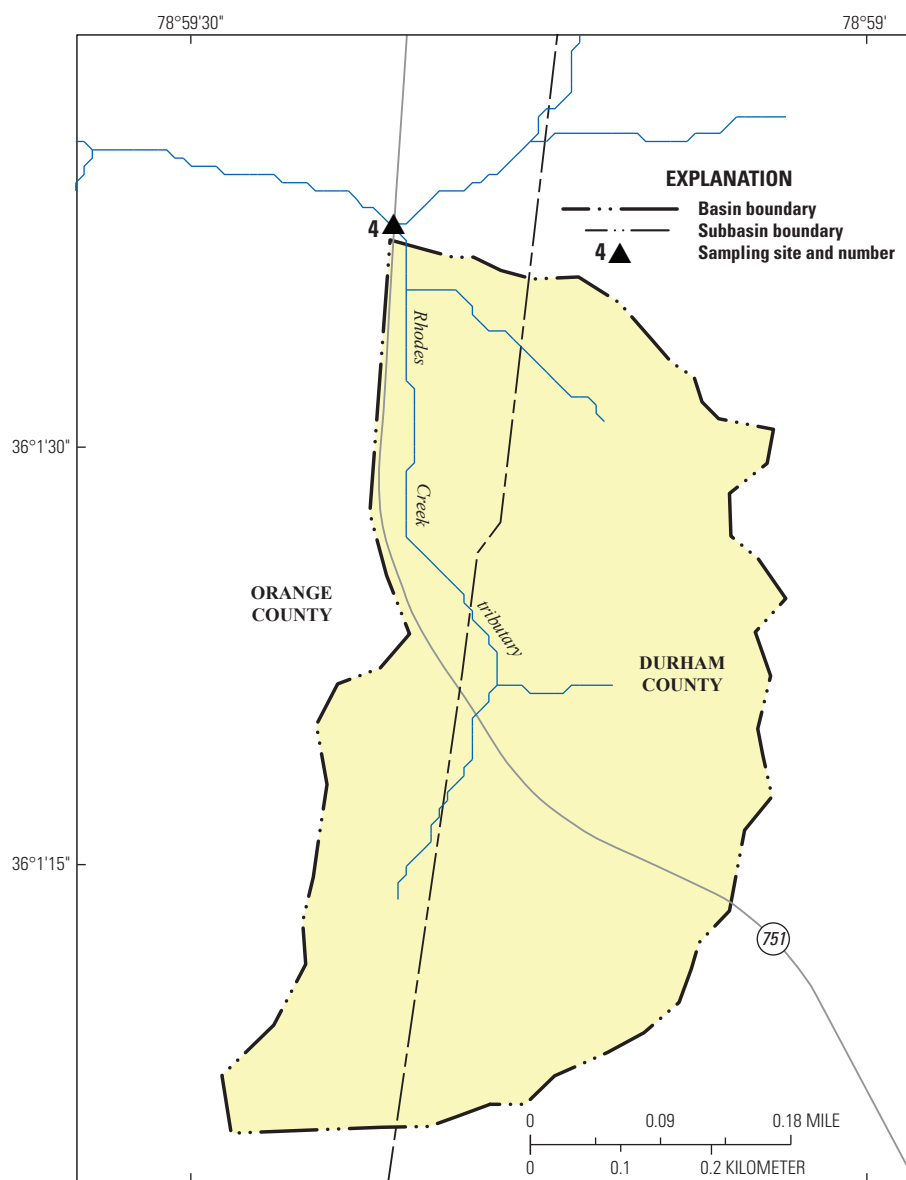
Site number (fig. 1)	USGS site identification number	Stream location	Wastewater treatment category	Major soil series <sup>a</sup>	Drain- age area (mi <sup>2</sup> )	Housing density (residential units/mi <sup>2</sup> ) <sup>b</sup>		Onsite waste- water treatment systems (% of residences)	2006 Land use characteristics (% of catchment area) <sup>d</sup>			
						2005	2012		Developed	Forested	Imper- vious area	Cultivated
River reach sites												
1	02085000	Eno River at Hillsborough, NC	upstream from WWTP	--	66.0	--	--	--	11.8	56.3	2.1	25.0
2	360428079051301	Town of Hillsborough NC Wastewater Treatment Plant	WWTP	NA	NA	NA	NA	NA	NA	NA	NA	NA
3	02085016	Eno River near Schley, NC	downstream from WWTP	--	99.4	--	--	--	12.3	58.3	2.2	22.9
Small stream sites												
4	0208503422	Rhodes Creek tributary above NC Hwy 751 near Durham, NC	undeveloped	Helena, Georgeville, Iredell	0.19	0	0	0	3.5	90.5	0.2	0.0
5	0208503920	Nancy Rhodes Creek above Cole Mill Road near Durham, NC	centralized	Appling, Georgeville, Wedowee	0.47	899	909	3	95.1	3.1	14.9	0.0
6	02085067	Black Meadow Run at Argonne Drive near Durham, NC	centralized	Iredell, White Store, Herndon	0.43	1,148	1,167	2	91.6	8.4	21.0	0.0
7	0208503990	Eno River tributary below Clover Hill Place near Durham, NC	centralized	Herndon, Georgeville	0.11	645	645	13	91.9	6.9	7.7	1.2
8	0208503945	Sevenmile Creek tributary at Inverness Drive near Durham, NC	onsite	Georgeville, Nason, Herndon	0.12	375	383	100	30.8	65.3	1.3	3.5
9	0208505880	Crooked Creek tributary at Greenbay Drive near Durham, NC	onsite	Herndon, Georgeville, Lignum	0.62	234	368	66	31.9	59.3	2.6	6.8
10	0208525095	Cabin Branch tributary at Paragon Circle near Durham, NC	onsite	Herndon, Lignum, Goldston	0.14	343	343	100	45.9	49.9	2.6	0.0

<sup>a</sup>soil series from U.S. Department of Agriculture (2006a,b).<sup>b</sup>estimated from records obtained through the Durham Spatial Data Explorer. City of Durham Geographic Information Systems Office, Durham, NC (City of Durham, North Carolina, 2005)<sup>c</sup>estimated from records of the Durham County Health Department<sup>d</sup>National Land Cover Database 2006 (Fry and others, 2011)





**Figure 4.** Stream channel at the Eno River near Schley, North Carolina.

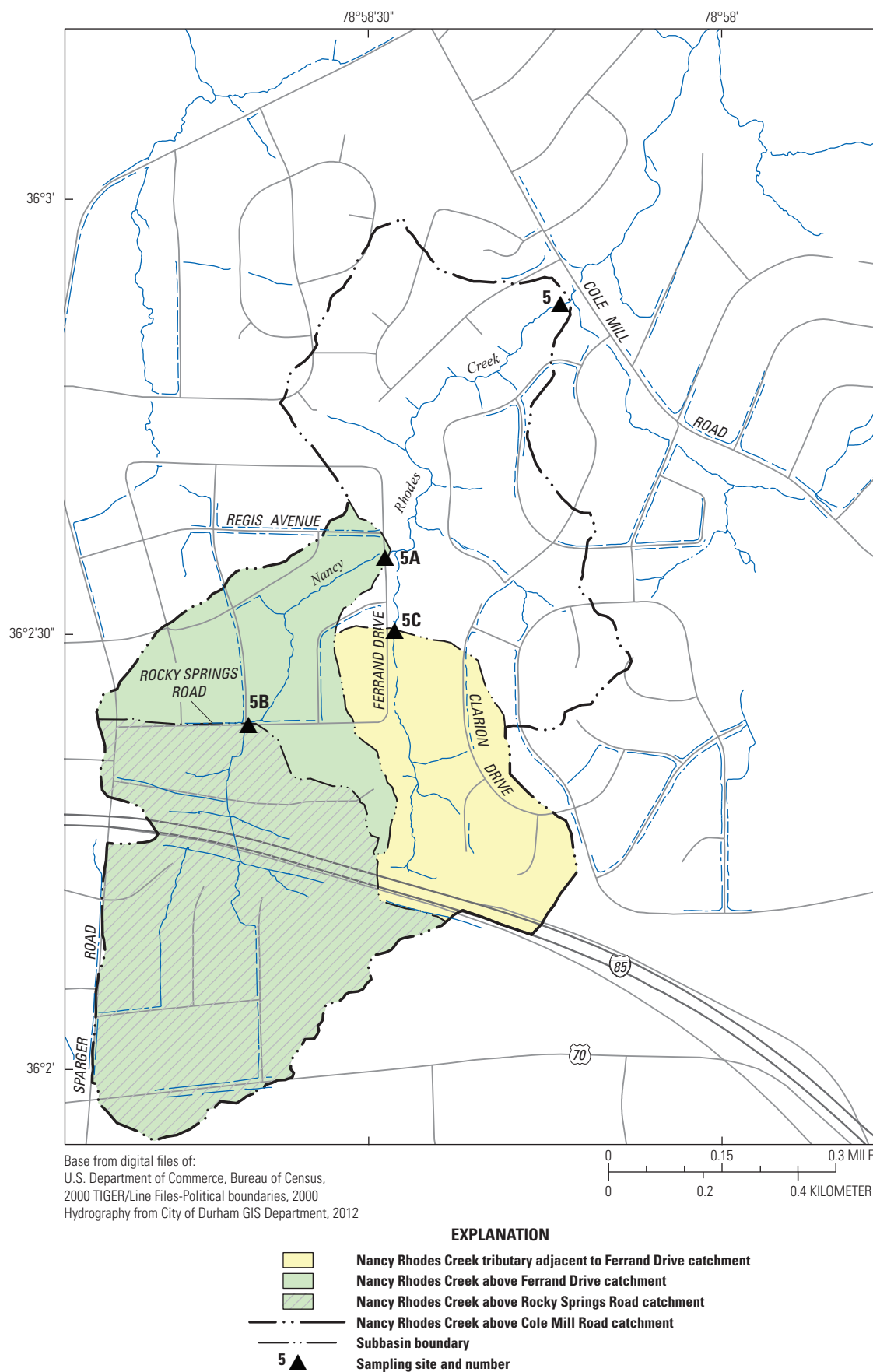


Base from digital files of:  
 U.S. Department of Commerce, Bureau of Census,  
 2000 TIGER/Line Files-Political boundaries, 2000  
 Hydrography from U.S. Geological Survey, Weaver and others, 2012

**Figure 5.** Study area for Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina.



**Figure 6.** Stream channel at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina.

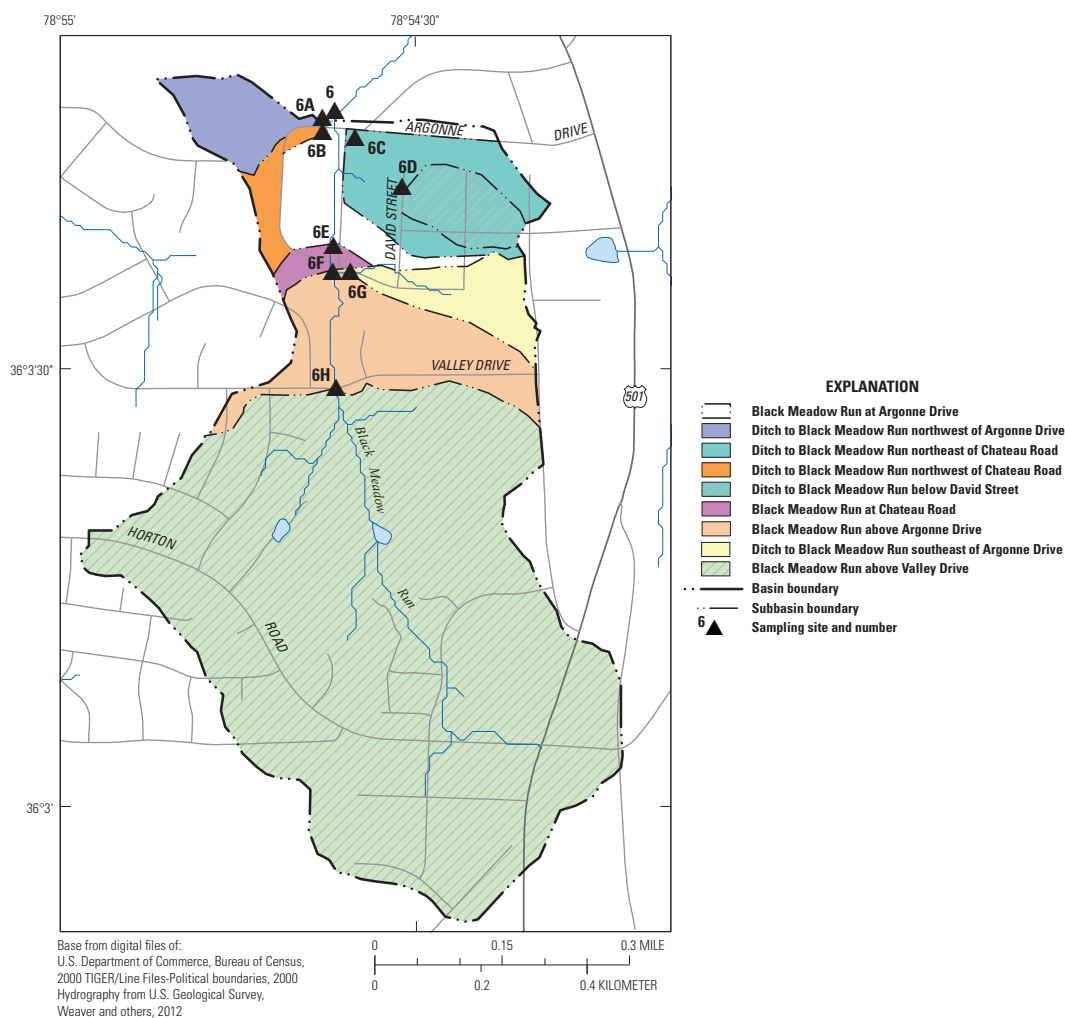


**Figure 7.** Study area for Nancy Rhodes Creek above Cole Mill Road near Durham, North Carolina.



**Table 2.** Characteristics of study sites in the Nancy Rhodes Creek above Cole Mill Road near Durham, North Carolina, catchment.[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile]

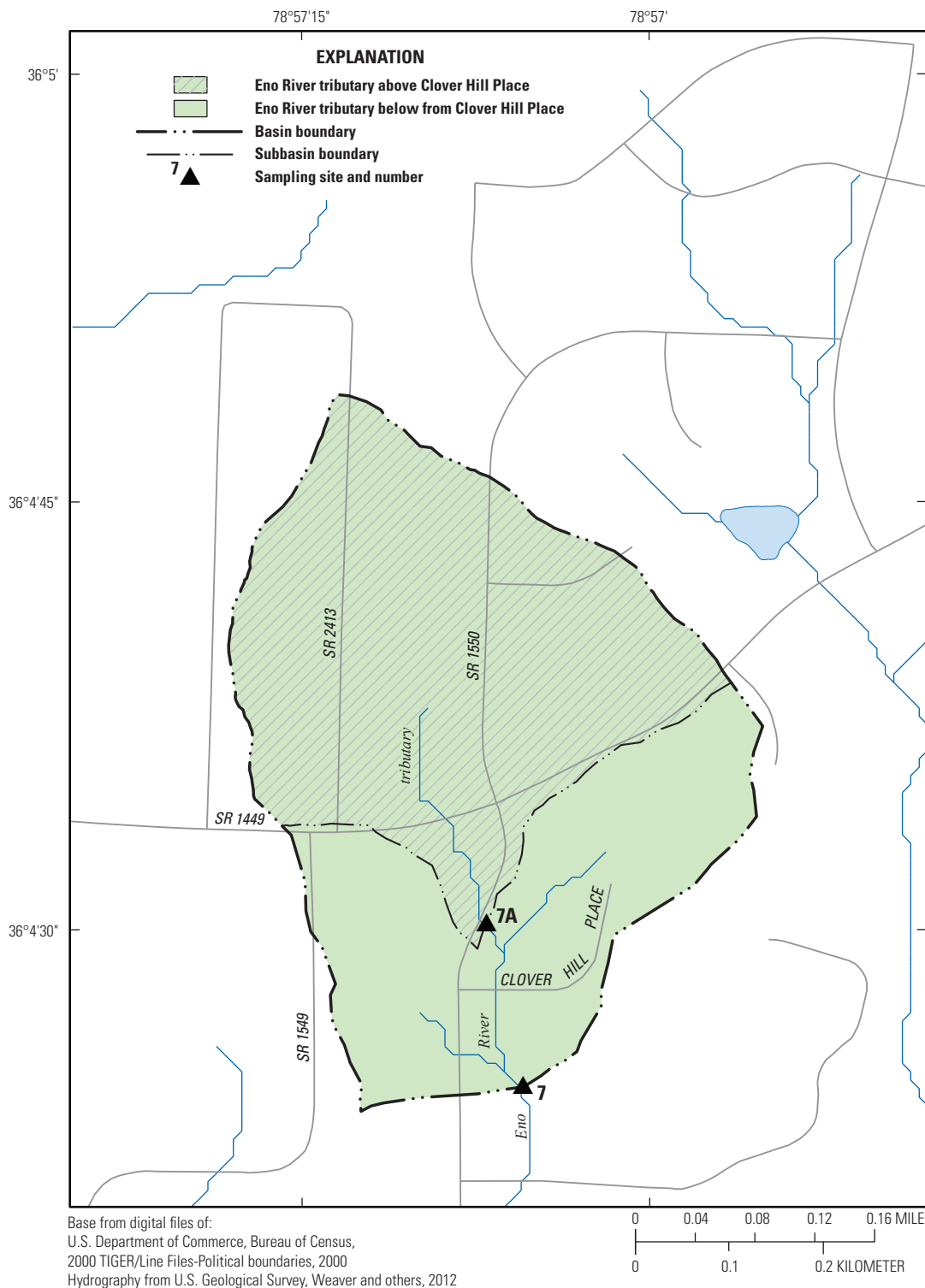
Site number (fig. 1)	USGS site identification number	Stream location	Drainage area (mi <sup>2</sup> )	Percent-age of total catchment	2006 Land Use Characteristics <sup>a</sup>			
					Developed	Forested	Impervious area	Cultivated
5A	020850391940	Nancy Rhodes Ck above Ferrand Drive at Durham, NC	0.23	49	100	0	16	0
5B	020850391920	Nancy Rhodes Ck above Rocky Springs Road at Durham, NC	0.17	36	100	0	24	0
5C	020850391960	Nancy Rhodes Ck tributary adjacent to Ferrand Drive at Durham, NC	0.07	15	100	0	14	0

<sup>a</sup>National Land Cover Database 2006 (Fry and others, 2011)**Figure 8.** Study area for Black Meadow Run at Argonne Drive near Durham, North Carolina.

**Table 3.** Characteristics of study sites in the Black Meadow Run at Argonne Drive near Durham, North Carolina, catchment.[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile; %, percent; --, not calculated]

Site number (fig. 1)	USGS site identification number	Stream location	Drainage area (mi <sup>2</sup> )	Percent-age of total catchment	2006 Land Use Characteristics <sup>a</sup>			
					Developed	Forested	Impervious area	Cultivated
6A	3603470785430801	Ditch to Black Meadow Run northwest of Argonne Drive at Durham, NC	0.01	3	--	--	--	--
6B	360347078543701	Ditch to Black Meadow Run northwest of Chateau Road at Durham, NC	0.01	1	--	--	--	--
6C	360338078543601	Ditch to Black Meadow Run northeast of Chateau Road at Durham, NC	0.03	8	--	--	--	--
6D	360343078543101	Ditch to Black Meadow Run below David Street at Durham, NC	0.01	3	--	--	--	--
6E	360338078543601	Black Meadow Run at Chateau Road at Durham, NC	0.37	86	96	4	24	0
6F	360337078543601	Ditch to Black Meadow Run southeast of Argonne Drive at Durham, NC	0.01	3	--	--	--	--
6G	02085066	Black Meadow Run above Argonne Drive at Durham, NC	0.33	77	96	4	25	0
6H	02085064	Black Meadow Run above Valley Drive at Durham, NC	0.30	70	95	5	27	0

<sup>a</sup>National Land Cover Database 2006 (Fry and others, 2011)**Figure 9.** Rock-lined stream channel upstream from the study site Black Meadow Run at Argonne Drive near Durham, North Carolina.



**Figure 10.** Study area for the Eno River tributary below Clover Hill Place near Durham, North Carolina.

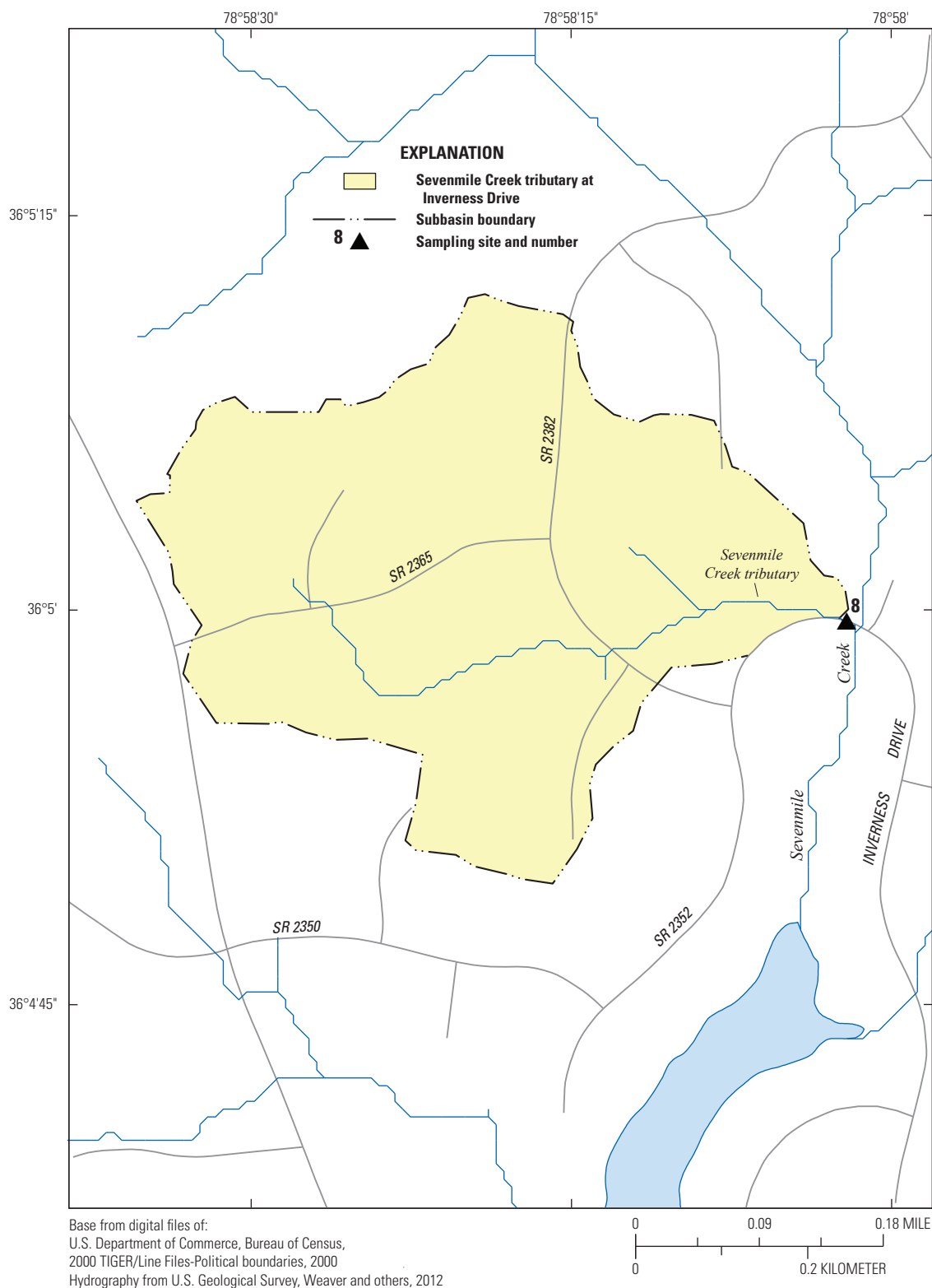


**Figure 11.** Stream channel at Eno River tributary below Clover Hill Place near Durham, North Carolina.

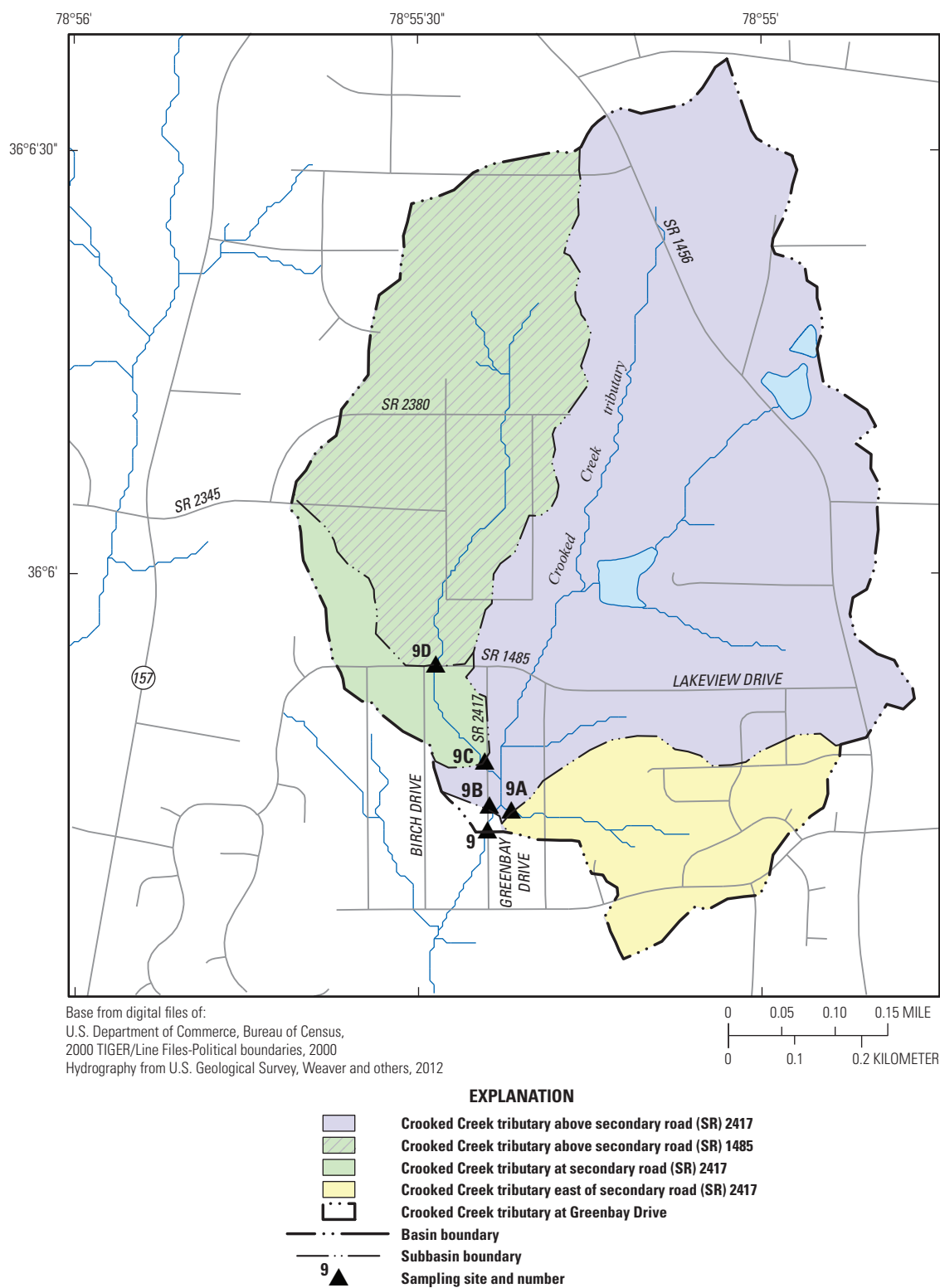
**Table 4.** Characteristics of study sites in the Eno River tributary below Clover Hill Place near Durham, North Carolina, catchment.  
[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile; %, percent]

Site number (fig. 1)	USGS site identification number	Stream location	Drainage area (mi <sup>2</sup> )	Percentage of total catchment	2006 Land Use Characteristics (% of catchment area) <sup>a</sup>			
					Developed	Forested	Impervious area	Cultivated
7A	360429078570701	Eno River tributary above Clover Hill Place at Durham, NC	0.05	43	94	6	7	0

<sup>a</sup>National Land Cover Database 2006 (Fry and others, 2011)



**Figure 12.** Study area for Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina.



**Figure 13.** Study area for Crooked Creek tributary at Greenbay Drive near Durham, North Carolina.



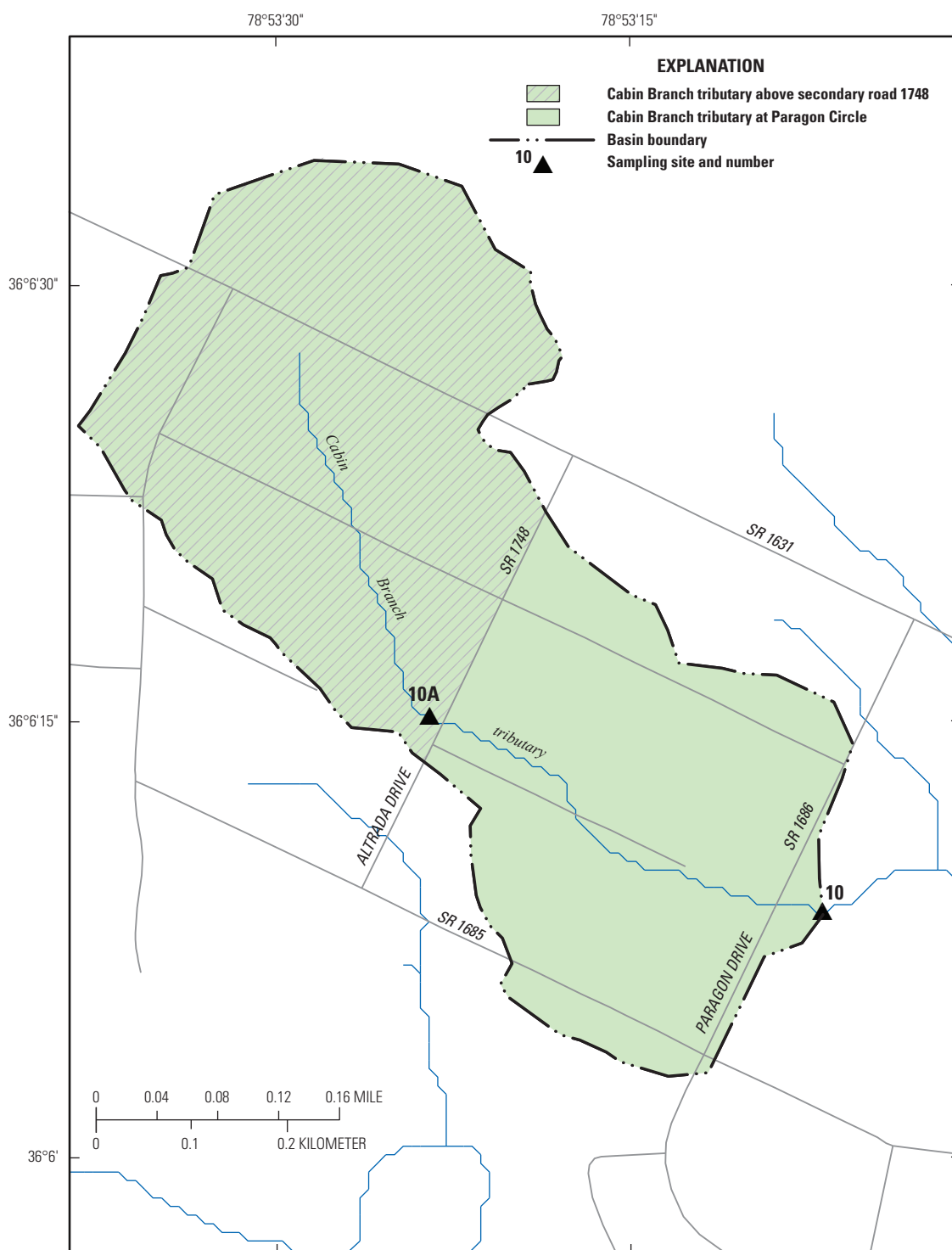


**Figure 14.** Crooked Creek tributary at Greenbay Drive near Durham, North Carolina.

**Table 5.** Characteristics of study sites in the Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, catchment.  
[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile; %, percent; SR, secondary road]

Site number (fig. 1)	USGS site identification number	Stream location	Drainage area (mi <sup>2</sup> )	Percent- age of total catchment	2006 Land use characteristics (% of catchment area) <sup>a</sup>			
					Developed	Forested	Impervious area	Cultivated
9A	360542078552201	Crooked Creek tributary east of SR 2417 near Durham, NC	0.08	12	9	78	1	5
9B	0208505878	Crooked Creek tributary above SR 2417 near Durham, NC	0.53	85	37	57	3	7
9C	360547078552401	Crooked Creek tributary west of SR 2417 near Durham, NC	0.19	31	39	61	2	0
9D	360554078552801	Crooked Creek tributary above SR 1485 near Durham, NC	0.17	27	32	68	1	0

<sup>a</sup>National Land Cover Database 2006 (Fry and others, 2011)



Base from digital files of:  
 U.S. Department of Commerce, Bureau of Census,  
 2000 TIGER/Line Files-Political boundaries, 2000  
 Hydrography from U.S. Geological Survey, Weaver and others, 2012

**Figure 15.** Study area for Cabin Branch tributary at Paragon Circle near Durham, North Carolina.



**Table 6.** Characteristics of study sites in the Cabin Branch tributary at Paragon Circle near Durham, North Carolina, catchment.[USGS, U.S. Geological Survey; mi<sup>2</sup>, square mile; SR, secondary road; %, percent]

Site number (fig. 1)	USGS site identification number	Stream location	Drainage area (mi <sup>2</sup> )	Percentage of total catchment	2006 Land Use Characteristics (% of catchment area) <sup>a</sup>			
					Developed	Forested	Impervious area	Cultivated
10A	360615078532301	Cabin Branch tributary above SR 1748 near Durham, NC	0.07	0	37	58	2	0

<sup>a</sup>National Land Cover Database 2006 (Fry and others, 2011)

## Previous Studies

Studies of the effects of onsite wastewater treatment on water quality have primarily been limited to microbes (Scandura and Sobsey, 1997; DeBorde and others, 1998) and nutrients (Aravena and others, 1993; McCray and others, 2005). Conn and others (2006), working in two counties in Colorado, found a variety of surfactant metabolites, disinfectants, caffeine, fecal sterols, and pharmaceutical compounds, including antibiotics, in residential septic tank effluent. In a subsequent study, Conn and others (2010) found that concentrations of most of these compounds decreased by more than 90 percent as effluent percolated through subsurface soils. Many pharmaceutical and hormonally active compounds escape removal by offsite conventional wastewater treatment processes (Buser and others, 1999; Glassmeyer and others, 2005). A study involving use of various wastewater indicators, including selected organic wastewater compounds, fecal bacteria, and optical brighteners to sources of wastewater in the Accotink Creek watershed in northern Virginia found highly variable concentrations of wastewater indicators during baseflow conditions (Hyer, 2007). Although identification of sources of wastewater were difficult to interpret due to low concentrations of the wastewater indicators in that study, the presence of higher concentrations facilitated identification of several sewer line leaks (Hyer, 2007). This present study in North Carolina extends results from the first study phase summarized by Ferrell and Grimes (2014).

## Methods of Data Collection and Analysis

The methods used for collection and analysis of water, wastewater effluent, and streambed sediment samples for the period 2004–2008 are described in the following section.

Because automatic samplers were used to collect samples obtained during 2012–2013, methods used during this phase of the study are described separately. Descriptions of the experimental analytical methods used in this study are provided in this section. In addition, quality-assurance and quality-control data associated with samples collected during this study are summarized.

## Sample Collection and Analysis

Water and wastewater samples were collected and processed onsite according to guidelines of the USGS national field methods manual (U.S. Geological Survey, variously dated). Samples were collected from December 2004 through October 2008 during baseflow conditions. Baseflow was considered to occur when no precipitation had fallen for the preceding 3 days. Water samples were analyzed by the USGS National Water Quality Laboratory (NWQL), Denver, Colorado, for nutrients, ions, metals, and methylene-blue active substances (table 7) using various methods. A large suite of organic wastewater compounds (OWWCs; table 8) was determined using capillary column gas chromatography/mass spectroscopy methods described by Zaugg and others (2002), including potential sources and uses as well as their reporting limits.

Antibiotics, selected antibiotic metabolites, the pharmaceutical compounds carbamazepine and ibuprofen, and selected hormones were analyzed by the USGS Organic Geochemistry Research Laboratory (OGRL) in Lawrence, Kansas. Antibiotics and pharmaceutical compounds were analyzed using online solid phase extraction and liquid chromatography/tandem mass spectrometry (LC/MS/MS) with an electrospray ionization method modified from Meyer and others (2007). Positive-ion mode was used except for identification of chloramphenicol and ibuprofen, for which negative-ion mode was used. Information regarding common uses of these compounds and their reporting limits is listed in table 9.

**Table 9.** Antibiotics, antibiotic metabolites, and pharmaceutical compounds analyzed in surface-water and wastewater-effluent samples collected in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to September 2008.

[CASRN®, Chemical Abstract Services Registry Number; -- not applicable; µg/L, micrograms per liter]

Analyte	Primary Usage	CASRN® <sup>2</sup>	Method reporting level (µg/L)
4-Epichlortetracycline hydrochloride <sup>1</sup>	--	83905-01-5	0.01
4-Epioxytetracycline <sup>1</sup>	--	298-46-4	0.01
4-Epitetracycline hydrochloride <sup>1</sup>	--	56-75-7	0.01
Anhydroerthromycin	--	57-62-5	0.005
Azithromycin	Human	85721-33-1	0.005
Carbamazepine	Human	564-25-0	0.005
Chloramphenicol	Human/Veterinary	93106-60-6	0.1
Chlortetracycline	Veterinary	514-53-4	0.01
Ciprofloxacin	Human/Veterinary	85721-33-1	0.005
Doxycycline	Human/Veterinary	35259-39-3	0.01
Enrofloxacin	Veterinary	23313-80-6	0.005
Erythromycin	Human/Veterinary	114-07-8	0.008
Ibuprofen	Human	114-07-8	0.05
Isochlortetracycline	Veterinary	15687-27-1	0.01
Isoepichlorotetracycline <sup>1</sup>	--	514-53-4	0.01
Lincomycin	Veterinary	154-21-2	0.005
Lomefloxacin	Human	98079-51-7	0.005
Norfloxacin	Human/Veterinary	70458-96-7	0.005
Ofloxacin	Human/Veterinary	82419-36-1	0.005
Ormetoprim	Aquaculture/Veterinary	6981-18-6	0.005
Oxytetracycline	Aquaculture/Beekeeping/Veterinary	79-57-2	0.01
Roxithromycin	Human	80214-83-1	0.005
Sarafloxacin	Aquaculture/Veterinary	98105-99-8	0.005
Sulfachlorpyridazine	Veterinary	80-32-0	0.005
Sulfadiazine	Human/Veterinary	68-35-9	0.005
Sulfadimethoxine	Aquaculture/Veterinary	122-11-2	0.005
Sulfamethazine	Veterinary	57-68-1	0.005
Sulfamethoxazole	Human	723-46-6	0.005
Sulfathiazole	Veterinary	72-14-0	0.005
Tetracycline	Human/Veterinary	738-70-50	0.01
Trimethoprim	Human/Veterinary	64-75-5	0.005
Tylosin	Veterinary	1401-69-0	0.01
Virginiamycin	Veterinary	8065-94-9	0.005

<sup>1</sup>degradation product

<sup>2</sup>This report contains Chemical Abstracts Services Registry Numbers (CASRN)®, which is a Registered Trademark of the American Chemical Society.

Hormone samples were analyzed using either a magnetic particle immunoassay procedure (Abraxis, 2010) or by an experimental direct injection LC/MS/MS research method (liquid chromatography hormone method, LCHM) developed by the OGRL (table 7). A detailed description of the method is provided in appendix 1. Hormone samples were passed through a 0.2-micron ( $\mu\text{m}$ ) syringe filter into glass containers and frozen until they were shipped on ice by overnight carrier to the OGRL. Methodology for samples analyzed by magnetic particle immunoassay corresponds to that described in Abraxis (2010) and Buehler and others (2009).

Streambed-sediment samples were collected on June 1, 2005, from the upper 10 centimeters (cm) at 5 to 10 points within the stream channel. The samples were collected with a polyethylene scoop, and only saturated sediments were collected. Streambed-sediment samples were dried, sieved to less than 150  $\mu\text{m}$  (100 mesh), and analyzed for selected elements by inductively coupled plasma–mass spectroscopy at the USGS Geochemistry Laboratory in Denver, Colorado, according to methods described by Arbogast (1996) and Briggs and Meier (2002).

Water samples collected for analysis of fecal bacteria in 2005 were placed on ice upon collection and transported overnight for analysis at the Duke University Marine Laboratory, Beaufort, N.C. Samples were analyzed for *Escherichia coli* (*E. coli*) and fecal coliform bacteria using the five-tube dilution method with A-1 media to which 4-methylumbelliferyl- $\beta$ -D-glucuronide (MUG) was added for identification of *E. coli* according to standard methods of the American Public Health Association and others (1995). Water samples collected for analysis of fecal bacteria in 2008 were placed on ice upon collection and transported to the Soil Laboratory at North Carolina State University, Raleigh, N.C., for analysis using Colisure® and Enterolert® test kits (IDEXX Laboratories, Inc., 2008a and 2008b).

Samples collected for analysis of optical brighteners were stored in darkness until the time of analysis. Samples collected during phase 1 (2004–2005) were analyzed by personnel of the Virginia Polytechnic Institute and State University in Christiansburg, Virginia, following the method described by Dickerson and others (2007). Samples collected during phase 2 (2008) were analyzed by personnel of the Soil Science Laboratory at North Carolina State University, Raleigh, N.C., following the analytical methods described by Hartel and others (2007). Samples collected during phase 3 (2012–2013) were analyzed by an experimental method developed by personnel from the USGS North Carolina Water Science Center (NCWSC) in Raleigh, N.C.. The method is described in appendix 2. Samples for phase 3 were collected during streamflow recession periods, from May 2012 through January 2013, using automated samplers. Ice was maintained in the sample carousel to minimize degradation of samples. Samples were retrieved on a daily basis and transported to the NCWSC for processing and analysis. Samples for nutrients were filtered using a 0.45- $\mu\text{m}$  filter and placed on ice for overnight shipment to the USGS NWQL for analysis.

Samples for optical brighteners were stored in darkness prior to analysis. Samples for 17- $\beta$ -estradiol were filtered with a 0.2- $\mu\text{m}$  filter and frozen until shipment on ice by overnight carrier to the OGRL.

Records of stream stage, referenced to an arbitrary datum, were obtained using submersible transducers. Transducers were suspended in perforated pvc pipe secured to a metal T-post that had been driven into the streambed. The top of the T-post was used as the reference datum. A secondary reference was obtained by measuring from the top of a piece of metal reinforcing bar that was also driven into the streambed to water surface. Data recorded by the transducers were downloaded and adjusted for atmospheric pressure.

## **Quality Assurance and Quality Control of Laboratory Data**

Overall quality assurance was assessed by various field- and laboratory-based quality control samples. These included blanks (field, source solution, and analytical/laboratory), surrogates to assess bias, and replicates (field, split, and analytical/laboratory) to assess variability. For analyses conducted by the NWQL, performance data generated by both the laboratory and the independent Branch of Quality Systems (BQS) within the U.S. Geological Survey were reviewed. These BQS programs included: Organic Blind Sample Project, Inorganic Blind Sample Project, and Blind Blank Project (<http://bqs.usgs.gov/> accessed 31 July, 2013). For bacteria counts, quality control procedures corresponded to those for the positive controls described in IDEXX (2008a,b). All quality control results for water and wastewater effluent except that for optical brighteners for the 2012–2013 period are shown in appendix 3.

For analytes determined at the NWQL, 653 results were obtained on several field and source solution blanks (table 10). There were two detections, one for zinc and one for hexahydrohexamethylcyclopentabenzopyran, and both were very near reporting levels. There were 23 qualitative (present but not quantifiable) detections among some of the OWWCs, and for ammonia nitrogen (hereafter ammonia). Several of the OWWCs are widely distributed throughout the environment and thus are commonly detected in blanks. Some analytes were common laboratory contaminants; concentrations had to exceed a threshold of 10 times the blank concentration to be reported as a positive detection not from laboratory contamination. Common laboratory contaminants include octylphenol- and nonylphenol-ethoxylates and polyethoxylates because of their tendency to adsorb to surfaces, resulting in carryover between samples. Likewise, benzophenone was considered to be a common laboratory contaminant because of its volatility. For analytes not considered to be common laboratory contaminants, a threshold of five times the concentration in associated blanks was used to report concentrations in environmental samples as positive detections.

Surrogate compounds are similar but not identical to the analytes being determined. They are added to every sample and used as a general measure of method performance. Recovery percentages (mass recovered divided by mass added times 100) of these compounds can indicate relatively large analytical anomalies such as spilled samples or inadvertent omission of an analytical step. Surrogate recoveries for the OWWCs were determined in three matrices: blank water, wastewater effluent, and surface water (table 11). Median recoveries were generally similar for all three compounds in all three matrices. These recoveries rule out significant sources of bias in the analysis of the wastewater compounds.

Variability in the final reported concentration has several sources, including sampling, handling, and analysis. Field replicates capture the overall variability from all three sources, whereas split replicates reflect only the handling and analysis. Analytical replicates capture just the variability in the laboratory analysis. All three replicates were used at times during the three study phases. For analytes in water determined at the NWQL, the relative percent difference (RPD, absolute difference divided by the average times 100) was calculated for each replicate set (table 12). Among the listed analytes, the median RPD ranged from 0 to 15 percent with relatively small absolute differences in concentration within the field replicate pairs. The split replicates had somewhat higher RPDs for most nutrients than did the field replicates, which was not expected. A possible explanation is a filter failure, but it is not possible to determine the actual reason.

Finally, one split replicate was analyzed among the bed sediment samples (appendix 4). The median RPD was 1 percent and the range of RPDs was 0 to 67 percent. The high RPD was for ytterbium and resulted from concentrations of 1 and 2 milligrams per kilogram (mg/kg) with a reporting level of 1 mg/kg. The median absolute difference in concentrations was 0.004 mg/kg and ranged from 0 to 30 mg/kg. The high value was for strontium, and the associated concentrations were 140 and 110 mg/kg.

Quality-control data for the antibiotic and pharmaceutical compounds measured in samples during this study are summarized in table 13 for spiked laboratory water (blanks), spiked environmental samples, and duplicate samples. Samples consisting of laboratory water that were spiked with antibiotic and pharmaceutical compounds show average apparent recoveries of 75 to 120 percent with relative standard deviations (RSD) of less than 25 percent. These data indicate that most of the compounds performed well with the method and in the laboratory water and environmental matrices. The two compounds analyzed in negative-ion mode, chloramphenicol and ibuprofen, were the poorest performing compounds, which may indicate some metals interference.

With a few exceptions, extracted laboratory water blanks showed 0 to 15 percent detections with mean concentrations of 0.001 micrograms per liter ( $\mu\text{g/L}$ ) or less (table 13). These trace-level detections were all 5 to 10 times below the compound reporting levels and were associated primarily with the more hydrophobic (macrolide) compounds and the compounds

that can have either multiple valence states or that contain complex metals (fluoroquinolones and tetracyclines). Thus, compound carryover is dependent on the sorption potential of the materials in the sample flow path. The nature of the blank data shows no chronic blank problems with the exception of erythromycin, which is well understood, and occasional trace-level carryover of certain analytes with the exception of tylosin.

Ten duplicate samples were analyzed during this study, and compounds were detected 13 times. The detections included azithromycin, carbamazepine, erythromycin, erythromycin- $\text{H}_2\text{O}$ , lincomycin, ciprofloxacin, ofloxacin, sulfamethoxazole, trimethoprim, and tylosin. All the compounds were detected once except for carbamazepine (three times), and erythromycin- $\text{H}_2\text{O}$  (two times). Nine of the detections were associated with wastewater effluent samples. The difference between the detections in the duplicate samples was less than 25 percent except for azithromycin (38 percent), lincomycin (50 percent), erythromycin (93 percent), and erythromycin- $\text{H}_2\text{O}$  (140 percent). The large differences were associated with the wastewater effluent sample. The reason for the larger difference for erythromycin is not known.

Quality-assurance and quality-control practices for the 17- $\beta$ -estradiol magnetic particle immunoassay include analysis of duplicate samples, blanks, matrix spikes, and the kit control samples, which are part of each enzyme linked-immunoassay set. Quality-control criteria require that duplicate samples must agree within 25 percent, matrix spikes and kit controls must be within 25 percent of the spiked or manufacturer value, blanks must come back as nondetects (less than the laboratory set detection limit), and the coefficient of determination ( $r^2$ ) of the standard curve equals or exceeds 0.99. On the basis of these criteria, a whole or partial set of an analytical run could be rejected, depending on the pattern of the criteria failure.

Quality-assurance and quality-control practices implemented for the experimental liquid chromatography hormone method (see appendix 1) are based on the  $r^2$  of the standard curve and the results of matrix spikes. Standard curves with correlation coefficients of 0.999 were obtained for all five estrogenic hormones. None of the five compounds

**Table 10.** Frequency and concentrations of analytes detected in field and source solution blanks.

[R.L., reporting level; µg/L, micrograms per liter]

Blank type	Analyte	Detections	Qualitative Detections
		(Result; R.L.)	(number)
Field	Ammonia nitrogen		2
Field	Zinc	0.7; 0.6 µg/L	
Field	2,6-Dimethylnaphthalene		1
Field	2-Methylnaphthalene		1
Field	4-tert-Octylphenol diethoxylate		2
Field	Benzophenone		2
Field	Caffeine		1
Field	Hexahydrohexamethylcyclopentabenzopyran	1; 0.5 µg/L	1
Field	Isophorone		1
Field	Methyl salicylate		1
Field	Naphthalene		2
Field	Phenanthrene		1
Field	Prometon		1
Field	Tributyl phosphate		4
Field	Triethyl citrate		1
Field	Tris(2-chloroethyl) phosphate		1
Field	Tris(dichloroisopropyl) phosphate		1
Source solution	4-tert-Octylphenol diethoxylate		1
Source solution	Benzophenone		1
Source solution	Naphthalene		1
Source solution	Tributyl phosphate		1

**Table 11.** Surrogate recoveries for organic wastewater compounds in blank water, surface-water, and wastewater-effluent samples, 2004–2008.

[C, carbon; d, deuterium]

Medium		Recovery (percent)		
		Caffeine- <sup>13</sup> C	Decafluorobiphenyl	Fluoranthene-d <sup>10</sup>
Blank (field and source solution)	Number	10	10	10
	Median	83	72	80
	Minimum	66	27	73
	Maximum	94	111	92
Wastewater effluent	Number	12	12	12
	Median	86	81	79
	Minimum	81	35	68
	Maximum	102	106	94
Surface water	Number	92	92	92
	Median	85	67	84
	Minimum	25	20	24
	Maximum	140	123	113



**Table 12.** Variability of replicate analyte determinations.

[RPD, relative percent difference; mg/L, milligrams per liter; µg/L, micrograms per liter; MPN/100 mL, most probable number of the colonies in 100 milliliters; <, both replicates were below the reporting level]

Analyte	Replicate type	Units	Repl- cate sets (number)	Median RPD (percent)	Median absolute difference (units)
Calcium	field	mg/L	1	1	0.1
Magnesium	field	mg/L	1	0	0.01
Potassium	field	mg/L	1	7	0.08
Sodium	field	mg/L	1	1	0.05
Chloride	field	mg/L	1	0	0.02
Fluoride	field	mg/L	1	0	0
Sulfate	field	mg/L	1	2	0.04
Ammonia plus organic nitrogen as nitrogen	field	mg/L	3	10	0.04
Ammonia as nitrogen	field (6), split (1)	mg/L	7	1 (field), 31 (split)	0.0005 (field), 0.083 (split)
Nitrate plus nitrite as nitrogen	field (9), split (1)	mg/L	10	1 (field), 3 (split)	0.005 (field), 0.012 (split)
Nitrite as nitrogen	field	mg/L	6	0	0
Orthophosphate as phosphorus	field (6), split (1)	mg/L	7	0 (field), 35 (split)	0 (field), 0.014 (split)
Copper	field	µg/L	1	<	<
Zinc	field	µg/L	1	1	0.4
Organic carbon	field (2), split (1)	mg/L	3	15 (field), 0.2 (split)	0.3 (field), 0.2 (split)
Wastewater contaminants (all)	field	µg/L	3	<	<
Fecal coliform	split	MPN/100 mL	1	57	40
<i>E. coli</i>	split (2)	MPN/100 mL	2	46, 18	12, 219

**Table 13.** Quality-control data for analyses of antibiotics, antibiotic metabolites, and pharmaceutical compounds.

[µg/L, micrograms per liter; RSD, relative standard deviation; n, number; %, percent]

Analyte	Average apparent percent recoveries and relative standard deviation for antibiotic compounds in 0.2 µg/L spiked laboratory water samples.			Average apparent percent recoveries and relative standard deviation for antibiotic compounds in 0.2 µg/L matrix spiked samples.			Average analyte concentration, relative standard deviation and percent detection in laboratory blank samples in micrograms/liter			
	Average	RSD	n	Average	RSD	n	Average	RSD	n	% Detections
Azithromycin	94	42.0	15	73	32.0	6	0.000	0.001	20	15
Tylosin	130	45.2	16	200	82.9	11	0.004	0.009	20	40
Total Erythromycin <sup>a</sup>	99	11.6	16	96	38.6	10	0.001	0.002	20	100
Roxithromycin	100	25.4	16	140	47.5	11	0.000	0.001	20	25
Virginiamycin	110	21.0	16	110	19.5	9	0.000	0.000	20	0
Carbamazepine	96	20.6	16	97	21.8	11	0.000	0.000	20	5
Chloramphenicol	77	122	11	14	3.37	6	0.000	0.000	20	0
Ibuprofen	61	49.2	16	39	15.3	10	0.000	0.000	20	0
Norfloxacin	96	18.2	16	77	28.8	9	0.000	0.000	20	0
Ciprofloxacin	91	14.8	16	67	21.4	9	0.000	0.000	20	5
Ofloxacin	100	17.5	16	82	18.2	9	0.000	0.001	20	15
Lomefloxacin	100	15.1	16	85	15.5	10	0.000	0.001	20	10
Enrofloxacin	93	25.5	16	76	40.2	9	0.000	0.001	20	5
Sarafloxacin	95	13.6	16	69	21.8	7	0.000	0.000	20	5
Lincomycin	91	24.1	16	85	15.5	10	0.000	0.000	20	15
Ormetoprim	78	27.8	16	71	24.9	10	0.000	0.000	20	10
Trimethoprim	85	19.3	16	93	29.1	10	0.000	0.000	20	0
Sulfadiazine	87	30	14	99	51.4	10	0.000	0.000	20	0
Sultathiazole	96	20.4	16	100	23.4	11	0.000	0.000	20	0
Sulfamethazine	105	19.4	16	130	33.6	11	0.000	0.000	20	5
Sulfachloropyridazine	110	13.3	16	110	25.7	11	0.000	0.001	20	10
Sulfamethoxazole	120	24.8	16	115	38.5	11	0.000	0.001	20	5
Sulfadimethoxine	120	20.9	16	130	24.4	11	0.000	0.000	20	0
Tetracycline	89	19.7	16	90.1	21.4	9	0.000	0.000	20	30
Oxytetracycline	95	21.5	16	97	8.02	8	0.000	0.000	20	5
Doxycycline	99	16.3	16	79	34.3	9	0.000	0.000	20	5
Total Chlortetracycline <sup>b</sup>	160	52.6	16	120	30.4	9	0.000	0.001	20	10

<sup>a</sup>Total Erythromycin = erythromycin + erythromycin-H<sub>2</sub>O<sup>b</sup>Total Chlorotetracycline = epi + epi-iso + isochlorotetracycline + chlorotetracycline

were detected in the 127 blank samples. The apparent percent recovery for the hormones for all 62 samples analyzed ranged from 85 to 93 percent and the RSDs ranged from 2 to 5 percent (table 14). The data indicate a slight low bias in the spiked samples relative to the standard curve, which may be due to matrix effects or to slight differences between the sample addition spike mix relative to the standard curve solutions. The low variation in the RSD indicates low variability among the samples with respect to analyte matrix effects. Finally, all the analytes in three sets of analytical replicates had concentrations below the method reporting level, so no precision could be calculated.

**Table 14.** Recovery of hormones in samples, May 2012 to January 2013.

[2.0 micrograms/liter in matrix spiked environmental RSD, relative standard deviation; n, number of samples]

Analyte	Average percent recovery <sup>a</sup>	RSD	n
estrone	87	2.12	62
estriol	85	2.52	62
17 beta-estradiol	91	4.78	62
17 alpha-estradiol	93	3.18	62
ethynylestradiol	92	3.18	62

<sup>a</sup>recoveries are for environmental samples spiked at a concentration of 2.0 micrograms/liter.

## Effects of Centralized and Onsite Wastewater Treatment on Stream Quality

Study results for the data collected during 2004–2008 and the 2012–2013 stormflow recession data are presented separately. All water and effluent results for the 2004–2008 period are shown in appendix 5. Streambed sediment results for 2005 are shown in appendix 6. All water-quality results except those for optical brighteners for the 2012–2013 period are shown in appendix 7. Fluorescence results for 2012–2013 are shown in appendix 8. Optical brightener results for 2012–2013 are shown in appendix 9. Finally, all stage data for the 2012–2013 period are shown in appendix 10. A third section, in which within catchment variation in wastewater indicator compounds are described, includes data from the entire 2004–2013 period.

### Quality of Streams and Wastewater Effluent, 2004–2008

Concentrations of dissolved sodium, dissolved sulfate, and dissolved nitrate plus nitrite, as nitrogen (hereafter nitrate plus nitrite), were higher in water samples collected downstream from the WWTP outfall (site 3) than upstream from the WWTP (site 1; figs. 16, 17). Concentrations of

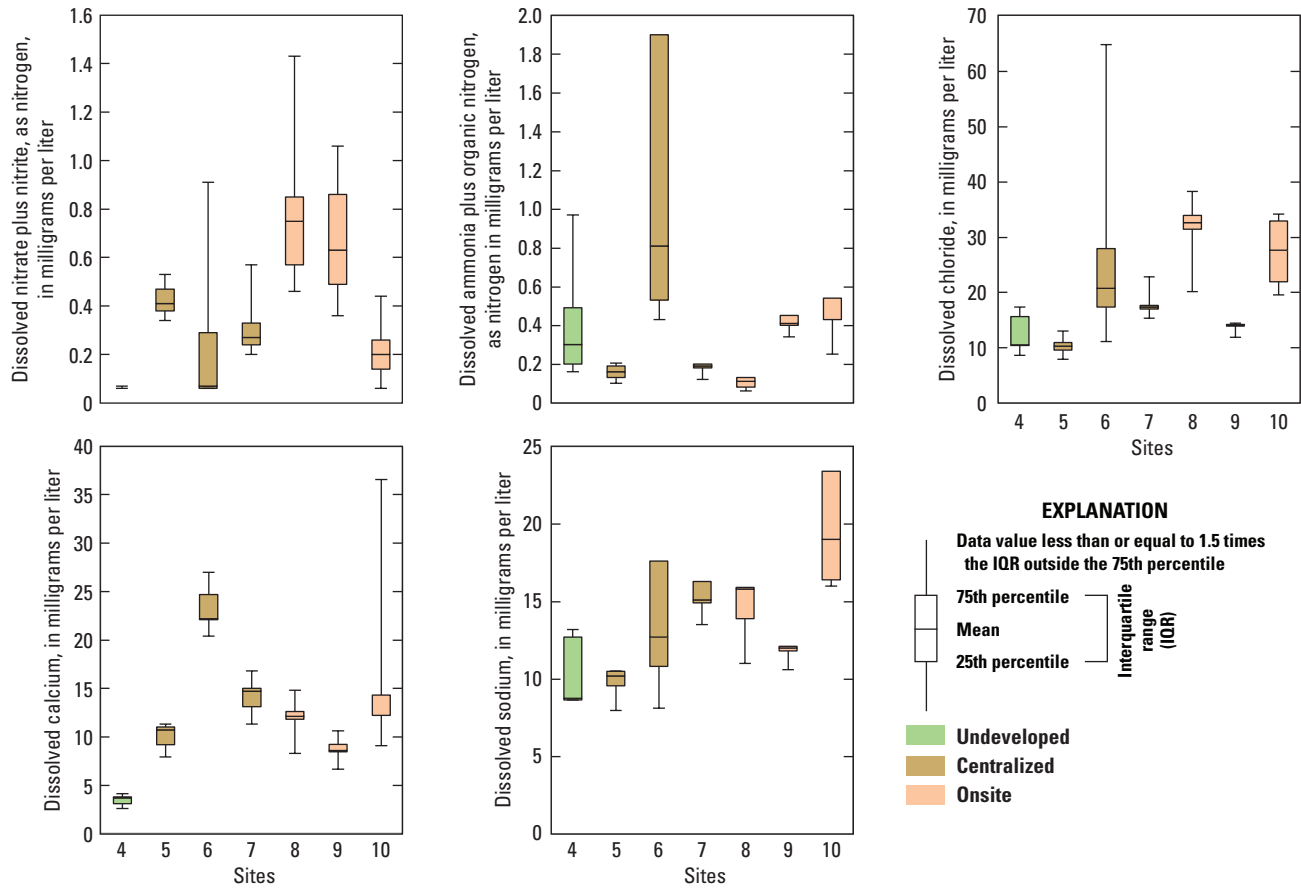
dissolved sodium, dissolved sulfate, and dissolved chloride in wastewater effluent samples were about ten times greater than those in the upstream site (table 15).

The total nitrogen concentration at the upstream site showed little variation during the study (fig. 17). Dissolved nitrate plus nitrite as the dominant form of nitrogen in wastewater effluent, whereas organic nitrogen comprised from 30 to 54 percent of the total nitrogen at the upstream site (site 1) and 17 to 45 percent of the total nitrogen at the downstream site (site 2; fig. 17). Dissolved orthophosphate concentrations in effluent samples ranged from 0.34 to 2.21 milligrams per liter (mg/L) (table 15) and were about 100 times larger than those at the sites upstream and downstream from the wastewater treatment plant, which were generally near or less than reporting levels.

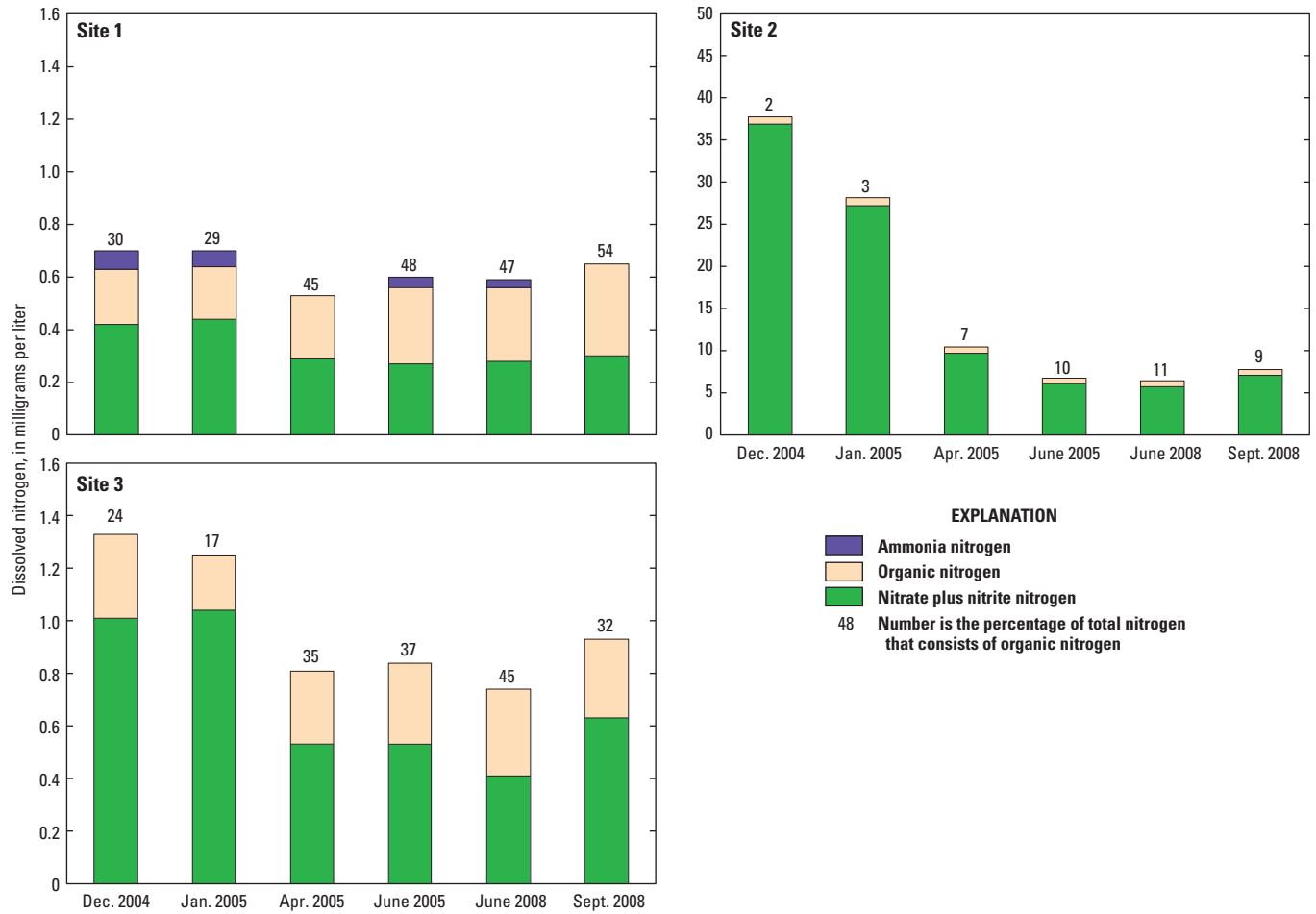
Nutrient concentrations generally were similar among samples from the residential small stream sites (sites 5–10) with the exception of site 6, which had higher concentrations of total nitrogen and ammonia nitrogen, a smaller proportion of nitrogen in the form of dissolved nitrate plus nitrite, and greater overall variability (figs. 18 and 19). Nitrogen concentrations in samples from the undeveloped catchment (site 4) were lower than those in samples from the residential catchments. The dissolved ammonia nitrogen concentrations in some samples from site 6 exceeded reporting limits, which suggests a sewer line leak as was also seen in this catchment in the previous study (Ferrell and Grimes, 2014). Dissolved nitrate plus nitrite concentrations in samples from the small stream sites generally were less than 1 mg/L and did not differ greatly between centralized and onsite wastewater treatment. Orthophosphate phosphorus concentrations were near or less than the reporting limit of 0.02 mg/L at all sites. Onsite wastewater treatment processes typically remove 85 to 95 percent of the influent phosphorus, whereas removal rates for nitrogen range from 10 to 40 percent (Sikora and Corey, 1976).

With the exception of site 6, variations in dissolved nitrogen concentrations in samples from the small stream sites were small and likely occurred in response to seasonal and hydrologic conditions (fig. 19). The variation in nitrogen concentrations observed in samples from site 6, if due to sewer line leakage, could reflect leakage changes and (or) decreased microbial transformation rates and biological uptake associated with colder temperatures during winter months. Additionally, dry conditions during May and June could have slowed subsurface flow and enabled increased nutrient assimilation. Deposition of particles within the sewer line also could have decreased rates of leakage (Ellis and others, 2003). Fertilizer applications to lawns are an additional source of nitrogen, especially in the catchments with centralized wastewater treatment (sites 5, 6, and 7) because of high household density (table 1) and the low amount of forested area in these locations. Finally, household densities in the basins with centralized wastewater treatment are about 2 to 5 times greater than in the basins with onsite wastewater treatment (table 1).





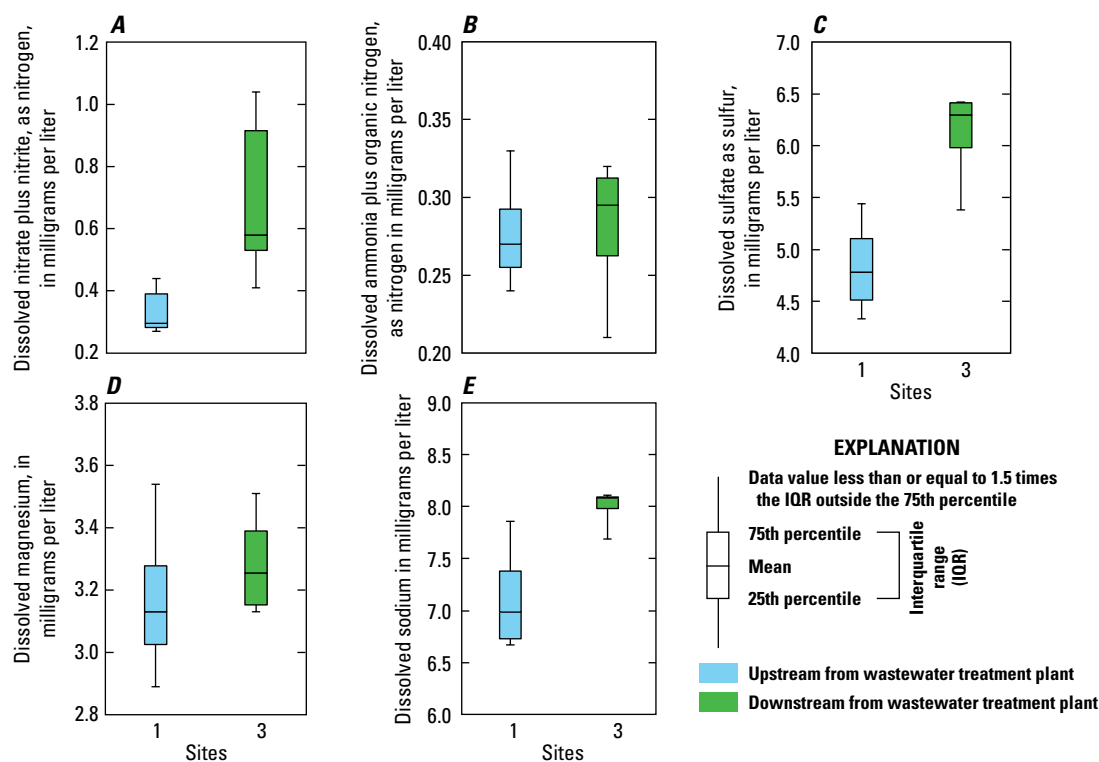
**Figure 16.** Distributions of concentrations of selected dissolved ions and nutrients in surface-water samples from river reach sites in Orange County, North Carolina, December 2004 to September 2008.



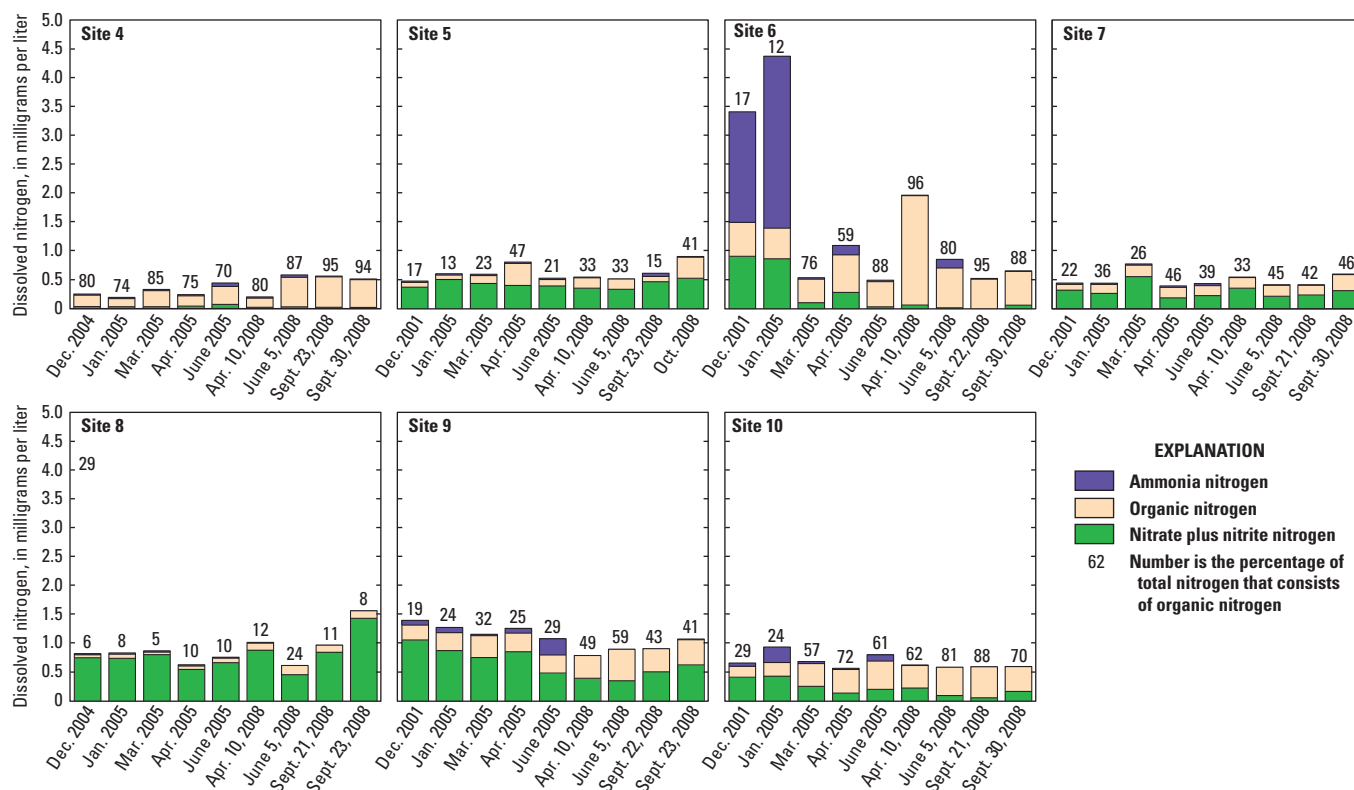
**Figure 17.** Temporal variation in concentrations of dissolved ammonia nitrogen, organic nitrogen, and nitrate plus nitrite nitrogen in water samples from the Eno River upstream and downstream from the Town of Hillsborough Wastewater Treatment Facility outfall, and effluent samples from the town of Hillsborough Water Treatment Plant, Hillsborough, North Carolina, December 2004 to September 2008.

[E, concentration is less than the method reporting limit; <, less than; --, not analyzed;  $\mu\text{g/L}$ , micrograms per liter;  $\text{mg/L}$ , milligrams per liter]

Site Number (fig. 1)	Date	TIMES Sample start time	Calcium	Magnesium	Potassium	Sodium	Chloride	Fluoride	Sulfate	Ammonia plus organic nitrogen	Ammonia	Nitrate plus nitrite, as N	Nitrite	Ortho- phosphate	Copper	Zinc	Arsenic
			(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(µg/L)
1	12/20/04	1100	6.67	3.07	1.71	5.51	5.87	E.05	4.57	0.28	0.07	0.42	E.004	<02	<2.0	19.2	<2
	01/26/05	1400	6.75	2.89	1.62	6.76	7.84	<10	4.99	0.26	0.06	0.44	E.005	<02	<2.0	16.6	<2
	04/29/05	830	7.22	3.19	1.19	5.65	6.51	E.08	4.33	0.24	<04	0.29	<008	<02	E1.0	15.9	<2
	06/17/05	1630	7.86	3.54	1.67	7.02	6.82	0.12	5.44	0.33	E.04	0.27	E.005	<02	E1.4	134	<2
	06/05/08	1145	--	--	--	--	--	--	--	0.31	0.034	0.28	0.005	0.007	--	--	--
	09/23/08	1115	--	--	--	--	--	--	--	0.35	E.018	0.3	0.003	0.007	--	--	--
2	12/16/04	1530	22.5	4.8	9.96	66	69.4	0.44	50.6	0.8	<04	36.9	E.004	0.34	7.4	57.8	<2
	01/26/05	1545	35.6	4.97	10.8	64.9	65.5	0.51	50.3	0.91	E.03	27.2	E.006	0.65	7.9	58.5	<2
	04/29/05	940	13.1	4.54	11	65	66.7	0.56	50.2	0.74	<04	9.7	<008	0.94	3.3	65.7	<2
	06/17/05	1745	14.9	4.41	11.1	83.2	95.1	0.75	42.1	0.67	<04	6.07	<008	1.18	8.4	111	<2
	06/05/08	1130	--	--	--	--	--	--	--	0.75	E.018	5.69	E.001	2.21	--	--	--
	09/23/08	1200	--	--	--	--	--	--	--	0.71	0.021	7.06	<006	0.809	--	--	--
3	12/16/04	1330	7.69	3.13	2.06	7.33	7.73	E.06	6.18	0.32	<04	1.01	E.005	<02	<2.0	18.4	<2
	01/26/05	1145	8.08	3.16	1.69	8.54	10.2	E.07	6.41	0.21	<04	1.04	<008	E.01	<2.0	23.7	<2
	04/29/05	1100	8.11	3.35	1.57	7.97	8.61	0.11	5.38	0.28	<04	0.53	E.004	<02	<2.0	9.6	<2
	06/17/05	1400	8.09	3.51	2.27	10.8	11.3	0.15	6.42	0.31	<04	0.53	<008	0.04	4.8	11.3	<2
	06/05/08	1625	--	--	--	--	--	--	--	0.33	<020	0.41	0.005	0.061	--	--	--
	09/23/08	1300	--	--	--	--	--	--	--	0.3	<020	0.63	0.002	0.021	--	--	--



**Figure 18.** Distributions of concentrations of selected dissolved nutrients and ions (A) nitrate plus nitrite, as nitrogen, (B) ammonia plus organic nitrogen, (C) sulfate as sulfur, (D) magnesium, and (E) sodium, in streamwater samples from small stream sites in Durham and Orange Counties, North Carolina, December 2004 to October 2008.



**Figure 19.** Temporal variation in concentrations of dissolved ammonia nitrogen, organic nitrogen, and nitrate plus nitrite nitrogen in water samples from small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to October 2008.

Concentrations of dissolved ions generally were lowest in the samples from the undeveloped site (site 4; table 16). Ion concentrations in samples from the residential sites showed no major differences with respect to wastewater treatment except that concentrations of calcium, potassium, and sulfate were more variable in samples from the site with the suspected sewer line leak (site 6) than from the other sites (fig. 18; table 16).

Organic wastewater compounds were detected more commonly in samples of treated wastewater effluent than in surface-water samples. The polycyclic musk, hexahydro-hexamethylcyclopentabenzopyran (HHCB), was the most commonly detected organic wastewater compound in effluent samples followed by the flame retardants tris(2-chloroethyl) phosphate and tris(dichloroisopropyl) phosphate (table 17). Detections of OWWCs in samples from sites above and below the WWTP outfall were similar and no effects of the effluent were evident (table 17), although the distance of several miles from the WWTP to the downstream sampling location precludes more interpretation.

Similar to the distribution observed for OWWCs, antibiotics and pharmaceutical compounds were more commonly detected in samples from the WWTP than in samples from the river reach sites (table 18). There was only one detection of an antibiotic, azithromycin, in samples from the upstream river site. Carbamazepine, an antiepileptic medication, was the most commonly detected compound and was present in all samples from the WWTP in concentrations ranging from 0.469 to 0.303  $\mu\text{g/L}$ . Carbamazepine is recalcitrant to degradation by conventional wastewater treatment practices and is commonly found in wastewater (Zhang and others, 2008). Carbamazepine was detected downstream from the wastewater treatment plant at concentrations about 20 times lower than that in the effluent. Sulfamethoxazole, the second most commonly detected pharmaceutical compound, was detected in 5 of 6 effluent samples, with a maximum concentration of 0.475  $\mu\text{g/L}$ . Sulfamethoxazole is commonly administered in conjunction with trimethoprim, which was detected in 2 of 6 effluent samples. Neither sulfamethoxazole nor trimethoprim was detected in samples from the downstream river site. Tylosin, a veterinary antibiotic, was detected in 3 of 6 samples from the downstream site but not in samples from the WWTP. Concentrations of the endogenous hormone, 17- $\beta$ -estradiol, were lower in effluent samples from the WWTP than in samples from the upstream and downstream sites (table 18).

Detections of OWWCs in samples from the small stream sites are summarized in table 19. The fewest detections were in samples from the undeveloped site. In contrast, the largest number of detections was in samples from the site with the suspected sewer line leak (site 6). With the exception of site 6, the remaining sites had similar frequencies of detections of OWWCs. Camphor was the most commonly detected organic wastewater compound in samples from the small stream sites followed by the musks HHCB and acetyl hexamethyl tetrahydronaphthalene (AHTN) (table 19).

Antibiotic and pharmaceutical compounds were not detected in water samples from the undeveloped catchment (table 20). Antibiotic and pharmaceutical compounds were detected more commonly in samples from the sites in areas primarily served by municipal sewers than from sites in areas primarily served by onsite wastewater treatment systems. Carbamazepine was the most commonly detected of these compounds and was detected in 6 of 24 water samples from the streams draining areas with centralized wastewater treatment and 5 of 24 samples from the streams draining areas with onsite wastewater treatment. Lincomycin and tylosin, veterinary antibiotics, were detected in 4 of 24 samples from the streams draining areas with primarily centralized wastewater treatment in contrast to only 1 of 24 in samples from the streams draining areas with onsite wastewater treatment. The greater population density of the catchments in areas of centralized wastewater treatment is likely associated with a greater density of pets and therefore a larger potential source of veterinary antibiotics in these catchments.

The endogenous hormone 17- $\beta$ -estradiol was detected in water samples from all sites (table 20). The maximum detected concentration, 26 nanograms per liter (ng/L), was in a sample from site 7, which drains a catchment served by centralized wastewater treatment. Within-site concentrations of 17- $\beta$ -estradiol were variable and no clear patterns regarding wastewater treatment are present. It is important to note that wildlife and pets are also potential sources of 17- $\beta$ -estradiol.

Values for optical brighteners, methylene blue active substances (MBAS), and fecal bacteria in samples collected in 2005 are shown in table 21. The highest optical brightener value, 310 fluorescence units, was in a sample collected from the culvert (site 6C) draining the area in which the sewer line leak was suspected. Samples from the WWTP (site 3) and the site with the suspected sewer line leak (site 6) also were high relative to the other sites. The lowest values for optical brighteners were in samples from site 8, which is in an area where wastewater is treated onsite. Concentrations of MBAS were below the reporting level for all samples (table 21). Bacteria were collected on two dates in June 2005. Fecal coliform bacteria were predominately *E. coli*. The highest bacteria densities were in samples from site 9, which is in an area where wastewater is treated onsite.

Bacteria data collected in 2008 for the river reach sites indicates slight differences upstream and downstream from the wastewater treatment plant outfall (table 22). Effluent samples from the WWTP showed no or very low bacterial densities, as would be expected following chlorination. Bacterial densities for the small stream sites were variable. The lowest densities were generally in water samples from site 4, which is in an undeveloped area, and site 7, which is in an area of centralized wastewater treatment. In general, site 5, which is in an area of centralized wastewater treatment, and site 9, which is in an area of onsite wastewater treatment, had the highest bacterial densities with median most probable numbers of *E. coli* of 1,000 and 367 colonies per 100 milliliters (mL), respectively (table 23). On September 18, 2008, samples were collected for

**Table 16.** Concentrations of selected dissolved ions, nutrients, and metals in surface-water samples from the small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.

[E, concentration is less than the method reporting limit; &lt;, less than; --, not analyzed; µg/L, micrograms per liter; mg/L, milligrams per liter]

Site Number (fig. 1)	Date	TIMES		Calcium (mg/L)	Magnesium (mg/L)	Potassium (mg/L)	Sodium (mg/L)	Chloride (mg/L)	Fluoride (mg/L)	Sulfate (mg/L)	Ammonia		Nitrate plus nitrite, as N (mg/L)	Ortho- phosphate (mg/L)	Copper (µg/L)	Zinc (µg/L)	Arsenic (µg/L)	
		Sample start time									plus organic nitrogen (mg/L)	Ammonia (mg/L)						
4	UNDEVELOPED																	
	12/16/04	1000	3.79	1.29	0.63	12.8	15.6	E.05	2.94	0.22	<.04	<.06	<.08	<.02	<2.0	57.5	<2	
	01/26/05	1020	3.69	1.29	0.61	12.7	17.3	<.10	4.25	0.16	E.02	<.06	<.08	<.02	<2.0	10.9	<4	
	03/31/05	1000	2.59	1.02	0.72	8.75	10.5	<.10	3.24	0.3	<.04	<.06	E.005	<.02	<2.0	82.2	<2	
	04/29/05	1300	3.08	1.23	0.75	8.65	10.4	E.06	1.55	0.2	<.04	E.04	<.08	<.02	<2.0	55	<2	
	06/16/05	1245	4.1	1.54	0.9	8.64	8.61	E.07	0.95	0.37	0.06	0.07	0.019	E.01	<2.0	145	<2	
	04/10/08	915	--	--	--	--	--	--	--	0.18	<.020	<.04	0.004	E.004	--	--	--	
06/05/08	1015	--	--	--	--	--	--	--	0.55	0.043	E.03	0.003	0.007	--	--	--		
5	CENTRALIZED WASTEWATER TREATMENT																	
	12/17/04	1545	11	2.97	1.1	10.5	10.9	E.05	8.5	0.1	<.04	0.38	<.08	<.02	E1.1	30.7	<2	
	01/29/05	1200	11.3	2.99	1.07	11	13	E.06	9.84	0.1	E.02	0.51	E.005	<.02	<2.0	9.8	<2	
	03/31/05	1830	10.7	3.03	1.25	9.56	10.3	E.06	11.6	0.16	<.04	0.44	<.08	<.02	<2.0	26.5	<2	
	04/30/05	1945	9.16	2.43	1.67	10.2	9.56	0.1	7.86	0.4	<.04	0.41	E.005	<.02	2.3	17.2	<2	
	06/18/05	1445	7.9	2.16	1.21	7.96	7.91	E.07	5.27	0.13	E.02	0.4	<.08	0.09	<2.0	18	<2	
	04/10/08	1045	--	--	--	--	--	--	--	0.19	<.020	0.36	0.013	E.005	--	--	--	
	06/05/08	1300	--	--	--	--	--	--	--	0.18	<.020	0.34	0.004	0.013	--	--	--	
09/23/08	1045	--	--	--	--	--	--	--	0.15	0.056	0.47	E.001	0.013	--	--	--		
10/01/08	1200	--	--	--	--	--	--	--	0.38	<.020	0.53	0.011	0.012	--	--	--		
6	12/17/04	945	22.2	10.5	3.45	17.6	27.9	0.12	17	2.5	1.91	0.91	0.043	0.06	3.1	22	<2	
	01/27/05	830	27	12.9	3.22	37	64.7	0.15	19.9	3.5	2.97	0.87	0.028	0.08	3.6	31.8	<2	
	03/31/05	1100	22.1	10.4	1.64	10.8	17.3	0.19	16	0.43	<.04	0.11	0.01	<.02	5	39.4	E1	
	04/29/05	1500	24.7	11.7	3.53	12.7	20.7	0.17	9.07	0.81	0.16	0.29	0.049	E.02	3	51	<2	
	06/15/05	1715	20.4	10	2.71	8.11	11.1	0.13	9.55	0.46	<.04	E.04	<.08	E.01	6	48.2	E2	
	04/10/08	1345	--	--	--	--	--	--	--	1.9	<.020	0.07	0.02	0.006	--	--	--	
	06/05/08	1530	--	--	--	--	--	--	--	0.84	0.148	E.02	0.004	0.073	--	--	--	
	09/22/08	1430	--	--	--	--	--	--	--	0.53	<.020	<.04	0.004	0.025	--	--	--	
09/30/08	1500	--	--	--	--	--	--	--	0.59	<.020	0.07	0.003	0.012	--	--	--		
7	12/20/04	1400	14.7	3.94	2.2	16.3	17.3	E.05	11.6	0.12	<.04	0.33	<.08	<.02	E1.1	11.3	<2	
	01/28/05	1600	16.8	4.34	2.24	17	22.8	<.10	12.9	0.18	<.04	0.27	E.005	<.02	<2.0	8	<2	
	03/31/05	1700	11.3	3.36	1.98	13.5	15.3	E.09	11.9	0.22	<.04	0.57	E.004	E.01	3.1	18.1	<2	
	04/30/05	1730	13.1	3.56	2.41	15.1	17.6	0.14	11	0.17	<.04	0.2	<.08	<.02	<2.0	78.5	<2	
	06/15/05	1215	15	3.59	2.68	14.9	17	E.07	10.5	0.2	E.03	0.24	<.08	E.01	5.1	25.7	<2	
	04/10/08	1045	--	--	--	--	--	--	--	0.19	<.020	0.36	0.011	E.006	--	--	--	
	06/05/08	1250	--	--	--	--	--	--	--	0.2	<.020	0.22	0.003	0.019	--	--	--	
	09/21/08	1300	--	--	--	--	--	--	--	0.19	<.020	0.24	0.002	0.012	--	--	--	
09/30/08	930	--	--	--	--	--	--	--	0.29	<.020	0.32	0.002	0.018	--	--	--		



[E, concentration is less than the method reporting limit; <, less than; --, not analyzed;  $\mu\text{g/L}$ , micrograms per liter;  $\text{mg/L}$ , milligrams per liter]

Site Number (fig. 1)	Date	TIMES Sample start time	ONSITE WASTEWATER TREATMENT													
			Calcium (mg/L)	Magnesium (mg/L)	Potassium (mg/L)	Sodium (mg/L)	Chloride (mg/L)	Fluoride (mg/L)	Sulfate (mg/L)	Ammonia plus organic nitrogen (mg/L)	Ammonia (mg/L)	Nitrate plus nitrite, as N (mg/L)	Ortho- phosphate (mg/L)	Copper (µg/L)	Zinc (µg/L)	Arsenic (µg/L)
8	12/20/04	1600	12.1	4.02	0.68	15.9	32.6	E.09	5.17	E.07	E.02	0.75	<0.08	<2.0	E3.6	<2
	01/27/05	1600	12.6	4.11	0.78	15.8	33.9	E.07	5.12	E.06	<0.4	0.81	E.004	<2.0	12.5	<2
	04/01/05	1330	8.27	3.26	0.61	11	20.1	E.07	4.98	E.08	<0.4	0.55	<0.08	<2.0	11.5	<2
	04/30/05	1300	11.8	3.89	0.82	13.9	31.4	<10	4.57	0.11	<0.4	0.57	<0.08	<2.0	18.5	<2
	06/15/05	1000	14.8	4.38	0.77	16.5	38.2	0.19	4.52	0.11	E.02	0.66	<0.08	4	76.7	<2
	04/10/08	1130	--	--	--	--	--	--	--	E.13	<0.020	0.88	E.004	--	--	--
	06/05/08	1600	--	--	--	--	--	--	--	0.16	<0.020	0.46	0.004	--	--	--
	09/21/08	1000	--	--	--	--	--	--	--	E.12	763	0.85	E.002	--	--	--
9	09/30/08	1030	--	--	--	--	--	--	--	0.13	<0.020	1.43	0.014	0.006	--	--
	12/17/04	1345	8.44	2.93	1.93	12.6	13.9	<10	5.12	0.34	0.08	1.06	E.006	E1.4	42	<2
	01/27/05	1300	8.56	2.62	1.83	12.1	14.4	<10	7.3	0.38	0.09	0.88	E.007	<2.0	10.1	<2
	03/31/05	1500	6.63	2.39	1.79	10.6	11.9	E.07	7.05	0.41	E.03	0.76	0.008	E.01	2.8	42.6
	04/30/05	1545	9.19	2.67	1.93	11.8	14.2	E.08	4.96	0.42	0.08	0.86	0.016	0.03	<2.0	53.1
	06/17/05	1140	10.6	3.09	2.47	12	14.1	0.12	3.29	0.63	0.28	0.49	0.012	0.05	<2.0	21.8
	04/10/08	1200	--	--	--	--	--	--	--	0.4	E.012	0.4	0.079	0.006	--	--
	06/05/08	1345	--	--	--	--	--	--	--	0.55	<0.020	0.36	0.021	0.026	--	--
10	09/22/08	1130	--	--	--	--	--	--	--	0.41	<0.020	0.51	0.003	0.039	--	--
	09/30/08	1130	--	--	--	--	--	--	--	0.45	<0.020	0.63	0.025	0.015	--	--
	12/17/04	1200	12.2	4.12	1.75	23.9	34.1	E.06	10.6	0.25	0.06	0.41	E.004	E1.7	35.1	<2
	01/27/05	1030	12.2	4.07	1.78	23.4	32.5	E.06	12.4	0.54	0.27	0.44	0.008	<2.0	15	<2
	03/31/05	1300	9.07	3.28	1.68	16	19.5	E.08	10	0.43	E.04	0.26	0.01	0.04	2.2	34.1
	04/29/05	1700	14.3	4.32	2.37	19	22.7	0.17	6.9	0.43	<0.4	0.14	0.016	<0.2	E1.2	47.4
	06/17/05	1000	36.6	7.3	2.03	16.4	15.8	0.14	8.05	0.64	0.11	0.2	0.012	0.04	<2.0	314
	04/10/08	1245	--	--	--	--	--	--	--	0.4	<0.020	0.23	0.029	0.007	--	--
	06/05/08	1500	--	--	--	--	--	--	--	0.49	<0.020	0.1	0.005	0.012	--	--
	09/21/08	1600	--	--	--	--	--	--	--	0.54	<0.020	0.06	0.015	0.014	--	--
	09/30/08	1230	--	--	--	--	--	--	--	0.43	<0.020	0.17	0.027	0.014	--	--

**Table 17.** Detections of organic wastewater compounds in samples from the River Reach study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, December 2004 to June 2005. Column entries are the number of quantitative detections with the number of qualitative detections (presence confirmed but not quantifiable) in parentheses.

[WWTP, wastewater treatment plant; Analytes suspected of hormonal activity are shown in bold;  $\Sigma$  - sum of the detections]

Analyte <sup>a</sup>	Sites			$\Sigma$
	1	2	3	
	Upstream	WWTP	Downstream	
1,4-Dichlorobenzene	0(0)	0(3)	0(0)	0(3)
3-beta-Coprostanol	0(0)	0(0)	0(1)	0(1)
3-Methyl-1H-indole	0(0)	0(0)	0(1)	0(1)
4-Nonylphenol (sum of all isomers)	0(0)	0(1)	0(1)	0(2)
4-tert-Octylphenol monoethoxylate	0(0)	0(1)	0(0)	0(1)
9,10-Anthraquinone	0(0)	1(2)	0(1)	1(3)
Acetyl hexamethyltetrahydronaphthalene	0(0)	0(6)	0(1)	0(7)
Benzophenone	0(0)	1(0)	0(0)	1(0)
beta-Sitosterol	0(1)	0(0)	0(1)	0(2)
beta-Stigmastanol	0(1)	0(0)	0(0)	0(1)
Caffeine	0(2)	0(1)	0(1)	0(4)
Camphor	0(2)	0(0)	0(1)	0(3)
Carbaryl	0(0)	0(1)	0(0)	0(1)
Cholesterol	0(2)	0(1)	0(1)	0(4)
Cotinine	0(0)	0(1)	0(0)	0(1)
Fluoranthene	0(1)	0(0)	0(0)	0(1)
Hexahydrohexamethylcyclopentabenzopyran	0(0)	6(0)	0(1)	6(1)
Indole	0(0)	0(1)	0(1)	0(2)
Isophorone	0(1)	0(1)	0(1)	0(3)
Metolachlor	0(2)	0(1)	0(0)	0(3)
Naphthalene	0(1)	0(0)	0(0)	0(1)
<i>p</i> -Cresol	0(3)	0(2)	0(2)	0(7)
Pyrene	0(1)	0(0)	0(0)	0(1)
Tetrachloroethene	0(3)	0(0)	0(0)	0(3)
Tribromomethane	0(0)	0(2)	0(0)	0(2)
Tributyl phosphate	0(0)	1(1)	0(1)	1(2)
Triethyl citrate	0(0)	0(2)	0(0)	0(2)
Triphenyl phosphate	0(0)	0(1)	0(0)	0(1)
Tris(2-butoxyethyl) phosphate	0(0)	0(2)	0(0)	0(2)
Tris(2-chloroethyl) phosphate	0(0)	2(4)	0(2)	2(6)
Tris(dichloroisopropyl) phosphate	0(0)	2(4)	0(1)	2(5)
$\Sigma$	0(20)	13(38)	0(18)	13(76)

<sup>a</sup>Analytes not detected or detected above a certain threshold concentration in the laboratory set blank omitted. See text for details.

<sup>b</sup>Analytes also detected in field blanks.

**Table 18.** Concentrations of antibiotics, antibiotic metabolites, and pharmaceutical compounds detected in surface-water and wastewater-effluent samples from river reach sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.

[ng/L, nanograms per liter; µg/L, micrograms per liter; &lt;, less than, --, not analyzed; detections highlighted]

Site number (fig. 1)	Date	TIMES Sample start time	17-beta-Estradiol (ng/L)	Anhydro-erthromycin (µg/L)	Azithromycin (µg/L)	Carbamazepine (µg/L)	Ciprofloxacin (µg/L)	Ofloxacin (µg/L)	Roxithromycin (µg/L)	Sulfamethoxazole (µg/L)	Trimethoprim (µg/L)	Tylosin (µg/L)
UPSTREAM RIVER SITE												
1	Dec. 20, 2004	1100	--	<.008	0.027	<.005	<.005	<.005	<.005	<.005	<.005	<.005
	Jan. 26, 2005	1545	--	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005
	Apr. 29, 2005	830	--	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005
	June 17, 2005	1630	--	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.005
	June 5, 2008	1145	26	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.008
	Sept. 23, 2008	1115	5.1	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	<.008
WASTEWATER TREATMENT PLANT												
2	Dec. 16, 2004	1530	--	0.017	0.048	0.469	<.005	0.008	<.005	0.127	<.005	<.005
	Jan. 26, 2005	1400	--	<.008	<.005	0.343	<.005	0.026	<.005	0.475	<.005	<.005
	Apr. 29, 2005	940	--	<.008	<.005	0.303	0.008	0.026	<.005	0.289	0.01	<.005
	June 17, 2005	1745	--	<.008	<.005	0.308	<.005	0.089	<.005	<.005	<.005	<.005
	June 5, 2008	1130	<1.5	<.008	<.005	0.448	<.005	0.028	<.005	0.362	0.014	<.008
	Sept. 23, 2008	1200	4.7	0.008	<.005	0.405	<.005	<.005	<.005	0.114	<.005	<.008
DOWNSTREAM RIVER SITE												
3	Dec. 16, 2004	1330	--	<.008	0.025	0.007	<.005	<.005	0.013	<.005	<.005	0.152
	Jan. 26, 2005	1145	--	<.008	<.005	0.008	<.005	<.005	<.005	<.005	<.005	<.005
	Apr. 29, 2005	1100	--	<.008	<.005	<.005	<.005	<.005	<.005	<.005	<.005	0.04
	June 17, 2005	1400	--	<.008	<.005	0.022	<.005	<.005	<.005	<.005	<.005	0.034
	June 5, 2008	1625	12	<.008	<.005	0.018	<.005	<.005	<.005	<.005	<.005	<.008
	Sept. 23, 2008	1300	16	<.008	<.005	0.017	<.005	<.005	<.005	<.005	<.005	<.008

**Table 19.** Detections of organic wastewater compounds in samples from the small stream study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008. Column entries are the number of quantitative detections with the number of qualitative detections (presence confirmed but not quantifiable) in parentheses.

[WWT, wastewater treatment; Analytes suspected of hormonal activity are shown in bold; Σ, sum of the detections]

Analyte <sup>a</sup>	Sites						
	4	5	6	7	8	9	10
	Undeveloped	Centralized WWT			Onsite WWT		
1,4-Dichlorobenzene	0(0)	0(0)	0(0)	0(0)	0(0)	0(1)	0(0)
1-Methylnaphthalene	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(0)
2-Methylnaphthalene <sup>b</sup>	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(0)
3-beta-Coprostanol	0(2)	0(0)	0(1)	0(0)	0(1)	0(0)	0(0)
3-Methyl-1H-indole	0(1)	0(1)	0(3)	0(2)	0(0)	0(2)	0(2)
<b>4-Nonylphenol (sum of all isomers)</b>	<b>0(1)</b>	<b>0(1)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(1)</b>	0(1)
<b>4-Nonylphenol diethoxylate (sum of all isomers)</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(0)</b>	0(0)
<b>4-tert-Octylphenol monoethoxylate</b>	<b>0(1)</b>	<b>0(1)</b>	<b>0(1)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(1)</b>	0(1)
5-Methyl-1H-benzotriazole	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(0)
9,10-Anthraquinone	0(0)	0(0)	0(4)	0(1)	0(0)	0(0)	0(1)
Acetyl hexamethyl tetrahydronaphthalene	0(0)	0(2)	0(2)	0(4)	0(0)	0(5)	0(5)
Anthracene	0(0)	0(0)	0(2)	0(0)	0(0)	0(0)	0(0)
<b>Benzo[a]pyrene</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(0)</b>	0(1)
<b>Benzophenone<sup>b</sup></b>	<b>0(0)</b>	<b>1(0)</b>	<b>1(0)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(1)</b>	0(1)
beta-Sitosterol	0(2)	0(2)	0(3)	0(0)	0(1)	0(2)	0(0)
beta-Stigmastanol	0(1)	0(1)	0(2)	0(1)	0(1)	0(0)	0(1)
Caffeine <sup>b</sup>	0(0)	0(2)	2(5)	0(3)	0(0)	0(4)	0(3)
Camphor	0(6)	0(4)	2(5)	0(3)	0(6)	0(6)	0(4)
Carbaryl	0(0)	0(0)	0(1)	0(1)	0(0)	0(0)	0(1)
Carbazole	0(0)	0(0)	0(2)	0(0)	0(0)	0(0)	0(0)
Cholesterol	0(2)	0(1)	0(5)	0(2)	0(1)	0(2)	0(4)
Cotinine	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(1)
Fluoranthene	0(0)	0(0)	0(2)	0(0)	0(0)	0(0)	0(0)
Hexahydrohexamethylcyclopentabenzopyran <sup>b</sup>	0(0)	0(1)	0(1)	0(5)	0(0)	0(4)	0(5)
Indole	0(1)	0(0)	0(2)	0(2)	0(0)	0(2)	0(2)
Isoborneol	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(0)
Isophorone <sup>b</sup>	0(3)	1(1)	0(4)	0(3)	0(0)	0(4)	0(4)
Isoquinoline	0(0)	0(0)	1(0)	0(0)	0(0)	1(0)	0(0)
Menthol	0(0)	0(1)	0(3)	0(1)	0(0)	0(2)	0(1)
Methyl salicylate <sup>b</sup>	0(3)	0(2)	0(4)	0(2)	0(0)	0(2)	0(3)
Metolachlor	0(0)	0(1)	0(1)	0(0)	0(0)	0(0)	0(0)
Naphthalene <sup>b</sup>	0(1)	0(0)	0(3)	0(0)	0(0)	0(0)	0(0)
<i>p</i> -Cresol	0(3)	0(2)	0(2)	0(1)	0(2)	0(3)	0(2)
Phenanthrene <sup>b</sup>	0(1)	0(0)	0(2)	0(0)	0(0)	0(0)	0(0)
Prometon <sup>b</sup>	0(0)	1(2)	0(2)	0(0)	0(0)	0(0)	0(0)
Pyrene	0(0)	0(0)	0(3)	0(0)	0(0)	0(0)	0(0)
Tetrachloroethene	0(0)	0(0)	0(1)	0(0)	0(0)	0(0)	0(0)
Tributyl phosphate	0(1)	1(2)	1(1)	0(0)	0(1)	1(0)	1(0)
<b>Triclosan</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(1)</b>	<b>0(0)</b>	<b>0(0)</b>	<b>0(1)</b>	0(0)
Triethyl citrate	0(0)	0(0)	0(0)	0(0)	0(0)	0(1)	0(0)
Triphenyl phosphate <sup>b</sup>	0(0)	0(0)	0(3)	0(0)	0(0)	0(0)	0(0)
Tris(2-butoxyethyl) phosphate	0(1)	0(0)	2(6)	0(1)	0(0)	0(1)	1(1)
Tris(2-chloroethyl) phosphate <sup>b</sup>	0(1)	0(2)	0(2)	0(0)	0(0)	0(0)	0(0)
Tris(dichloroisopropyl) phosphate <sup>b</sup>	0(0)	0(1)	0(3)	0(1)	0(0)	0(0)	0(1)
Σ	0(32)	4(30)	9(86)	0(36)	0(13)	2(45)	2(45)

<sup>a</sup>Analytes not detected or detected above a certain threshold concentration in the laboratory set blank omitted. See text for details.<sup>b</sup>Analytes also detected in field blanks.

**Table 20.** Concentrations of antibiotics, antibiotic metabolites, and pharmaceutical compounds detected in surface-water samples from small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.

[ng/L, nanograms per liter; µg/L, micrograms per liter; &lt;, less than; --, not analyzed; detections highlighted]

Site Number (fig. 1)	Date	Time	17-beta-estradiol (ng/L)	Anhydro-erythromycin (µg/L)	Azithromycin (µg/L)	Carbamazepine (µg/L)	Ibuprofen (µg/L)	Lincomycin (µg/L)	Tylosin (µg/L)
UNDEVELOPED									
4	Dec. 16, 2004	1000	--	<.008	<.005	<.005	<.050	<.005	<.005
	Jan. 26, 2005	1020	--	<.008	<.005	<.005	<.050	<.005	<.005
	Mar. 31, 2005	1000	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 29, 2005	1300	--	<.008	<.005	<.005	<.050	<.005	<.005
	June 16, 2005	1245	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 10, 2008	915	<1.5	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1015	5.6	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 23, 2008	1000	2.6	<.008	<.005	<.005	<.050	<.005	<.008
CENTRALIZED WASTEWATER TREATMENT									
5	Dec. 17, 2004	1545	--	<.008	<.005	<.005	<.050	<.005	<.005
	Jan. 29, 2005	1200	--	<.008	<.005	0.007	<.050	<.005	<.005
	Mar. 31, 2005	1100	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 30, 2005	1945	--	<.008	<.005	<.005	<.050	<.005	<.005
	June 18, 2005	1445	--	<.008	<.005	0.005	<.050	<.005	<.005
	Apr. 10, 2008	1045	<1.5	<.008	<.005	0.006	<.050	<.005	<.008
	June 5, 2008	1300	4.3	<.008	<.005	0.005	<.050	<.005	<.008
	Sept. 23, 2008	1045	6	<.008	<.005	<.005	<.050	<.005	<.008
6	Dec. 17, 2004	945	--	<.008	<.005	<.005	<.050	<.005	<.005
	Jan. 27, 2005	830	--	0.017	<.005	<.005	<.050	<.005	0.005
	Mar. 31, 2005	1100	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 29, 2005	1500	--	<.008	<.005	<.005	<.050	<.005	0.007
	June 15, 2005	1715	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 10, 2008	1345	3.1	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1530	19	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 22, 2008	1430	<2.5	<.008	<.005	<.005	<.050	<.005	<.008
7	Dec. 20, 2004	1400	--	<.008	0.011	<.005	<.050	<.005	<.005
	Jan. 28, 2005	1600	--	<.008	<.005	<.005	<.050	<.005	<.005
	Mar. 31, 2005	1700	--	0.014	<.005	0.011	<.050	0.027	<.005
	Apr. 30, 2005	1730	--	<.008	<.005	<.005	<.050	<.005	0.019
	June 15, 2005	1215	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 10, 2008	1045	<1.5	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1250	26	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 21, 2008	1300	<2.5	<.008	<.005	<.005	<.050	<.005	<.008

**Table 20.** Concentrations of antibiotics, antibiotic metabolites, and pharmaceutical compounds detected in surface-water samples from small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, 2004–2008.—Continued

[ng/L, nanograms per liter; µg/L, micrograms per liter; <, less than; --, not analyzed; detections highlighted]

Site Number (fig. 1)	Date	Time	17-beta-estradiol (ng/L)	Anhydro-erythromycin (µg/L)	Azithromycin (µg/L)	Carbamazepine (µg/L)	Ibuprofen (µg/L)	Lincomycin (µg/L)	Tylosin (µg/L)
UNDEVELOPED									
8	Dec. 20, 2004	1600	--	<.008	0.008	<.005	<.050	<.005	0.025
	Jan. 27, 2005	1600	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 1, 2005	1330	--	<.008	<.005	0.004	<.050	<.005	<.005
	Apr. 30, 2005	1300	--	<.008	<.005	<.005	<.050	<.005	<.005
	Jun. 15, 2005	1000	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 10, 2008	1130	<1.5	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1600	19	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 21, 2008	1000	<2.5	<.008	<.005	<.005	<.050	<.005	<.008
CENTRALIZED WASTEWATER TREATMENT									
9	Dec. 17, 2004	1345	--	<.008	<.005	0.01	<.050	<.005	<.005
	Jan. 27, 2005	1300	--	<.008	<.005	0.023	<.050	<.005	<.005
	Mar. 31, 2005	1500	--	<.008	<.005	0.018	<.050	<.005	<.005
	Apr. 30, 2005	1545	--	<.008	<.005	<.005	<.050	<.005	<.005
	June 17, 2005	1140	--	<.008	<.005	0.014	<.050	<.005	<.005
	Apr. 10, 2008	1200	<1.5	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1345	5.6	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 22, 2008	1130	3	<.008	<.005	<.005	<.050	<.005	<.008
10	Dec. 17, 2004	1200	--	<.008	<.005	0.005	0.084	<.005	<.005
	Jan. 27, 2005	1030	--	<.008	<.005	<.005	<.050	<.005	<.005
	Mar. 31, 2005	1300	--	<.008	<.005	<.005	<.050	<.005	<.005
	Apr. 29, 2005	1700	--	<.008	<.005	<.005	<.050	<.005	<.005
	June 17, 2005	1000	--	<.008	<.005	0.01	<.050	<.005	<.005
	Apr. 10, 2008	1245	14	<.008	<.005	<.005	<.050	<.005	<.008
	June 5, 2008	1500	5.2	<.008	<.005	<.005	<.050	<.005	<.008
	Sept. 21, 2008	1600	3.1	<.008	<.005	<.005	<.050	<.005	<.008



**Table 21.** Values for optical brighteners, methylene–blue active substances, and fecal bacteria at study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, January to June, 2005.[MBAS, methylene–blue active substances; *E. coli*, *Escherichia coli*; mg/L, milligrams per liter; MPN, most probable number; col/100 mL, colonies per 100 milliliters; %, percent; <, less than; --, not analyzed; WWTP, wastewater–treatment plant]

Site category	Site number (table 1)	Optical brighteners				MBAS	Fecal coliform density	<i>E. coli</i> density	Percentage <i>E.coli</i>	Fecal coliform density	<i>E. coli</i> density	Percentage <i>E.coli</i>
		(fluorescence units)				(mg/L)	MPN (col/100 ml) <sup>†</sup>	MPN (col/100 ml) <sup>†</sup>	(%)	MPN (col/100 ml) <sup>†</sup>	MPN (col/100 ml) <sup>†</sup>	(%)
		January 2005	April 2005	1 June 2005	16 June 2005	April 2005	1 June 2005			16 June 2005		
RIVER REACH SITES												
upstream	1	86.4	57.8	58.8	62.5	<0.10	40(20–140)	46(20–140)	100	49(20–170)	33(17–77)	67
WWTP	2	161	129	141	119	<0.10	49(20–150)	49(20–150)	100	46(15–130)	46(15–130)	100
downstream	3	76.1	52.2	60.8	63.1	<0.10	31(10–110)	31(10–110)	100	23(9–86)	23(9–86)	100
SMALL STREAM SITES												
undeveloped	4	99	105	71.8	81.5	<0.20	23(9–86)	23(9–86)	100	140(60–340)	140(60–340)	100
centralized	5	34.3	121	38.3	45.9	<0.10	70(30–210)	70(30–210)	100	240(100–940)	240(100–940)	100
centralized	6	128	179	164	128	0.15‡	11(4–29)	11(4–29)	100	130(50–390)	130(50–390)	100
centralized	6C	--	--	191	310	--	--	--	--	130(50–390)	49(20–170)	38
centralized	7	46.2	44.1	30.8	42.6	<0.10	23(9–86)	23(9–86)	100	130(50–390)	130(50–390)	100
onsite	8	20.6	20.4	20.2	14.1	<0.20	7(2–20)	7(2–20)	100	170(70–480)	170(70–480)	100
onsite	9	98.5	88.9	82.8	110	<0.20	350(160–820)	350(160–820)	100	540(200–2,000)	540(200–2,000)	100
onsite	10	107	148	191	136	<0.20	13(5–38)	13(5–38)	100	240(1–940)	240(1–940)	100

<sup>†</sup>value shown in parentheses is the 95% confidence interval for the MPN.<sup>‡</sup>estimated concentration.**Table 22.** Bacteriological data for samples of surface water and treated effluent at the river reach sites in the upper Neuse River Basin, Orange County, North Carolina, June to September, 2008.[WWTP, Wasterwater Treatment Plant; CFU, colony forming units; *E. coli*, *Escherichia coli*; MPN, most probably number; mL, milliliter; <, less than; %, percent]

Site Number (fig. 1)	Site category	Collection Date	Fecal coliforms (CFU/100 mL)	Enterocci (CFU/100 mL)	<i>E. coli</i>		
					MPN/100 mL	lower 95% confidence interval	upper 95% confidence interval
1	upstream	6/5/2008	440	60	78	52	110
		9/23/2008	60	10	48	29	72
2	WWTP	6/5/2008	<1	<1	<1	0	4
		9/23/2008	<1	<1	2	0	7
3	downstream	6/5/2008	260	10	17	8	31
		9/23/2008	100	10	52	34	77

**Table 23.** Bacteriological data for samples of surface water at the small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, April to September 2008.

[MPN, most probable number; col/100 mL, colonies in 100 milliliters; *E. coli*, *Escherichia coli*; %, percent; <, less than; --, not analyzed; red highlight; samples collected on September 18, 2008; WWT, wastewater treatment]

Site Number (fig. 1)	Site Category	Date of sample collection	Fecal coliforms MPN (col/100 ml)	Enterococci MPN (col/100 ml)	<i>E. coli</i>		
					MPN (col/100 ml)	Lower 95% confidence interval	Upper 95% confidence interval
4	Undeveloped	04/10/08	12	2	1	0	6
		06/05/08	370	160	372	251	538
		09/18/08	--	--	124	88	170
		09/21/08	148	74	95	66	132
		09/23/08	100	80	109	76	151
		09/30/08	90	<1	104	72	150
		median	100	74	107	--	--
5	Centralized WWT	04/10/08	366	114	166	115	238
		06/05/08	660	210	476	330	680
		09/18/08	--	--	1,380	1,037	1,800
		09/21/08	1,000	750	1,000	715	1,380
		09/23/08	1,910	220	1,510	1107	1,990
		median	830	215	1,000	--	--
5A		09/18/08	--	--	437	303	630
		09/21/08	1,030	1,680	957	682	1,305
		09/23/08	2,470	2,500	1,830	1,410	2,349
5B		09/18/08	--	--	957	682	1,305
		09/21/08	540	30	282	212	364
		09/23/08	50	10	72	48	102
5C		09/18/08	--	--	489	396	604
		09/21/08	280	20	217	155	300
		09/23/08	480	50	394	288	522
		median	510	40	437	--	--
6	Centralized WWT	04/10/08	50	122	32	21	47
		04/10/08	90	4	20	12	31
		06/05/08	790	100	216	154	294
		09/18/08	--	--	95	68	128
		09/22/08	170	<1	65	43	96
		09/30/08	440	20	182	133	242
		median	170	60	80	--	--
6C		04/10/08	204	95	101	74	134
		09/18/08	--	--	230	178	291
6D		09/18/08	--	--	1,100	800	14,400
6E		09/22/08	320	120	259	190	349
		09/18/08	--	--	873	674	1,100
6F		09/30/08	250	20	167	119	228
		06/05/08	440	390	413	294	566
		09/22/08	630	80	202	149	270
6G		09/18/08	--	--	355	267	454
		09/18/08	--	--	585	428	777
6H		09/30/08	440	90	297	211	400
		09/22/08	450	30	202	148	270

**Table 23.** Bacteriological data for samples of surface water at the small stream sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, April to September 2008.—Continued

[MPN, most probable number; col/100 mL, colonies in 100 milliliters; *E. coli*, *Escherichia coli*; %, percent; WWT, wastewater treatment; <, less than; --, not analyzed; red highlight; samples collected on September 18, 2008]

Site Number (fig. 1)	Site Category	Date of sample collection	Fecal coliforms	Enterococci	<i>E. coli</i>		
			MPN (col/100 ml)	MPN (col/100 ml)	MPN (col/100 ml)	Lower 95% confidence interval	Upper 95% confidence interval
7	Centralized WWT	04/10/08	58	14	12	7	21
		06/05/08	20	10	32	18	51
		09/18/08	--	--	122	87	167
		09/21/08	240	<1	240	166	330
		09/22/08	180	10	173	119	228
		09/23/08	140	40	97	68	136
		09/30/08	400	10	420	291	602
		median	160	10	122	--	--
7A		09/18/08	--	--	794	550	1,111
		09/21/08	<1	30	10	5	24
8	Onsite WWT	04/10/08	108	6	17	10	27
		06/05/08	210	180	142	101	197
		09/18/08	--	--	224	160	308
		09/21/08	510	70	252	184	344
		09/23/08	220	20	203	149	272
		09/30/08	360	320	267	196	349
		median	220	70	213	--	--
9	Onsite WWT	04/10/08	252	184	142	101	197
		06/05/08	460	160	597	414	846
		09/18/08	--	--	367	269	486
		09/22/08	290	220	237	169	325
		09/30/08	2,640	120	1,658	1,248	2,217
		median	375	172	367	--	--
9A		09/22/08	480	160	597	414	846
9B		06/05/08	570	370	420	291	602
		09/18/08	--	--	499	356	682
9C		09/22/08	660	10	197	145	267
		06/05/08	330	10	43	26	67
		09/18/08	--	--	442	341	566
9D		09/22/08	890	210	722	515	997
		09/18/08	--	--	519	370	687
10	Onsite WWT	04/10/08	264	16	68.3	48.7	93
		06/05/08	540	130	102	71	143
		09/18/08	--	--	832	592	1,110
		09/21/08	860	100	229	163	311
		09/30/08	470	130	291	213	386
		median	505	115	229	--	--
10A		09/18/08	--	--	467	351	614

*E. coli* at all sites, including the subbasins, and are highlighted in red. The largest bacterial densities were 1,380 colonies per 100 mL at site 5 and 1,100 colonies per 100 mL at site 6D, a ditch in the area of the suspected sewer line leak. The high bacterial densities combined with positive detections of optical brighteners (table 24) support the suspicion of a sewer line leak in this area. A positive optical brightener detection also was observed at site 6C, which is downstream from 6D (table 24). The bacterial densities also were high at several sampling sites within the site 5 catchment and may indicate sewer line leaks. In general, no clear differences with regard to wastewater treatment category were evident with respect to bacteria.

**Table 24.** Presence of optical brighteners in samples of surface water and wastewater effluent from study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, April to June, 2008.

[Analytical method conforms to that of Hartel and others (2007); NEG, negative; POS, positive; EQ, equivocal; WWC, wastewater compound; WWT, wastewater treatment]

Site number	Category	Collection Date	Result
RIVER REACH SITES			
1	Upstream	06/05/08	NEG
2	WWTP	06/05/08	POS
3	Downstream	06/05/08	NEG
SMALL STREAM SITES			
4	Background	04/10/08	NEG
5	Centralized WWC	04/10/08	NEG
6	Centralized WWC	04/10/08	NEG
6C	Centralized WWC	04/10/08	POS
6D	Centralized WWC	04/10/08	POS
6E	Centralized WWC	04/10/08	NEG
7	Centralized WWC	04/10/08	NEG
8	Onsite WWT	04/10/08	NEG
9	Onsite WWT	04/10/08	NEG
9	Onsite WWT	04/10/08	NEG
10	Onsite WWT	04/10/08	NEG
4	Background	06/05/08	NEG
5	Centralized WWC	06/05/08	NEG
6	Centralized WWC	06/05/08	EQ
6E	Centralized WWC	06/05/08	NEG
7	Centralized WWC	06/05/08	NEG
8	Onsite WWT	06/05/08	NEG
9	Onsite WWT	06/05/08	NEG
9B	Onsite WWT	06/05/08	NEG
9A	Onsite WWT	06/08/08	NEG
10	Onsite WWT	06/05/08	NEG

## Elemental Quality of Streambed Sediment, 2005

Concentrations of selected elements in streambed sediments generally were lowest in samples from the undeveloped site (site 4), which suggests that urban land-use activities contribute to elevated levels of some elements in streambed sediments (table 25; fig. 20). With the exception of organic carbon, elemental concentrations were similar or greater in the sample collected at the site upstream (site 1) from the WWTP outfall than at the downstream site (site 3), which indicates that there are minimal effects of WWTP effluent on elemental streambed-sediment chemistry at the downstream site (site 3). The higher concentration of organic carbon likely is related to increased biomass associated with the greater nutrient availability downstream from the WWTP outfall (figs. 3, 4). Concentrations of chromium, cobalt, nickel, and vanadium were more than two times greater in the sample from site 6 than in samples from the other residential small stream sites (fig. 20) and possibly could be related to the suspected sewer-line leak at this site. Chromium is a component of domestic wastewater and was identified in various household products (Tjandraatmadja and others, 2010).

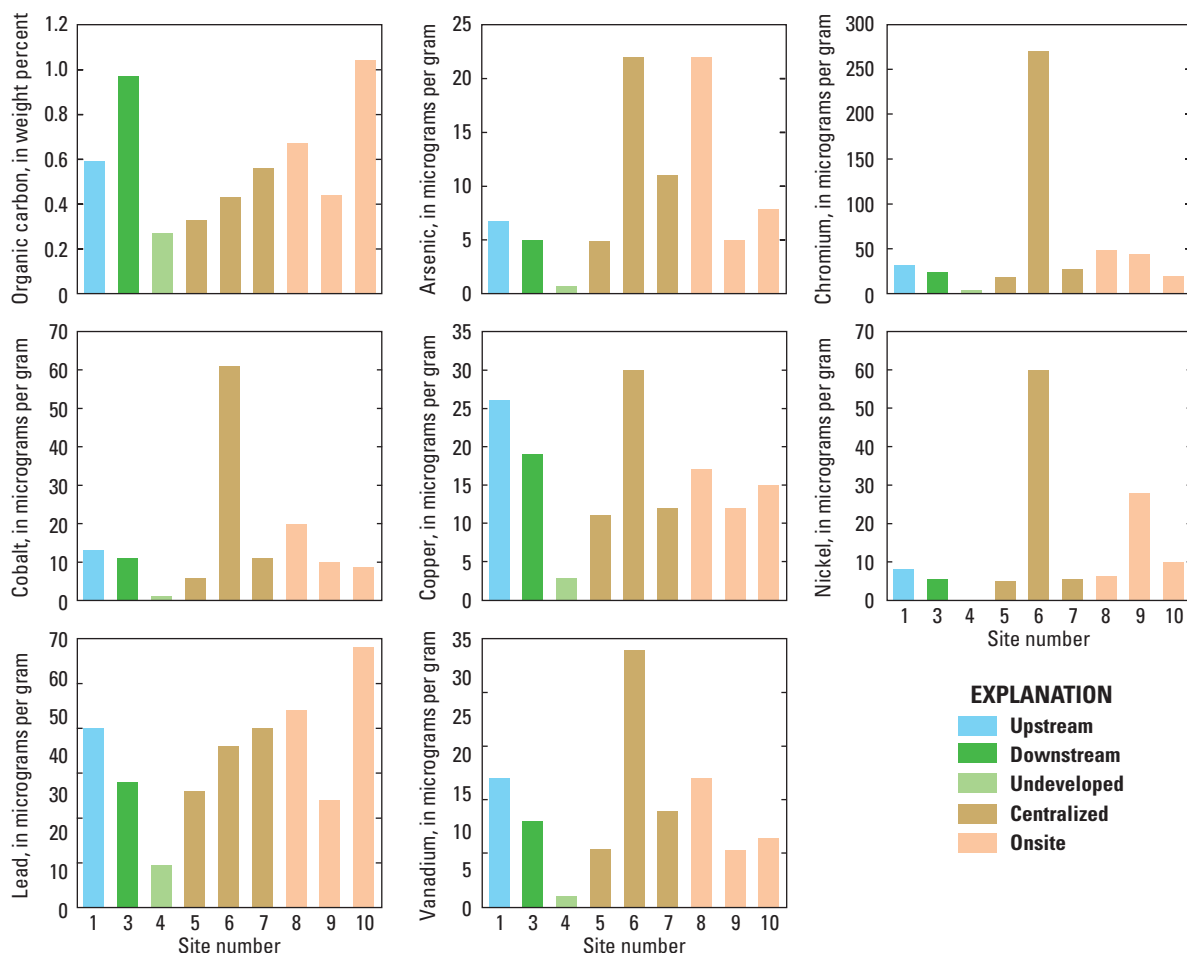
Although these elements occur naturally, there are many anthropogenic sources in addition to domestic wastewater. Concentrations of many elements, derived from various sources, are typically elevated in urban streambed sediments (Khamar and others, 2000; Davis and others, 2001; Van Metre and Mahler, 2003). Urbanization has been linked to elevated concentrations of metals, especially zinc, chromium, copper, nickel, and cadmium, in water and streambed sediments from New Jersey streams (Wilber and Hunter, 1977). Elevated concentrations of these metals, which are derived from brake linings, tires, and engine wear, also are found in runoff from roadways (Adachi and Tainosho, 2004; Councell and others, 2004; Lee and others, 2005; Lough and others, 2005). Natural differences in the elemental composition of the underlying geology in the study area also contribute to the observed variation in streambed-sediment chemistry.

**Table 25.** Concentrations of selected elements in streambed sediments collected on June 1, 2005, from study sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina.

[WWT, wastewater treatment; %, percent; µg/g, micrograms per gram; &lt;, less than]

Element <sup>a</sup>	Reporting Level	Unit	River reach sites		Small stream sites						
			Up-stream	Down-stream	Un-developed	Centralized WWT				Onsite WWT	
			site 1	site 3	Site 4	Site 5	Site 6	Site 7	Site 8	Site 9 <sup>b</sup>	Site 10
Aluminum	0.005	%	4.5	3.9	0.97	4.2	4.0	5.2	4.9	4.7	4.8
Antimony	0.1	µg/g	0.78	0.65	<0.1	0.36	0.72	0.71	1.1	0.4	0.55
Arsenic	0.1	µg/g	6.7	5	0.68	4.9	22	11	22	5.0	7.9
Barium	1	µg/g	260	340	100	410	530	540	470	300	380
Beryllium	0.1	µg/g	0.76	0.83	0.19	0.95	1.2	1.7	1.3	0.93	1.0
Cadmium	0.1	µg/g	<0.1	<0.1	<0.1	<0.1	0.12	<0.1	<0.1	<0.1	<0.1
Calcium	0.005	%	1.7	1.3	0.059	0.22	1.8	0.48	0.40	1.1	0.49
Carbon, total	0.01	%	0.61	1.03	0.28	0.34	0.45	0.57	0.69	0.45	1.05
Carbon, inorganic	0.01	%	0.02	0.06	<0.01	<0.01	0.02	0.01	0.02	0.01	0.01
Carbon, organic	0.01	%	0.59	0.97	0.27	0.33	0.43	0.56	0.67	0.44	1.04
Cerium	1	µg/g	29	24	5.7	19	37	36	40	22	27
Chromium	1	µg/g	32	24	3.9	18	270	27	48	44	20
Cobalt	1	µg/g	13	11	1.0	5.7	61	11	20	10	8.6
Copper	1	µg/g	26	19	2.8	11	30	12	17	12	15
Gallium	1	µg/g	11	8.6	2.0	9.1	8.2	12	13	9.2	11
Iron	0.005	%	4.2	3.6	0.31	2.2	9.4	4.8	6.0	3.0	3.2
Lanthanum	1	µg/g	14	12	2.8	8.7	12	14	16	10.4	12
Lead	1	µg/g	20	14	4.8	13	18	20	22	12	29
Lithium	1	µg/g	7.0	7.0	3.0	5.5	7.2	16	18	9.0	12
Magnesium	0.005	%	0.37	0.31	0.03	0.28	0.94	0.31	0.34	0.80	0.40
Manganese	4	µg/g	960	1,000	160	510	2,000	1,200	1400	570	890
Mercury	0.02	µg/g	0.02	0.02	<0.02	<0.02	<0.02	<0.02	0.03	0.02	0.02
Molybdenum	0.5	µg/g	0.95	0.56	<0.5	5	0.86	0.61	0.96	<0.5	<0.5
Neodymium	1	µg/g	14	13	2.2	8.3	13	12	15	9.6	11
Nickel	2	µg/g	8.1	5.6	<2	5.0	60	5.5	6.3	28	10
Niobium	4	µg/g	<4	4.0	4.0	4.0	<4	5.7	5.7	5.1	6.3
Phosphorus	0.005	%	0.045	0.052	<0.005	0.027	0.048	0.038	0.055	0.035	0.040
Potassium	0.005	%	0.57	0.66	0.25	1.3	0.50	1.5	1.0	0.80	1.2
Scandium	2	µg/g	13	9.3	<2	5.8	17	6.1	11	8.1	7.0
Selenium	0.1	µg/g	0.41	0.18	<0.1	0.29	0.42	0.41	0.40	0.18	0.32
Silver	0.1	µg/g	<0.1	<0.1	<0.1	0.24	<0.1	0.10	0.30	0.10	0.12
Sodium	0.005	µg/g	0.56	0.80	0.23	0.68	0.82	0.85	0.67	1.5	0.86
Strontium	2	µg/g	260	200	16	72	130	130	120	120	83
Thorium	1	µg/g	3.6	2.8	1.1	3.4	3.4	6.5	4.4	3.1	4.3
Tin	1	µg/g	11	1.6	<1	1.2	<1	1.3	1.4	1.0	1.2
Titanium	0.005	µg/g	0.32	0.39	0.10	0.20	0.26	0.37	0.47	0.27	0.31
Uranium	0.1	µg/g	1.4	1.6	0.58	1.2	2.1	2.6	1.7	1.4	1.9
Vanadium	2	µg/g	120	80	9.5	54	240	89	120	53	64
Yttrium	1	µg/g	11	12	2.0	5.8	13	10	13	9.6	10
Ytterbium	1	µg/g	1.4	1.4	<1	<1	1.6	1.4	2.0	1.4	1.4
Zinc	2	µg/g	70	57	14	43	83	56	71	54	64

<sup>a</sup>Concentrations of bismuth, europium, gold, holmium, tantalum, thallium, and sulfur were less than reporting levels in all samples and are not shown.<sup>b</sup>Concentrations are the mean of two replicate samples.



**Figure 20.** Elemental concentrations of organic carbon, arsenic, chromium, cobalt, copper, nickel, lead, and vanadium in streambed-sediment samples from sites in the upper Neuse River Basin, Durham and Orange Counties, North Carolina, June 1, 2005.

## Water Quality During Stormflow Recession Periods, May 2012–January 2013

Fluorescence, specific conductance, dissolved nutrient concentrations, estrogen hormone concentrations, and optical brighteners were measured in stream samples during stormflow recession events. No estrogen hormones were detected in any of these samples. Fluorescence, measured at a wavelength of 347 nanometers (nm), is considered to be an indicator of naturally occurring organic compounds as well as optical brighteners and can be used to indicate relative amounts of these compounds. Data obtained during 11 recession events are graphically presented in figures 21–31, and the results are shown in appendix 7 (including nutrients and specific conductance), appendix 8 (fluorescence), appendix 9 (optical brighteners), and appendix 10 (stage data).

In general, rainfall patterns appear to have a large effect on the response of the stream with regard to specific conductance, fluorescence, and nutrient concentrations. An effort was made to

collect samples during times of groundwater discharge. However, rainfall events are variable and factors such as antecedent soil moisture conditions and intensity of rainfall affects soil infiltration and in turn, groundwater discharge. Specific conductance generally increased during the recession period. Patterns observed in fluorescence, considered to be an indicator of the relative amount of dissolved organic carbon, were variable both between and within sites and are likely related to rainfall patterns and relative amounts of overland flow compared to groundwater reaching the stream. Observed patterns in nutrient concentrations varied with respect to site and recession event. In general, nutrient concentrations were higher during recession events than in the baseflow samples collected during 2004–2008.

Site 4 (fig. 5), in an undeveloped catchment in Duke Forest, was sampled in May and September 2012. No optical brighteners were detected during either recession event (appendix 9a). Diurnal fluctuations in specific conductance and fluorescence are apparent in the May 2012 recession data (fig. 21; appendixes 7, 8a) and likely reflect evapotranspiration during the growing season. This pattern was less apparent during



the September 2013 recession period (fig. 22; appendixes 7, 8a). Concentrations of nutrients were low, typically less than reporting levels, and showed slight change during the recession (fig. 22; appendix 7).

At Site 6 (fig. 8), a large amount of impervious surface (table 1) is in the upper part of the catchment resulting rapid flow of stormwater that has eroded the stream channel. In response, the channel has been stabilized with rock (fig. 9). Stormflow recession events were sampled during May 2012 and from December 2012 to January 2013, and graphs of resulting data are provided in figures 23 and 24. The response of the stream to runoff from several precipitation periods in December 2012 and January 2013 is shown by the rapid declines in specific conductance following rainfall events (fig. 24; appendix 7). Optical brighteners were detected in samples collected during both stormflow recession events and were more common in the May sampling period (fig. 23; appendix 9c) than the December–January period (fig. 24; appendix 9c). Optical brightener detections appear to occur about 2 days after each of the December 2012 rainfall events (fig. 24; appendix 9c). Nutrient concentrations in samples collected during the May 2012 recession period are lower and show less fluctuation over time than those for the December–January period (appendix 7). The higher rates of biological activity in May could contribute to the lower stream nutrient concentrations during that time. Nitrate plus nitrite concentration generally declined during the December recession event (fig. 24; appendix 7), with the highest concentrations occurring about 24 hours following the December 26, 2012, precipitation event.

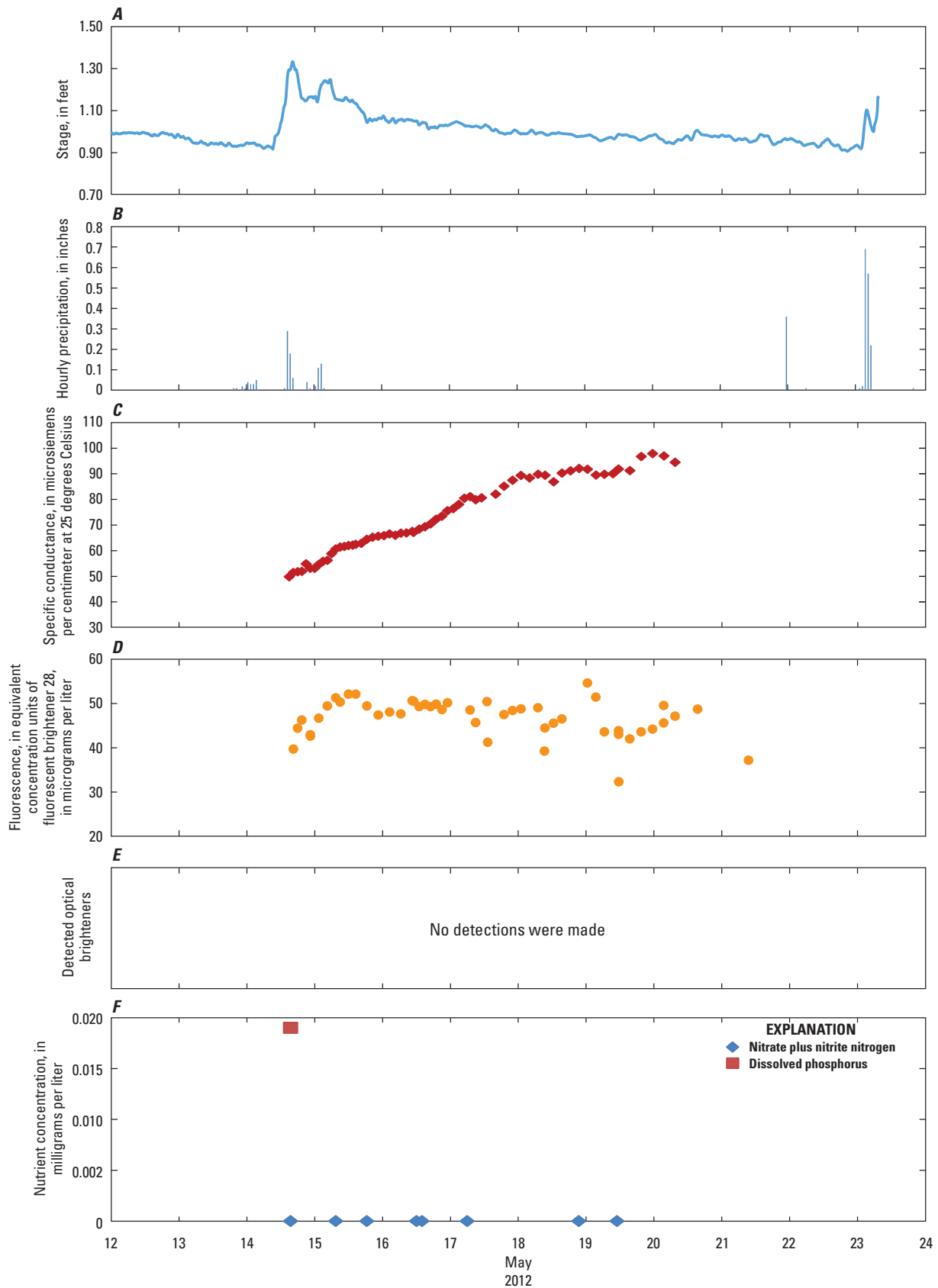
Stormflow recession events were sampled in May 2012 and December 2012 to January 2013 at site 8, a residential catchment served by onsite wastewater treatment systems. Stage, precipitation, specific conductance, fluorescence, optical brightener, and nutrient data associated with these sampling events are provided in figures 25 and 26 and appendixes 7, 8c, 9e, 10e, and 10f. Fluorescence initially increased, then decreased during the recession, which suggests that water rapidly drained from the soil. Nutrient concentrations were higher during the December event than during the May recession event. Nutrient concentrations rose following rainfall during both events (fig. 26). Optical brighteners were detected at both sites following precipitation (figs. 25, 26) at intervals about 4 days after the initial precipitation event.

Site 9 is a residential catchment served primarily by onsite wastewater treatment (table 1). A number of sand filter treatment systems are in this catchment, many of which are adjacent to Crooked Creek tributary. Stormflow recession events were monitored during October 2012 and December 2012 to January 2013 (figs. 27 and 28). Optical brighteners were detected throughout both recession events as well as in samples collected prior to the rainfall for these events (appendix 9g). Sand filter systems commonly do not discharge during dry periods and much of the summer (oral commun., R. Jordan, Durham County Health Department). In mid-September, discharges were observed from sand filter systems in the vicinity of site 9. Thus, it is likely that effluent from the sand filter treatment systems contributed to the observed detections of optical brighteners, especially prior

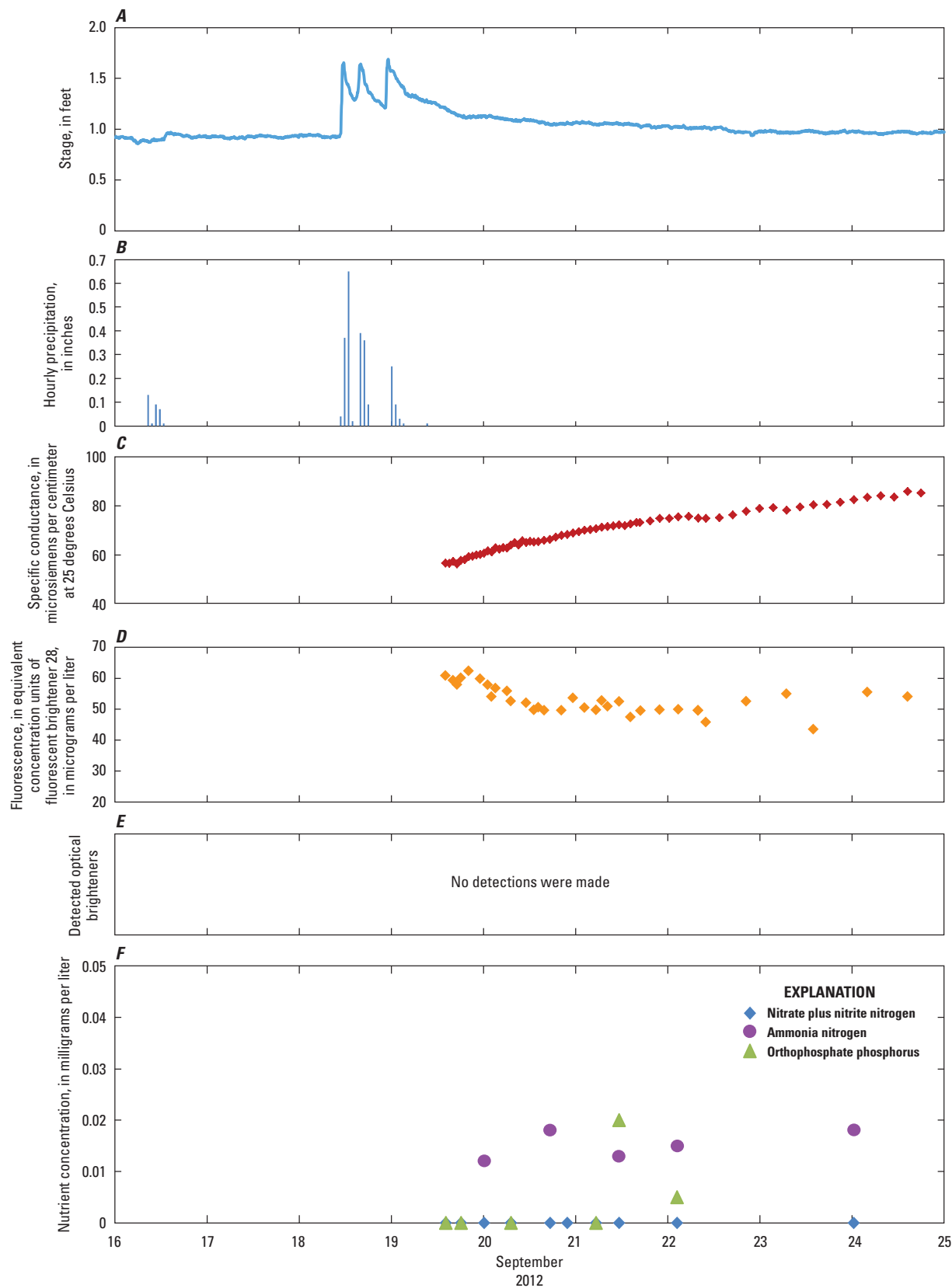
to precipitation events, when the discharge from these systems constituted a greater percentage of the streamflow than during stormflow recession periods where runoff and groundwater discharge were significant components of streamflow. Nutrient concentrations generally increased following precipitation (figs. 27 and 28; appendix 7). Nutrient concentrations were slightly higher during the December–January stormflow recession than during the October 2012 period.

Site 10 is a catchment served by onsite wastewater treatment (table 1). Several sand filter wastewater treatment systems are in the catchment. Samples were collected during three stormflow recession periods at this site, September 2012, October 2012, and December 2012 to January 2013. Stage, precipitation, specific conductance, fluorescence, optical brighteners, and nutrient data are graphically presented for these recession events in figures 29–31 and in appendixes 7, 8e, 9i 10i, 10j, and 10k. Detections of optical brighteners were common during the September 2012 recession and likely reflect the seasonal onset of discharge from sand filter systems (fig. 29). Optical brighteners were also detected in many of the samples preceding the precipitation event for the October 2012 sampling period (fig. 30). Interestingly, no optical brighteners were detected following the precipitation event preceding the October 2012 recession period. Optical brighteners were detected in samples collected during the December 2012 to January 2013 recession event (fig. 31). Nutrient concentrations generally increased in conjunction with precipitation, as can be seen in figure 29, and in association with precipitation that occurred following the initial event of these recession periods (figs. 30 and 31). Nutrient concentrations in samples collected during the September sampling event generally were lower than in samples collected during the other events, with the highest nutrient concentrations being associated with the December to January recession period (figs. 29–31). None of the nitrate plus nitrite concentrations exceeded 1 mg/L.

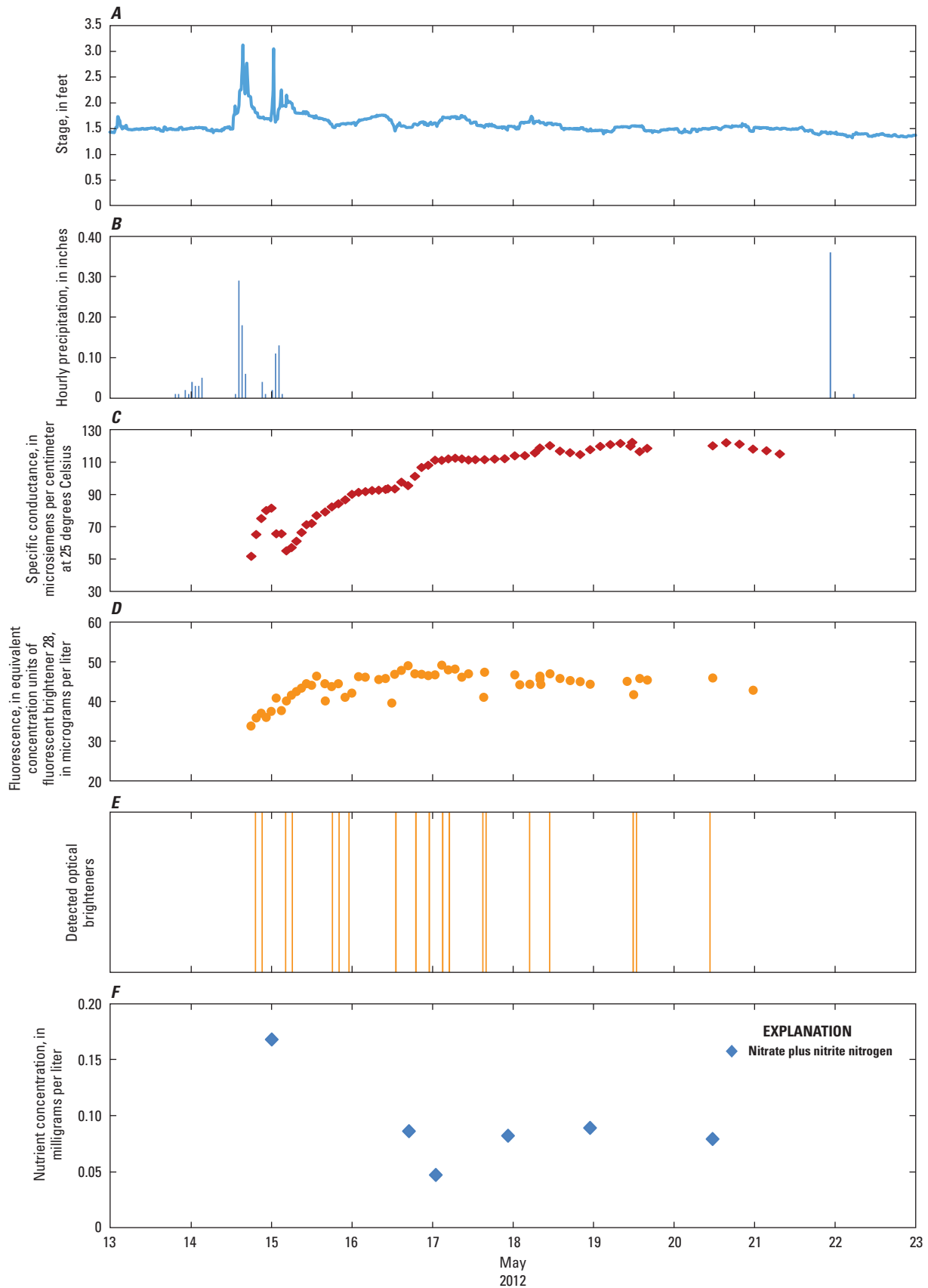
The association of optical brightener detections with stormflow recession events and wastewater discharge is uncertain, based on available data. Discharge of treated effluent to streams from sand filter wastewater treatment systems is a confounding factor and was observed at sites 9 and 10. Records of the Durham County Health Department indicate that no sand filter systems have been identified in the catchment of site 8. In general, the occurrence of optical brightener detections did not correspond to increases in nutrient concentrations, which suggests that wastewater associated with the optical brighteners had adequate treatment to remove much of nutrients in the waste stream. The higher nutrient concentrations observed in samples collected during stormflow recession events in comparison to during baseflow conditions suggests that shallow groundwater discharge is an important source of nutrients in these streams. Land use of the study sites likely affects the nutrient composition of shallow groundwater, including fertilizer applications to lawns, onsite wastewater treatment, and vegetative buffers along the stream channels. Nutrient concentrations generally were higher during the winter months, for both stormflow recession and baseflow samples, which indicates that biological activity is an important component of nutrient discharge to streams.



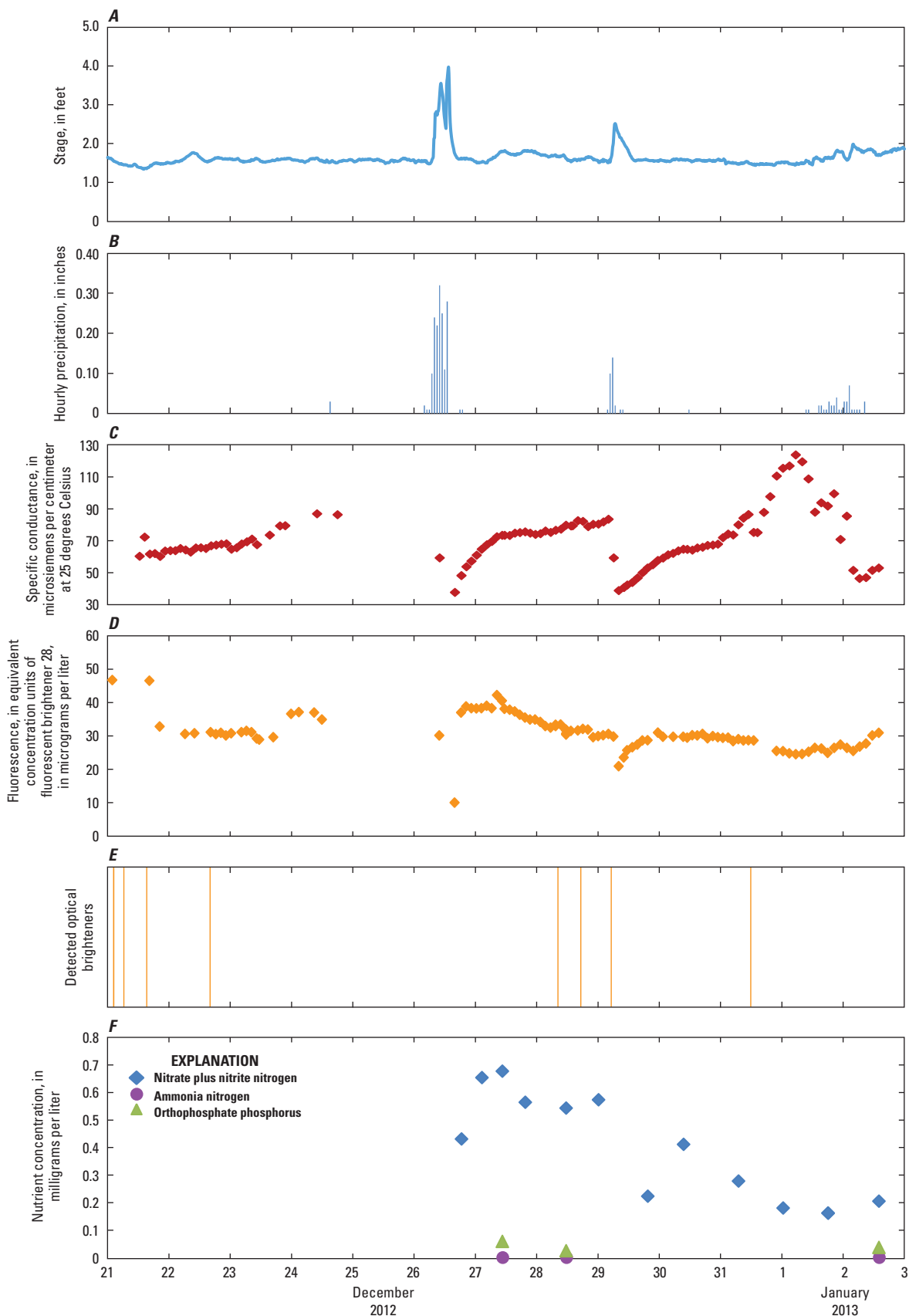
**Figure 21.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina, May 12–24, 2012.



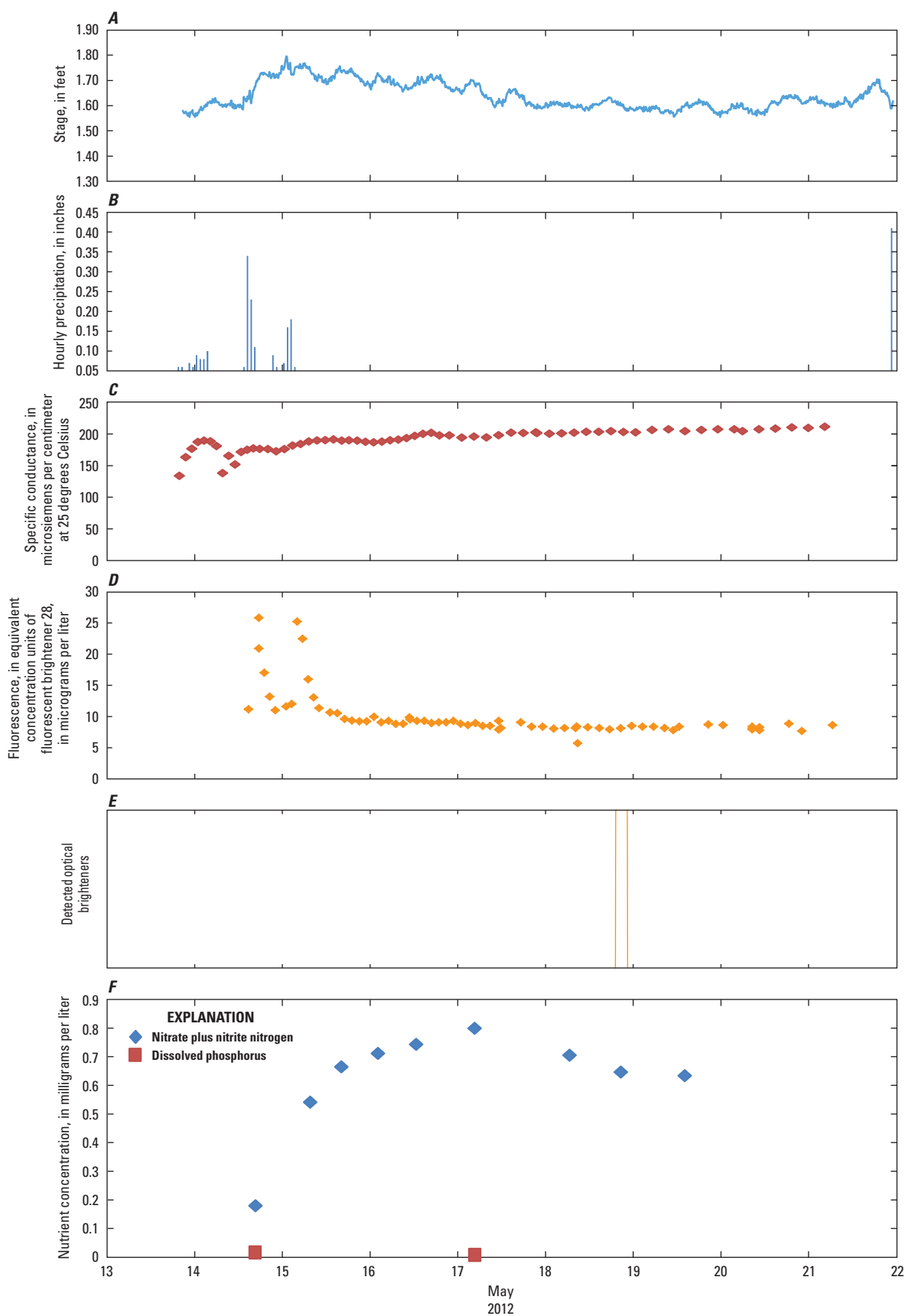
**Figure 22.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Rhodes Creek tributary above North Carolina Highway 751 near Durham, North Carolina, September 16–25, 2012.



**Figure 23.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Black Meadow Run at Argonne Drive near Durham, North Carolina, May 14–22, 2012.

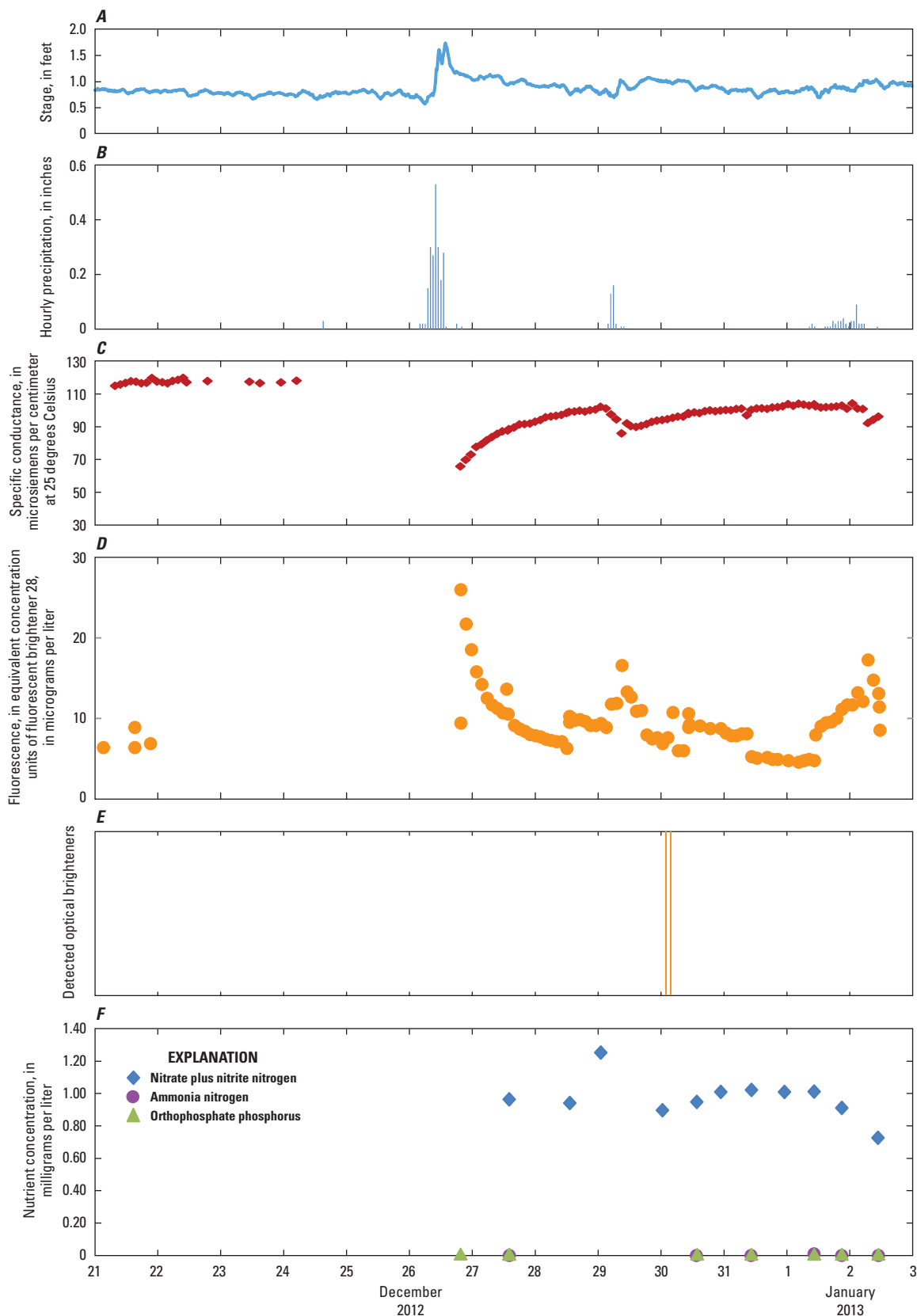


**Figure 24.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Black Meadow Run at Argonne Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.

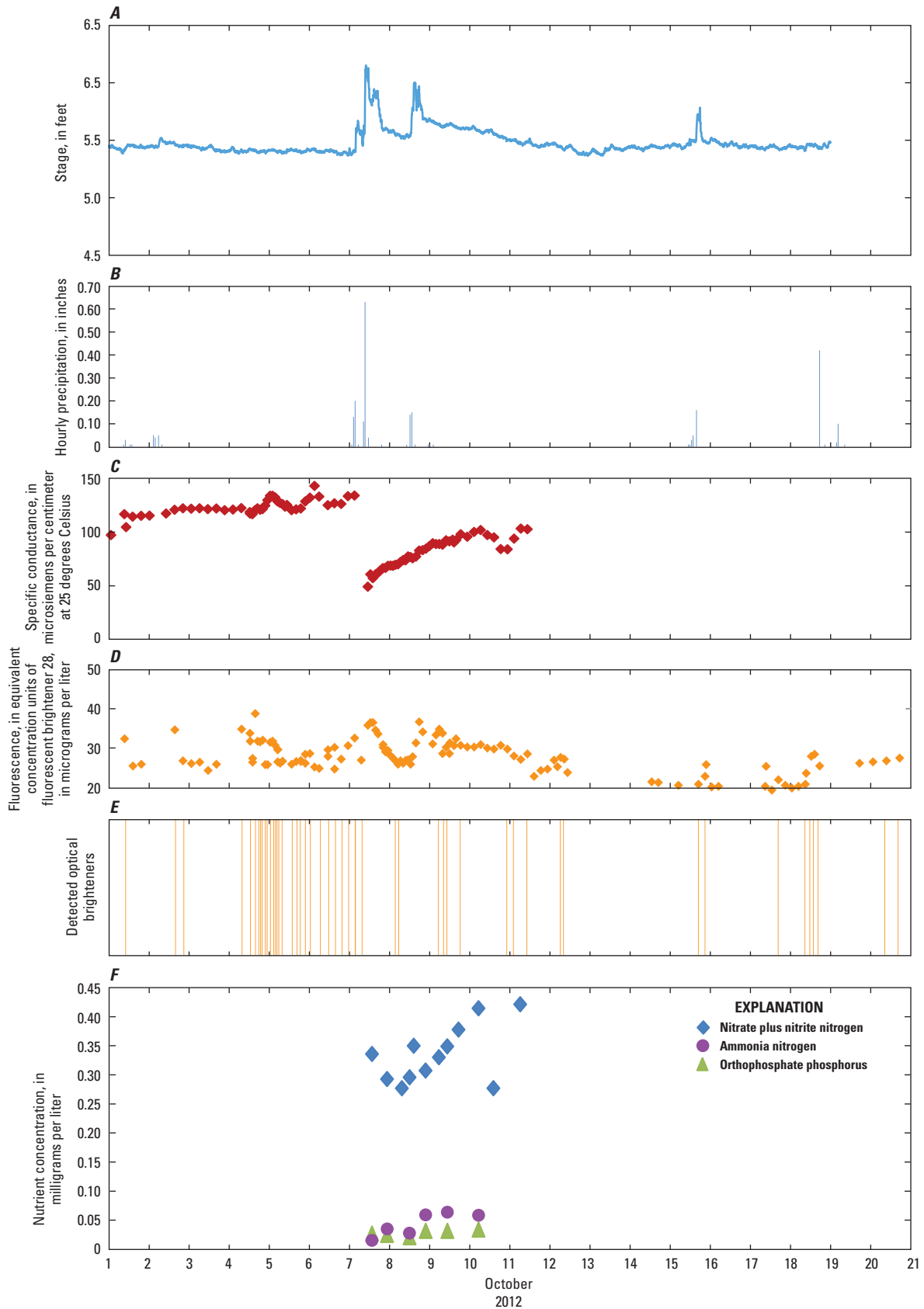


**Figure 25.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, May 14–21, 2012.

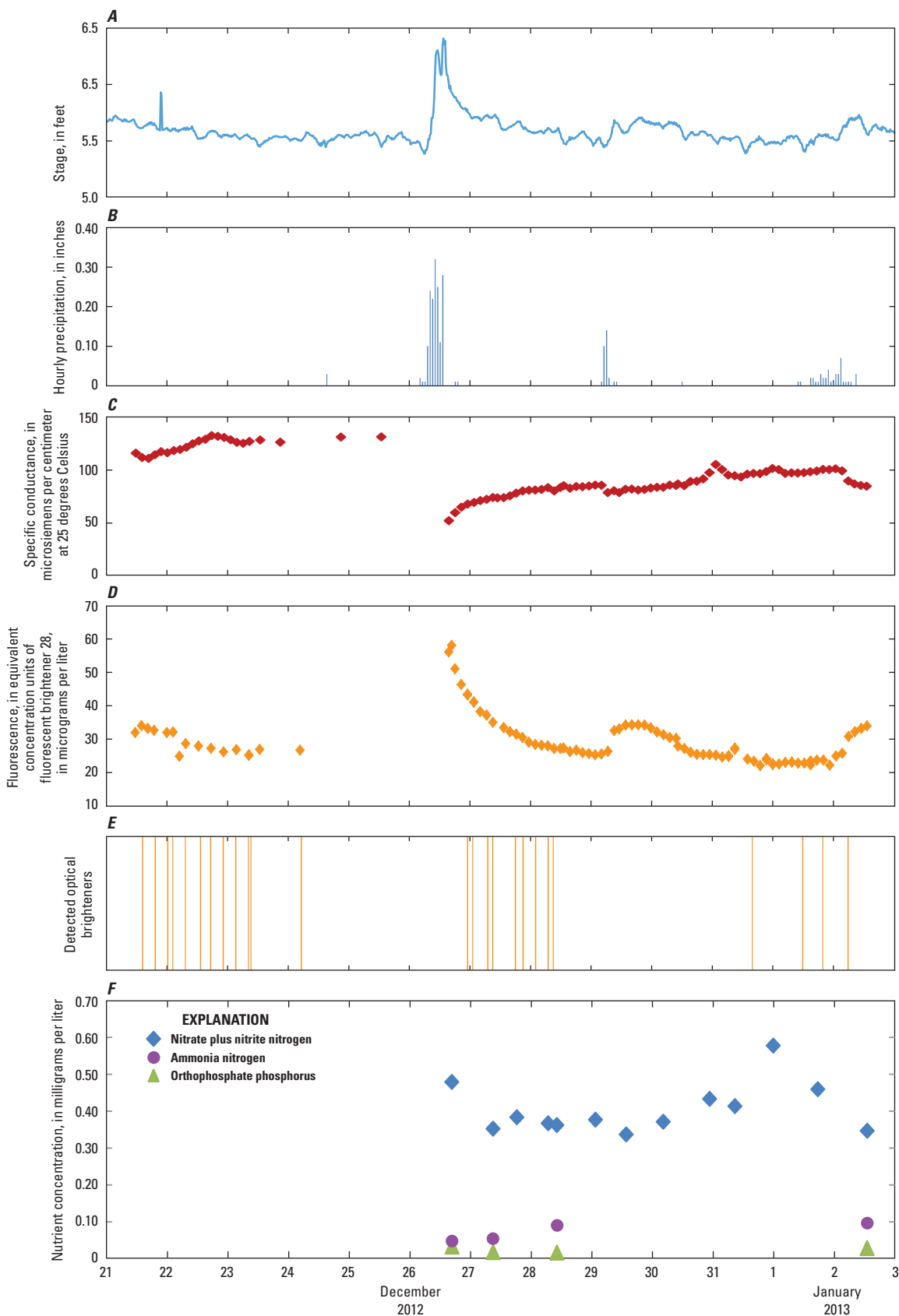




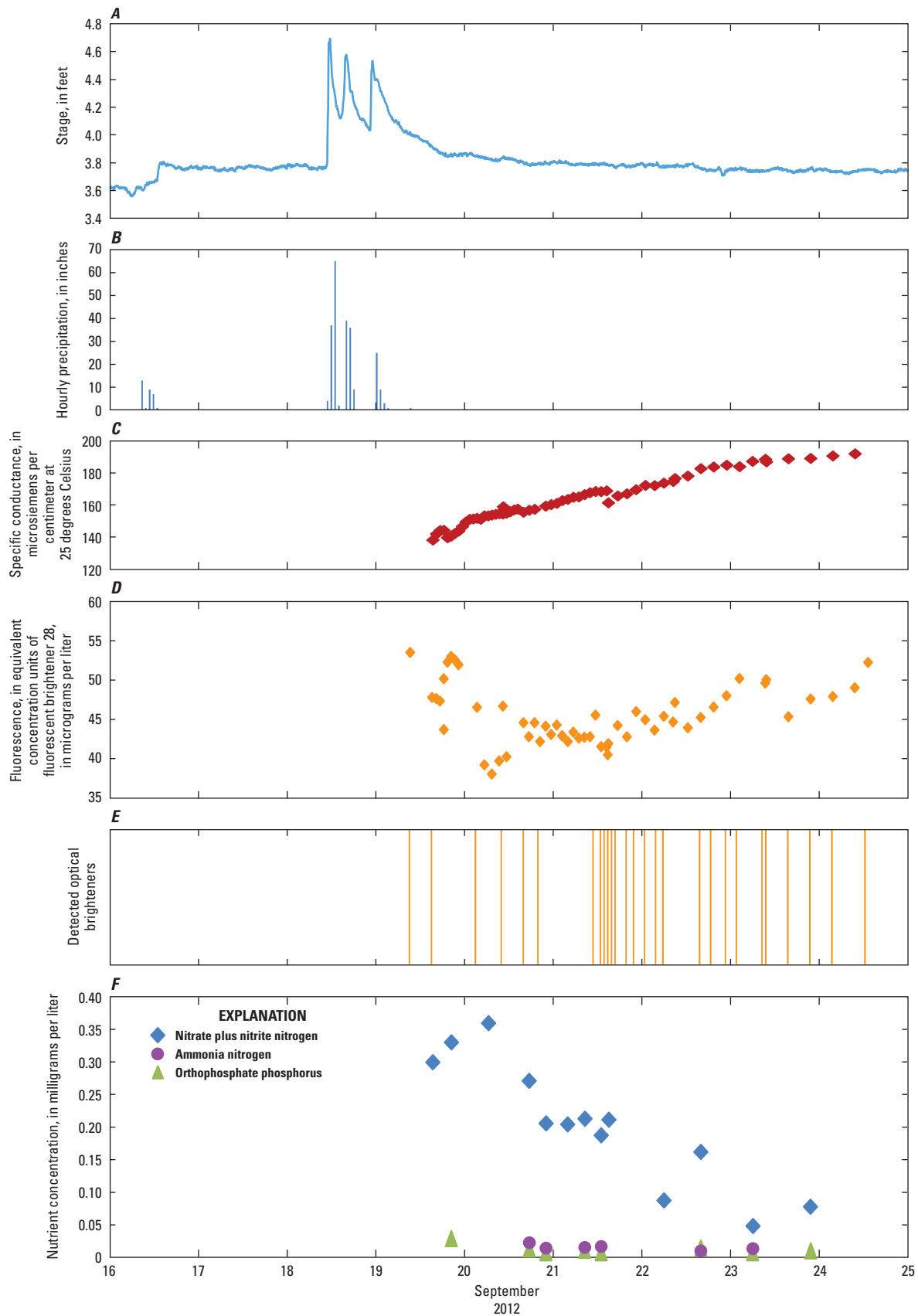
**Figure 26.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Sevenmile Creek tributary at Inverness Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.



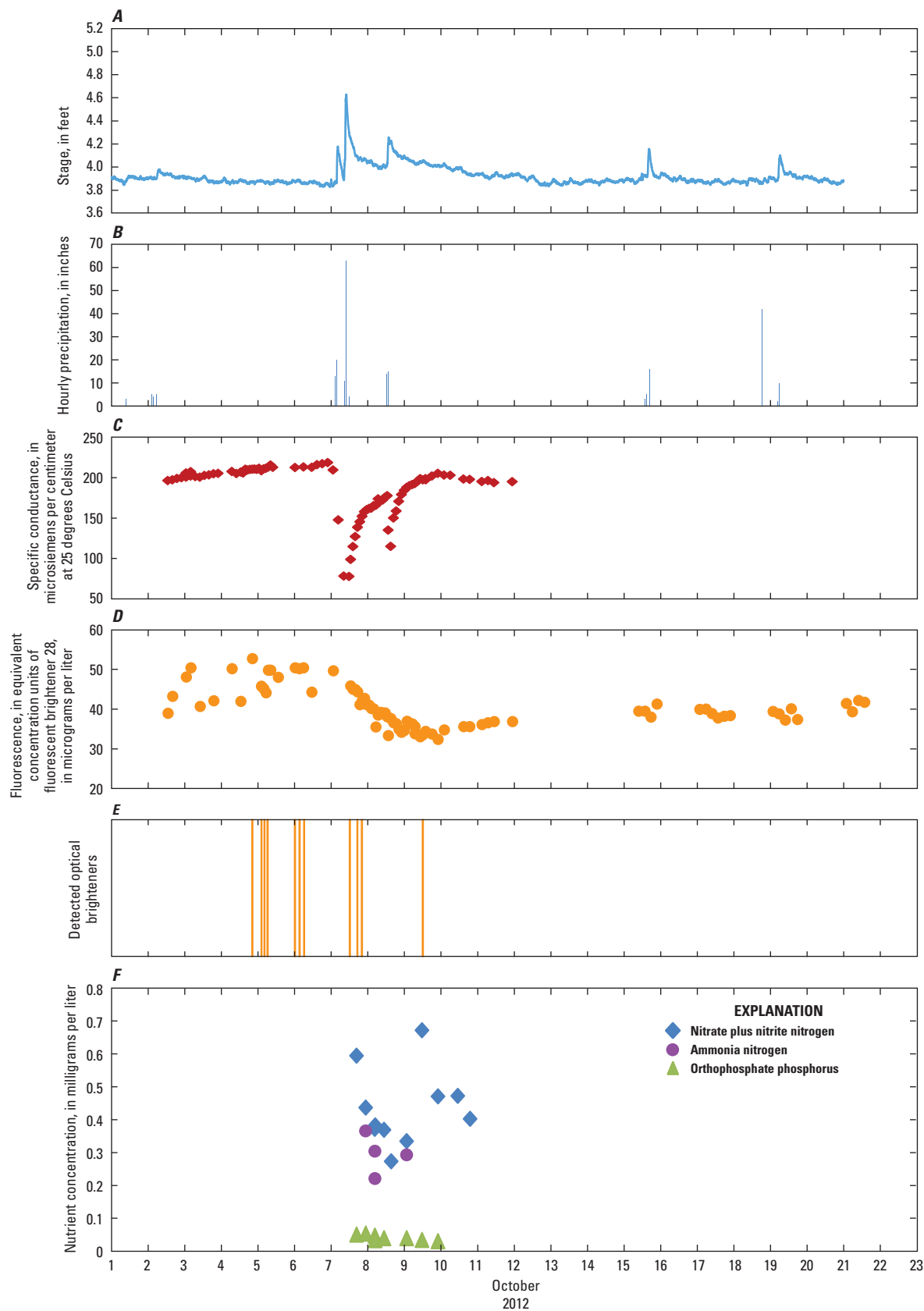
**Figure 27.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, October 1–21, 2012.



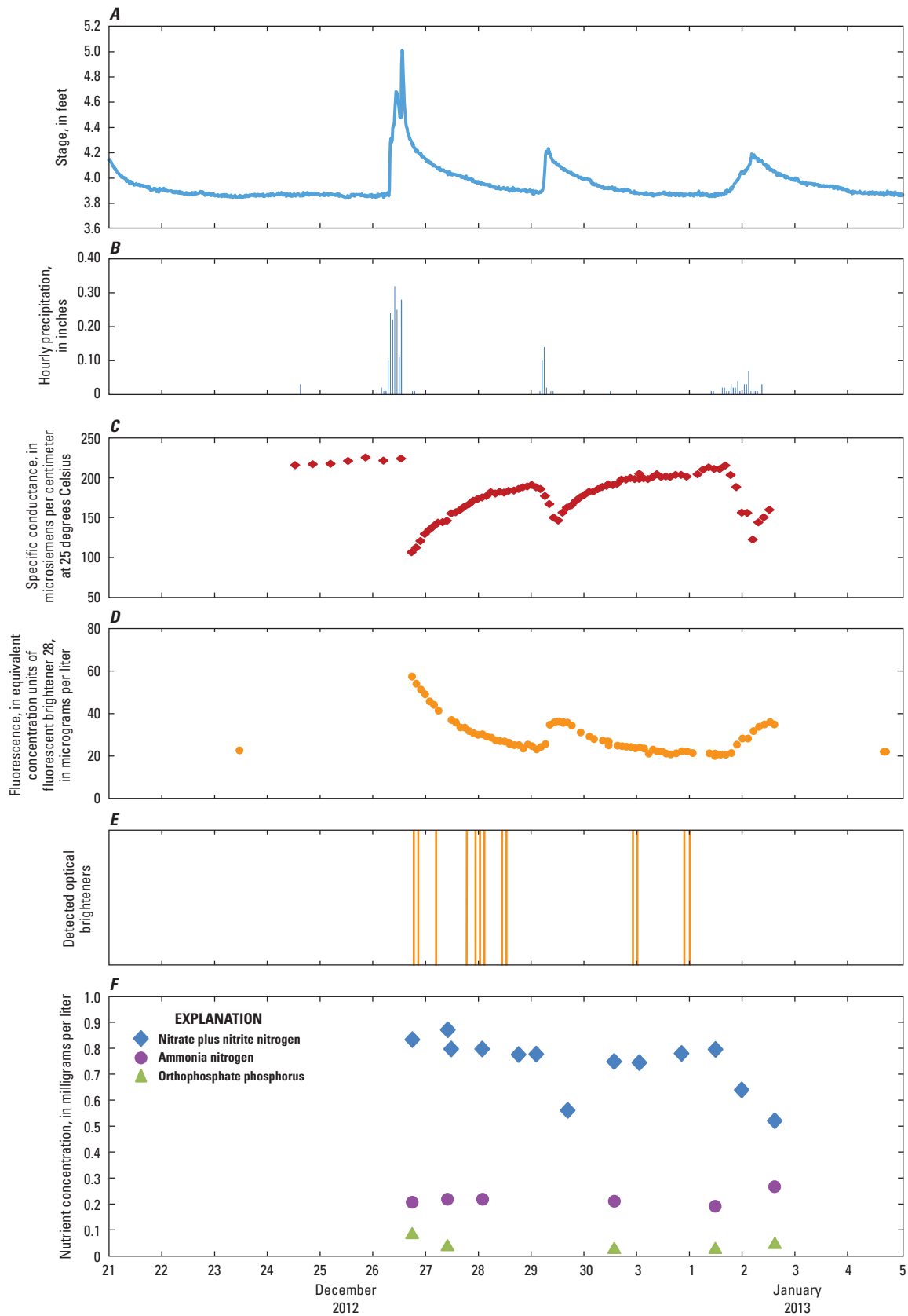
**Figure 28.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Crooked Creek tributary at Greenbay Drive near Durham, North Carolina, December 21, 2012, to January 3, 2013.



**Figure 29.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, September 16–25, 2012.



**Figure 30.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, October 1–23, 2012.



**Figure 31.** (A) Stage, (B) precipitation, (C) specific conductance, (D) fluorescence, (E) optical brighteners, and (F) nutrient concentrations at Cabin Branch tributary at Paragon Circle near Durham, North Carolina, December 21, 2012, to January 5, 2013.



## Summary and Conclusions

Within the river reach study for the upper Neuse River Basin from 2004 to 2013, with the caveat that the downstream site was several miles away, wastewater plant effluent appeared to have raised the concentrations of dissolved sodium, sulfate, and nitrate plus nitrite in the downstream reach compared to those concentrations in the upstream reach. Nitrate, the dominant form of nitrogen in the effluent, accounted for a larger percentage of total nitrogen downstream compared to upstream. Although dissolved phosphorus concentrations in the effluent were two orders of magnitude higher than in the stream, concentrations were not elevated downstream. Phosphorus concentrations in the stream were uniformly low everywhere, consistent with rapid uptake and short residence times consistent with phosphorus limitation of primary production in this section of the river.

Wastewater indicators in samples from streams in small catchments served primarily by centralized or onsite wastewater treatment systems generally showed little differences relative to type of wastewater treatment. Based on water-quality data collected during this study, the stream (Site 6) that appears to have been most affected by wastewater is in an area served by centralized wastewater treatment. Because of the few onsite systems in that catchment, it is assumed that the likely source of the wastewater compounds is a sewer line leak. Optical brightener and bacteria data suggest a general location of the leak. Because no clear differences were discerned between water quality data from the remaining sites, it appears that onsite wastewater treatment systems do not have major effects on the quality of the study streams.

Concentrations of nutrients in samples collected during baseflow conditions were generally lower than those collected under stormflow recession conditions, which suggests that shallow groundwater is an important source of nutrients in these streams. Nutrient concentrations were generally larger during winter months, which indicates that biological activity is a factor in controlling nutrient concentrations in streams.

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