

Prepared in cooperation with the North Carolina Department of Environment and Natural Resources, Division of Water Quality, Aquifer Protection Section

Reconnaissance of Organic Wastewater Compounds at a Concentrated Swine Feeding Operation in the North Carolina Coastal Plain, 2008



Open-File Report 2009–1128

Cover photograph. Looking upstream on Sandy Run tributary west of confluence with Plum Tree Branch, Greene County, North Carolina.

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By Stephen L. Harden

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U.S. Geological Survey**

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Conversion Factors

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 ²)	259.0	hectare (ha)
square mile (mi ²)	2.590	square kilometer (km ²)

Vertical coordinate information is referenced to North American Vertical Datum of 1988 (NAVD 88).

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

Altitude, as used in this report, refers to distance above the vertical datum.

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

Abbreviations and Acronyms

CAFO	Concentrated animal feeding operation
DEET	N,N-diethyl- <i>meta</i> -toluamide
DO	Dissolved oxygen
DWQ	NCDENR Division of Water Quality
GC/MS/MS	Gas chromatography with tandem mass spectrometry
HIA	Household, industrial, and agricultural-use compounds
HPLC/MS/MS	High-performance liquid chromatography with tandem mass spectrometry
IDS	Isotope-dilution standard
LC/MS	Liquid chromatography with mass spectrometry
NCDENR	North Carolina Department of Environment and Natural Resources
NWQL	USGS National Water Quality Laboratory
OWC	Organic wastewater compound
PAH	Polycyclic aromatic hydrocarbon
RL	Reporting level
SPE	Solid-phase extraction
USGS	U.S. Geological Survey
WWTP	Wastewater-treatment plant

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By Stephen L. Harden

Abstract

Water-quality and hydrologic data were collected during 2008 to examine the occurrence of organic wastewater compounds at a concentrated swine feeding operation located in the North Carolina Coastal Plain. Continuous groundwater level and stream-stage data were collected at one monitoring well and one stream site, respectively, throughout 2008. One round of environmental and quality-control samples was collected in September 2008 following a period of below-normal precipitation and when swine waste was not being applied to the spray fields. Samples were collected at one lagoon site, seven shallow groundwater sites, and one surface-water site for analysis of 111 organic wastewater compounds, including household, industrial, and agricultural-use compounds, sterols, pharmaceutical compounds, hormones, and antibiotics.

Analytical data for environmental samples collected during the study provide preliminary information on the occurrence of organic wastewater compounds in the lagoon-waste source material, groundwater beneath fields that receive spray applications of the lagoon wastes, and surface water in the tributary adjacent to the site. Overall, 28 organic wastewater compounds were detected in the collected samples, including 11 household, industrial, and agricultural-use compounds; 3 sterols; 2 pharmaceutical compounds; 5 hormones; and 7 antibiotics. The lagoon sample had the greatest number (20) and highest concentrations of compounds compared to groundwater and surface-water samples. The antibiotic lincomycin had the maximum detected concentration (393 micrograms per liter) in the lagoon sample. Of the 11 compounds identified in the groundwater and surface-water samples, all with reported concentrations less than 1 microgram per liter, only lincomycin identified in groundwater at 1 well and 3-methyl-1H-indole and indole identified in surface water at 1 site also were identified in the lagoon waste material.

Introduction

During the past 10 years, the North Carolina Department of Environment and Natural Resources (NCDENR) and U.S. Geological Survey (USGS) have conducted studies to characterize the quality of groundwater and surface water in the Coastal Plain (Harden and Spruill, 2004; Spruill and others, 2005; Tesoriero and others, 2005; Harden and Spruill, 2008), and in the Piedmont and Mountain regions (Chapman and others, 2005; McSwain and others, 2008; Pippin and others, 2008) of North Carolina. Characterizing water-quality conditions in different hydrogeologic settings includes understanding the potential effects of local land use. Some of the sites with different land-use conditions previously investigated by NCDENR and USGS include agricultural areas with concentrated animal feeding operations (CAFOs) in which lagoon and spray-field treatment systems are used (Harden and Spruill, 2004; Spruill and others, 2005; Harden and Spruill, 2008) and a wastewater-treatment plant (WWTP) where past operations have included applications of municipal biosolids to surrounding fields (McSwain and others, 2008). A common objective of these studies was to document the distribution and amounts of nutrients in groundwater and/or surface water originating from fields with different sources of applied nutrients, such as conventional fertilizers, swine lagoon effluent, or municipal biosolids.

Results of additional studies have shown that wastewater and/or biosolids from livestock operations and municipal WWTPs can be contaminant sources of industrial chemicals, pesticides, flame retardants, pharmaceuticals and personal-care products, hormones, and antibiotics in both groundwater (MacKie and others, 2006; Arnon and others, 2008; Barnes and others, 2008) and surface water (Kolpin and others, 2002; Managaki and others, 2007; Focazio and others, 2008; Topp and others, 2008). There is growing interest in understanding

the occurrence, fate, and transport of organic wastewater compounds (OWCs) in the environment because of concerns that these compounds may influence human and ecological health through toxicity, endocrine disruption, and promotion of antimicrobial resistance (Daughton and Ternes, 1999; Kolpin and others, 2002; MacKie and others, 2006; Arnon and others, 2008; Barnes and others, 2008). In North Carolina and throughout the Nation, increased knowledge is needed on how different waste and fertilizer management practices affect the occurrence of OWCs in the environment. Part of the mission of the NCDENR Division of Water Quality (DWQ) is to preserve and protect the groundwater and surface-water resources of North Carolina by characterizing groundwater flow in a variety of hydrogeologic settings and documenting the occurrence and mobility of both naturally occurring and land-use related contaminants. In 2008, NCDENR and USGS conducted a pilot study of OWCs at a swine CAFO in North Carolina, which is one of the top swine-producing states in the Nation (Center on Globalization, Governance & Competitiveness, 2007).

Purpose and Scope

This report summarizes a single set of water-quality and hydrologic data collected during 2008 at one swine CAFO located in the Neuse River basin, North Carolina (fig. 1). Water-quality data were collected during one sampling event in September 2008 at one lagoon site, seven groundwater sites, and one surface-water site. Continuous groundwater level and stream-stage data were collected at one monitoring well (L2) and one stream site (S7), respectively, throughout 2008. Data presented in this report are intended to assist the DWQ in developing a broader water-quality sampling program to understand the presence, transport, and fate of OWCs in areas affected by applications of animal waste and municipal biosolids.

Description of the Study Area

The study was conducted at a farm located in Greene County in the North Carolina Coastal Plain Physiographic Province and south of the confluence of Sandy Run and Middle Swamp (figs. 1, 2). The hydrogeologic framework and water-quality conditions at the farm site have been well characterized during more than 10 years of study (Harden and Spruill, 2004, 2008; Spruill and others, 2005; Tesoriero and others, 2005; Harden, 2008). A 5,000-head swine CAFO in which a lagoon and spray-field treatment system are used has been in operation at this location since 1995. Most of the site is drained by a first-order stream, known locally as Plum Tree Branch, which is a tributary to Sandy Run and drains 0.59 square mile (mi²). Land use in the area is primarily agricultural, with typical row crops being corn, wheat, and soybeans. Agricultural fields at the site consist of somewhat poorly drained to well-drained soils.

Methods

The sampling network for the study (fig. 2; table 1) included one waste-lagoon site (LG1), seven well sites (L2M, L2D, L15M, L15D, L70D, L71D, and L72), and one surface-water site (S7). Submersible pressure transducers were used to record stream stage at site S7 and groundwater levels at site L2, adjacent to groundwater sampling sites L2M and L2D (fig. 2). Data were recorded at these sites at 15-minute intervals throughout 2008. Wells at the CAFO study site are located in and around the spray-application fields. The wells were completed in the surficial aquifer at depths less than 25 feet (table 1). Shallow groundwater at the site flows to the northwest and north toward the discharge areas at Plum Tree Branch and Sandy Run, respectively (Tesoriero and others, 2005; Harden and Spruill, 2008). Wells L2, L2M, and L2D are collocated along the southern boundary of the spray fields (fig. 2) in an upgradient area of the groundwater-flow path. Well pair L15M and L15D and well L71D are located along the northern boundary of the spray fields in a downgradient area of the flow path. Well L70D is located in the middle of the spray fields, and well L72 is near the western boundary of the spray fields in the riparian buffer next to Plum Tree Branch (fig. 2). Surface-water site S7, located upstream of well L72, receives drainage from the spray fields, non-spray fields, and areas upstream from the study site.

Water-quality samples were collected during September 22–24, 2008, for analysis of OWCs, including household, industrial, and agricultural-use (HIA) compounds, sterols, pharmaceutical compounds, hormones, and antibiotics. Specific conductance, pH, water temperature, and dissolved-oxygen (DO) concentrations in samples were determined in the field at the time of sampling. Established, documented protocols for collecting and processing water samples for chemical analyses were followed (U.S. Geological Survey, variously dated). Sampling personnel also adhered to stringent quality-control requirements during sample collection, which included avoiding contact with insect repellents, antibacterial cleaners, caffeine, tobacco, and targeted pharmaceuticals (U.S. Geological Survey, variously dated). Samples from the wells and the lagoon were collected by using Teflon® tubing and a peristaltic pump. At stream site S7, a grab sample was collected directly into a sample bottle. Quality-control samples, including a source-solution blank, field-equipment blank, and a replicate sample, were collected during the sampling event to ensure that sampling procedures did not introduce contamination during the collection of the environmental samples. All samples were collected into pre-cleaned amber glass bottles or Teflon® bottles (depending on analytical requirements), chilled, and shipped by overnight delivery to the USGS analyzing laboratories.

Three laboratory analytical methods were used in this study to test for as many as 111 compounds in each sample, including 55 HIAs, 4 sterols, 4 pharmaceutical compounds,

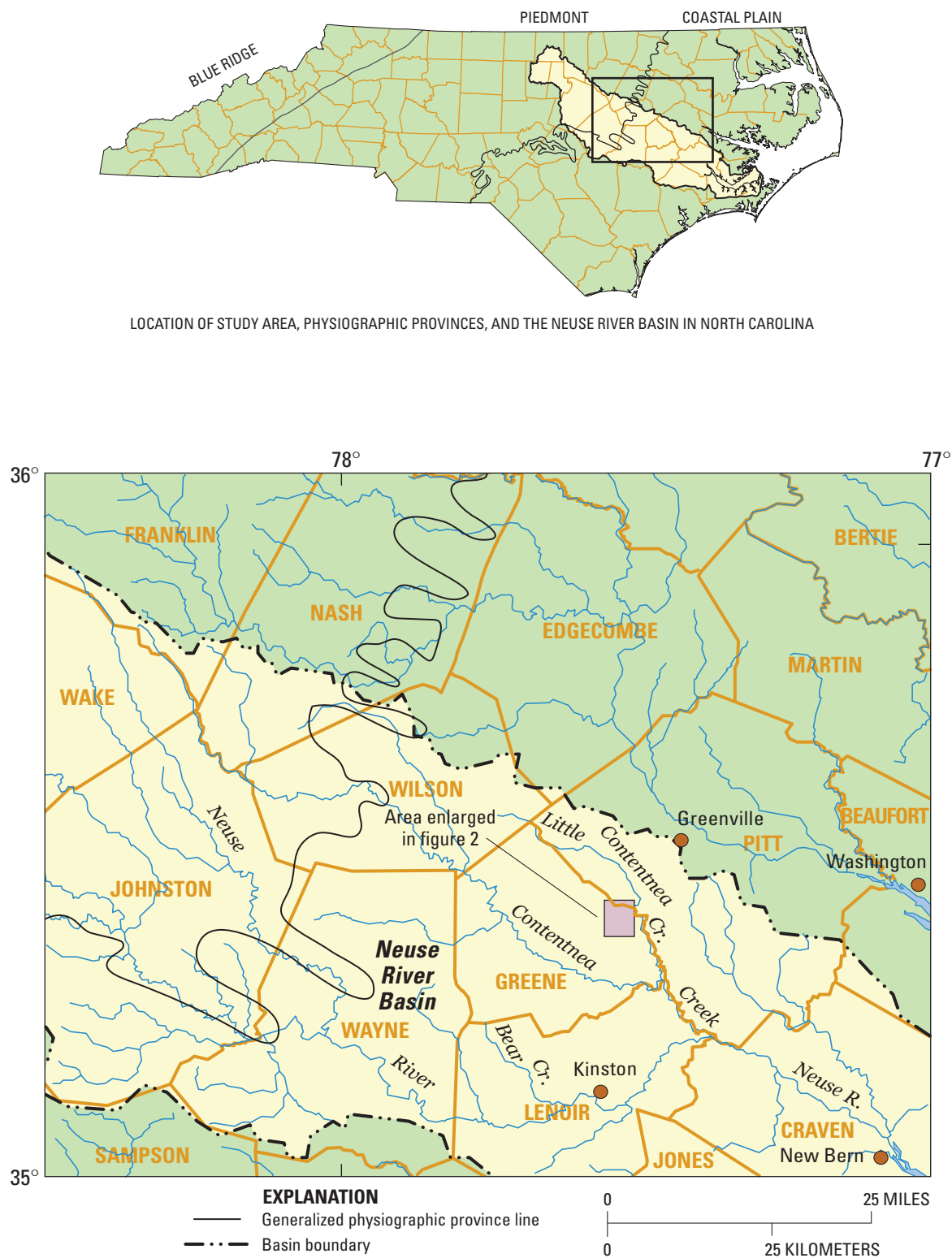


Figure 1. Location of study area in the Neuse River basin of North Carolina.

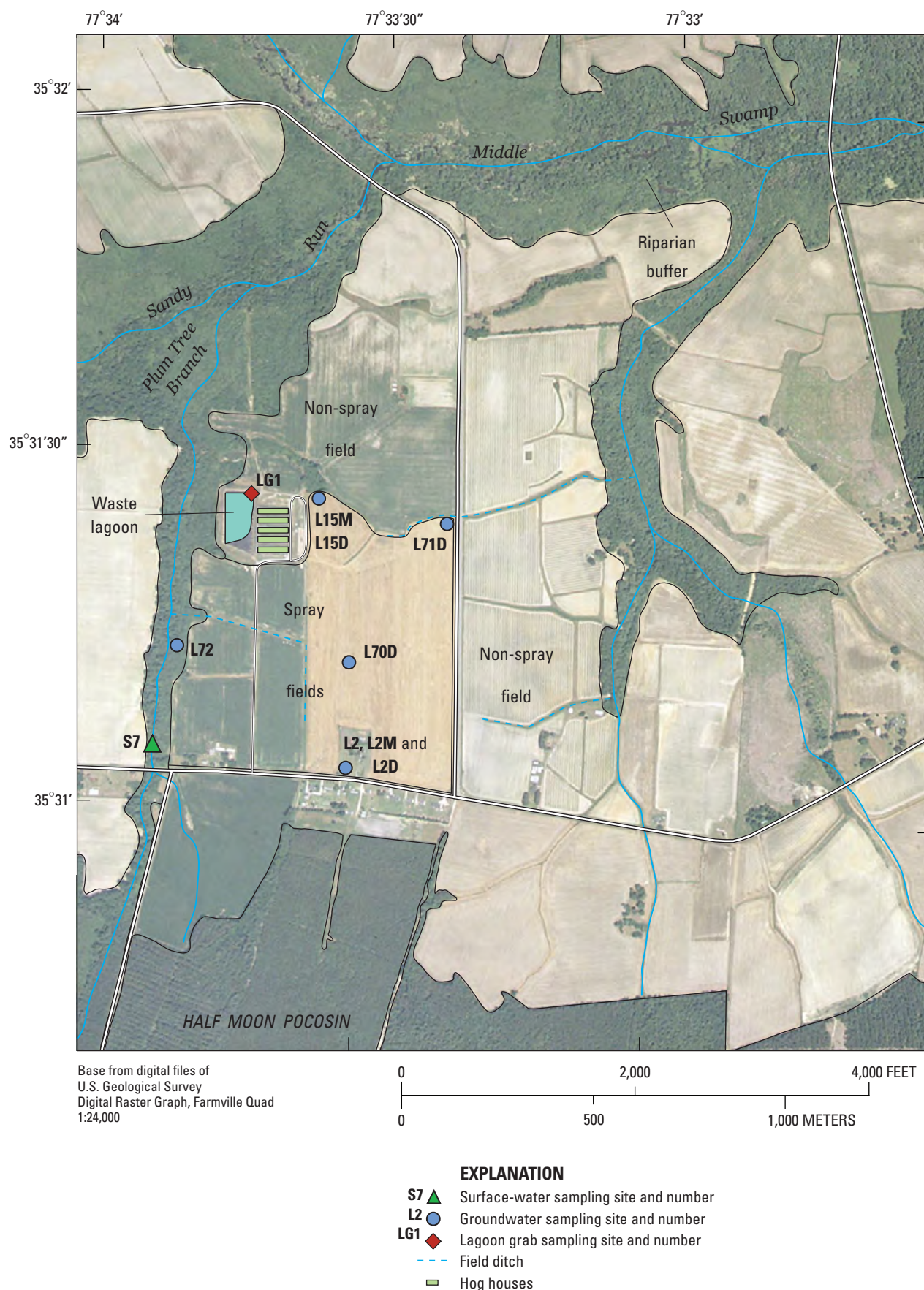


Figure 2. Groundwater, surface-water, and lagoon sampling locations at the concentrated animal feeding operation study site in Greene County, North Carolina.

Table 1. Water-quality and hydrologic data network at the concentrated animal feeding operation study site in Greene County, North Carolina, 2008.

[USGS, U.S. Geological Survey; ft bls, feet below land surface; —, not applicable]

USGS station name	Map number (see fig. 2)	Well depth (ft bls)	USGS site number	Sample/data type	Sample date/time
SR5-LG1	LG1	—	353126077334201	Lagoon grab sample	09/24/08 1240
GR-082 L2 N26q2 nr Lizzie, NC (surficial)	L2	16	353103077333401	Groundwater levels	2008
GR-088 LWQ2M	L2M	11.4	353103077333407	Groundwater sample	09/23/08 1720
GR-088 L2D	L2D	20	353103077333402	Groundwater sample	09/24/08 0855
GR-167 LWQ15M	L15M	11.6	353127077333705	Groundwater sample	09/22/08 1415
GR-109 L15D	L15D	23	353127077333704	Source solution blank	09/22/08 1000
				Groundwater sample	09/22/08 1650
GR-169 LWQ70D	L70D	14.6	353114077333102	Field equipment blank	09/23/08 1325
				Groundwater sample	09/23/08 1500
				Replicate sample	09/23/08 1505
GR-171 LWQ71D	L71D	14.2	3531260773332102	Groundwater sample	09/24/08 1035
GR-203 LWQ72	L72	10	3531140773334905	Groundwater sample	09/24/08 1310
Unnamed trib to Sandy Run at SR1335 near Lizzie, NC	S7	—	0209173150	Surface-water sample	09/23/08 1150
				Stream stage	2008

17 hormones, and 31 antibiotics (table 2). All analyses were performed on filtered samples and, therefore, the results represent compounds present in the dissolved phase. Analytical method 1 is a USGS-approved production method (Zaugg and others, 2006) that was used to analyze for 60 organic compounds spanning several classes of chemicals, including 54 HIAs (such as fire retardants, non-ionic surfactants, plasticizers, solvents, disinfectants, polycyclic aromatic hydrocarbons (PAHs), and high-use domestic pesticides), 4 sterols, and 2 pharmaceutical compounds (caffeine and cotinine). Analytical method 2 is a research method that was used to analyze for 17 hormones, 1 HIA, and 2 sterols. This method employs isotope-dilution quantitation to improve quantitative accuracy by accounting for sample-specific procedural losses in the determined analyte concentrations (William Foreman, U.S. Geological Survey, written commun., 2009). Deuterium-labeled isotope-dilution standards (IDSs) were added to all environmental and quality-assurance samples prior to extraction. The method analytes and IDSs in samples were extracted by using solid-phase extraction (SPE). Analysis was performed by using capillary-column gas chromatography with tandem mass spectrometry (GC/MS/MS). Analyte concentrations were quantified relative to specific IDS concentrations in the sample, which directly compensates for procedural losses (or recovery) in the determined and reported analyte concentrations. The two sterols analyzed by using method 2 (3-*beta*-coprostanol and cholesterol) also were analyzed by using method 1 (table 2). Analyses for methods 1 and 2 were conducted at the USGS National Water Quality Laboratory (NWQL) in Lakewood, Colorado.

Analytical method 3 is a research method that was used to analyze for 2 pharmaceutical compounds (carbamazepine and ibuprofen) and 31 human and veterinary antibiotics and selected degradation products. Target analytes were extracted by using online SPE and analyzed by high-performance liquid chromatography with tandem mass spectrometry (HPLC/MS/MS) using positive and negative electrospray-ionization with multiple reaction monitoring modified from the online SPE LC/MS method of Meyer and others (2007). Target compounds were identified by using relative retention time and the ratio of the quantitation-to-confirming daughter-ion ratio and were quantified by using a multilevel extracted standard curve. Method 3 was performed by the USGS Organic Geochemistry Research Laboratory in Lawrence, Kansas. The laboratory reporting levels (RLs) for the OWCs analyzed in the groundwater and surface-water samples collected during the study are provided in table 2. Analytical RLs for the lagoon sample generally are 2 to 100 times higher than those listed in table 2 because the concentrated waste material required dilutions prior to laboratory analysis.

Data Results

Upon initiation of this project, scheduling constraints required the water-quality sampling to be conducted in late summer 2008. One round of water-quality sampling was performed during September 22–24, 2008, to test for the presence of OWCs in groundwater, surface water, and lagoon waste

Table 2. Summary of dissolved organic wastewater compounds analyzed in samples collected from a concentrated swine feeding operation study site, Greene County, North Carolina, 2008.

[Analytical method 1 is an approved U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Cholesterol and 3-*beta*-coprostanol were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration]

Analytical method (see text)	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration detected (µg/L)	Maximum concentration detected (µg/L)	Number of detections	Total number of samples
Household, industrial, and agricultural-use compounds						
1	1,4-dichlorobenzene	0.1	—	—	0	12
1	1-methylnaphthalene	0.1	—	—	0	12
1	2,6-dimethylnaphthalene	0.1	—	—	0	12
1	2-methylnaphthalene	0.1	—	—	0	12
1	3-methyl-1H-indole (skatol)	0.08	E0.014	38	2	12
1	3- <i>tert</i> -butyl-4-hydroxyanisole (BHA)	0.6	—	—	0	12
1	4-cumylphenol	0.1	—	—	0	12
1	4-nonylphenol	1.0	E0.7	E0.7	1	12
1	4-nonylphenol diethoxylate (NP2EO)	5.0	—	—	0	12
1	4-normal-octylphenol	0.16	—	—	0	12
1	4- <i>tert</i> -octylphenol	1.0	—	—	0	12
1	4- <i>tert</i> -octylphenol diethoxylate (OP2EO)	1.0	—	—	0	12
1	4-octylphenol monoethoxylate (OP1EO)	1.0	—	—	0	12
1	5-methyl-1H-benzotriazole	0.08	—	—	0	12
1	9,10-anthraquinone	0.2	—	—	0	12
1	Acetophenone	0.4	—	—	0	12
1	Acetyl-hexamethyl tetrahydronaphthalene (AHTN)	0.5	—	—	0	12
1	Anthracene	0.1	—	—	0	12
1	Benzo[<i>a</i>]pyrene	0.1	—	—	0	12
1	Benzophenone	0.1	E0.03	E0.1	2	12
2	Bisphenol-A	0.100	0.139	0.139	1	12
1	Bromacil	0.4	—	—	0	12
1	Camphor	0.1	E0.1	E0.1	1	12
1	Carbaryl	1.0	—	—	0	12
1	Carbazole	0.1	—	—	0	12
1	Chlorpyrifos	0.1	—	—	0	12
1	Diazinon	0.1	—	—	0	12
1	<i>d</i> -Limonene	0.4	—	—	0	12
1	Fluoranthene	0.1	—	—	0	12
1	Hexahydro-hexamethyl cyclopentabenzopyran (HHCb)	0.5	—	—	0	12
1	Indole	0.1	E0.01	E4.5	2	12
1	Isoborneol	0.1	—	—	0	12
1	Isophorone	0.1	—	—	0	12
1	Isopropylbenzene (cumene)	0.1	—	—	0	12
1	Isoquinoline	0.2	—	—	0	12
1	Menthol	0.2	—	—	0	12
1	Metalaxyl	0.1	—	—	0	12
1	Methyl salicylate	0.1	—	—	0	12
1	Metolachlor	0.1	—	—	0	12
1	Naphthalene	0.1	—	—	0	12
1	N,N-diethyl- <i>meta</i> -toluamide (DEET)	0.1	0.8	1.4	2	12
1	<i>para</i> -Cresol	0.18	—	—	0	12

Table 2. Summary of dissolved organic wastewater compounds analyzed in samples collected from a concentrated swine feeding operation study site, Greene County, North Carolina, 2008.—Continued

[Analytical method 1 is an approved U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Cholesterol and 3-*beta*-coprostanol were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration]

Analytical method (see text)	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration detected (µg/L)	Maximum concentration detected (µg/L)	Number of detections	Total number of samples
Household, industrial, and agricultural-use compounds—Continued						
1	Phenanthrene	0.1	—	—	0	12
1	Phenol	0.2	100	100	1	12
1	Prometon	0.2	—	—	0	12
1	Pyrene	0.1	—	—	0	12
1	Tetrachloroethylene	0.1	—	—	0	12
1	Tris(2-butoxyethyl) phosphate	0.4	—	—	0	12
1	Tris(2-chloroethyl) phosphate (FYROL CEF)	0.1	E5.3	E5.3	1	12
1	Tris(dichloroisopropyl) phosphate (FYROL FR2)	0.1	E0.1	E0.1	1	12
1	Tributyl phosphate	0.2	—	—	0	12
1	Tribromomethane	0.1	—	—	0	12
1	Triclosan	0.2	—	—	0	12
1	Triethyl citrate (ethyl citrate)	0.2	—	—	0	12
1	Triphenyl phosphate	0.1	E0.1	E0.1	1	12
Sterols						
1	3- <i>beta</i> -coprostanol	1.0	E170	E170	1	12
2	3- <i>beta</i> -coprostanol	2	E174	E174	1	12
1	<i>beta</i> -sitosterol	2.0	—	—	0	12
1	<i>beta</i> -stigmastanol	1.0	E78	E78	1	12
1	Cholesterol	1.0	—	—	0	12
2	Cholesterol	2	26	26	1	12
Pharmaceuticals						
1	Caffeine	0.1	E4.2	E4.2	1	12
3	Carbamazepine	0.005	—	—	0	12
1	Cotinine	0.4	—	—	0	12
3	Ibuprofen	0.05	0.477	0.477	1	12
Hormones						
2	11-ketotestosterone	0.002	0.186	0.186	1	12
2	17- <i>alpha</i> -estradiol	0.008	—	—	0	12
2	17- <i>beta</i> -estradiol	0.0008	—	—	0	12
2	17- <i>alpha</i> -ethynylestradiol	0.0008	—	—	0	12
2	Norethindrone	0.0008	—	—	0	12
2	4-androstene-3,17-dione	0.0008	E0.00042	E0.00051	2	12
2	Cis-androsterone	0.0008	E0.00025	0.165	2	12
2	Dihydrotestosterone	0.004	—	—	0	12
2	Epitestosterone	0.004	—	—	0	12
2	Equilenin	0.002	—	—	0	12
2	Equilin	0.004	—	—	0	12
2	Estriol	0.002	0.0877	0.0877	1	12
2	Estrone	0.0008	0.804	0.804	1	12
2	Mestranol	0.0008	—	—	0	12
2	Progesterone	0.008	—	—	0	12
2	Trans-diethylstilbestrol	0.0008	—	—	0	12
2	Testosterone	0.0008	—	—	0	12

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Table 2. Summary of dissolved organic wastewater compounds analyzed in samples collected from a concentrated swine feeding operation study site, Greene County, North Carolina, 2008.—Continued

[Analytical method 1 is an approved U.S. Geological Survey production method; methods 2 and 3 are U.S. Geological Survey research methods. Cholesterol and 3-*beta*-coprostanol were analyzed by more than one method. µg/L, micrograms per liter; —, not detected; E, estimated concentration]

Analytical method (see text)	Organic wastewater compound	Reporting level (µg/L)	Minimum concentration detected (µg/L)	Maximum concentration detected (µg/L)	Number of detections	Total number of samples
Antibiotics (*degradation product)						
3	Azithromycin	0.005	—	—	0	12
3	Chloramphenicol	0.10	—	—	0	12
3	Chlorotetracycline	0.01	—	—	0	12
3	Ciprofloxacin	0.005	—	—	0	12
3	Doxycycline	0.01	—	—	0	12
3	Enrofloxacin	0.005	—	—	0	12
3	Epi-iso-chlorotetracycline*	0.01	36	36	1	12
3	Epi-chlorotetracycline*	0.01	—	—	0	12
3	Epi-oxytetracycline*	0.01	—	—	0	12
3	Epi-tetracycline*	0.01	0.02	0.02	1	12
3	Erythromycin	0.008	—	—	0	12
3	Erythromycin-H2O*	0.008	—	—	0	12
3	Iso-chlorotetracycline*	0.01	52	52	1	12
3	Lincomycin	0.005	0.018	393	2	12
3	Lomefloxacin	0.005	—	—	0	12
3	Norfloxacin	0.005	—	—	0	12
3	Ofloxacin	0.005	—	—	0	12
3	Ormetoprim	0.005	—	—	0	12
3	Oxytetracycline	0.01	55	55	1	12
3	Roxithromycin	0.005	—	—	0	12
3	Sarafloxacin	0.005	—	—	0	12
3	Sulfachloropyridazine	0.005	—	—	0	12
3	Sulfadiazine	0.01	—	—	0	12
3	Sulfadimethoxine	0.005	—	—	0	12
3	Sulfamethazine	0.005	1.15	1.15	1	12
3	Sulfamethoxazole	0.005	—	—	0	12
3	Sulfathiazole	0.05	—	—	0	12
3	Tetracycline	0.010	0.572	0.572	1	12
3	Trimethoprim	0.005	—	—	0	12
3	Tylosin	0.008	—	—	0	12
3	Virginiamycin	0.005	—	—	0	12

at the CAFO study site. Groundwater-level and streamflow data were collected throughout 2008 as part of other work at the study site. The hydrologic conditions during the sampling event and the analytical results of water-quality samples collected at the CAFO are summarized below. Analytical data for environmental samples collected during the study provide preliminary results on the occurrence of OWCs, hormones, and antibiotics in the lagoon-waste source material, the groundwater beneath the fields that receive spray applications of the lagoon wastes, and in the surface water in the tributary adjacent to the site.

Hydrologic Conditions during Sampling

The September 22–24, 2008, sampling was preceded by a period of below-average precipitation, as noted in data recorded from a rain gauge (USGS Station number 353137077332801) at the site. The total monthly precipitation (in inches) for May (2.19), June (1.21), July (2.43), and August (2.44) in 2008 was considerably less than the median monthly totals recorded for May (4.39), June (4.41), July (4.63), and August (5.67) during the 7-year period from 2001 through 2007. The relatively dry conditions from May to August 2008 are reflected in the groundwater hydrograph at site L2 and the stream hydrograph at site S7 (fig. 3). During sampling, the groundwater level at site L2 and stream stage at site S7 were near the annual low for the 2008 period of data collection (fig. 3). Gaps in the stream hydrograph at site S7 from late May through early July indicate missing data because of equipment failure, and data gaps from mid-July to early September indicate dry periods when no streamflow occurred. No recent applications of lagoon-waste material in the spray fields were noted immediately before or during sample collection. Based on the available data, it is not known whether the constituent concentrations observed during September 2008 are representative of those that would be observed during periods of normal amounts of rainfall, higher groundwater levels, higher streamflows, or more recent applications of lagoon waste.

Organic Wastewater Compounds

A total of 9 environmental samples (1 lagoon sample, 7 groundwater samples, and 1 stream sample) and 3 quality-control samples, including a source-solution blank, field-equipment blank, and replicate sample, were collected for analyses of 111 OWCs. The analytes listed in table 2 include the RLs, minimum and maximum observed concentrations, and number of detections for both the regular environmental samples and the quality-control samples.

Of the 111 analytes examined, 28 were detected in samples collected at the study site, including 11 HIAs, 3 sterols, 2 pharmaceutical compounds, 5 hormones, and 7 antibiotics (table 2). Of the 28 analytes detected, 21 were detected only once; 7 analytes each were detected twice (table 2).

Concentrations of the 28 detected analytes, including estimated values, are summarized for each sample in table 3. Concentrations of 3-*beta*-coprostanol, determined by using both methods 1 and 2, are included in table 3. Approximately half of the analyte detections were reported as qualified “E” values, indicating that the compounds were identified (based on stringent laboratory analyte confirmational criteria) in samples at estimated concentrations less than the analytical RLs. In these cases, the analyzing laboratory confirmed the presence of the analytes in the samples but with less confidence (greater uncertainty) in the reported concentrations.

Data for the quality-control samples were examined to determine if any of the analytes detected in the environmental samples potentially were influenced by sampling and/or analytical methodologies. Results of the source-solution blank indicate that the water used to prepare the field-equipment blank contained none of the analytes detected in the environmental samples (table 3). The field-equipment blank was used to determine if the sampling equipment (tubing and bottles) and processing or laboratory handling were potential sources of contaminants identified in the environmental water samples.

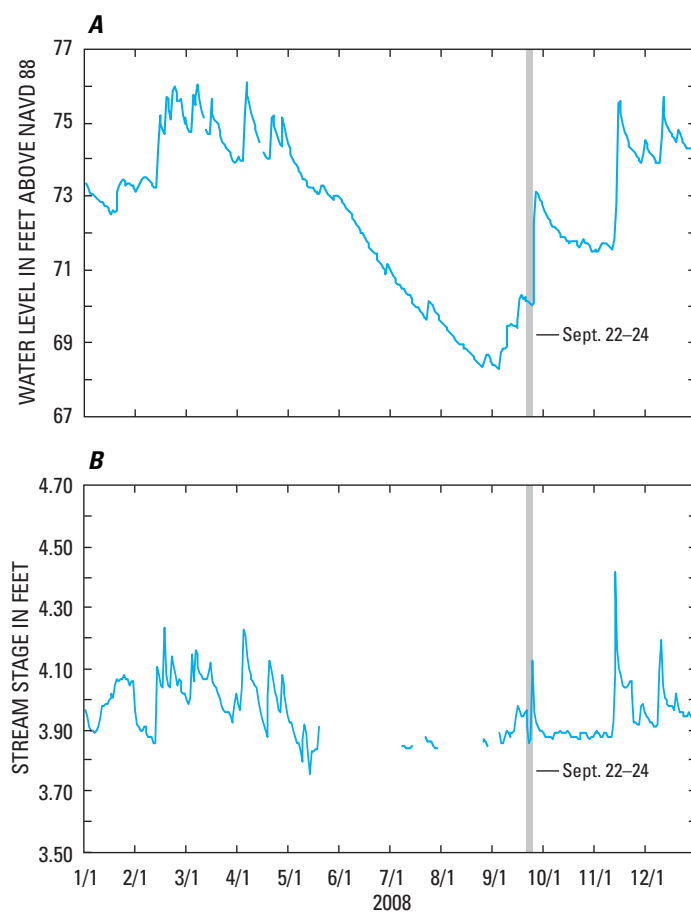


Figure 3. Continuous data for (A) groundwater levels at well L2 and (B) stream stage at site S7 at the study site in Greene County, North Carolina, 2008.

Table 3. Concentrations of organic wastewater compounds detected in environmental and quality-control samples collected from a concentrated swine feeding operation study site, Greene County, North Carolina, 2008.

[HIAs, household, industrial, and agricultural-use compounds; µg/L, micrograms per liter; Numbers in parentheses are U.S. Geological Survey parameter codes; <, less than; E, estimated concentration; italics indicate quality-control samples]

Map number (fig. 2, table 1)	Sample date/time	Sample type	HIAs										Pharamaceuticals		Hormones
			3-Methyl-1H-indole, filtered, µg/L (62058)	4-Nonylphenol (all isomers), filtered, µg/L (62085)	Benzo-phenone, filtered, µg/L (62067)	Bisphenol-A, filtered, µg/L (67304)	Camphor, filtered, µg/L (62070)	Indole, filtered, µg/L (62076)	DEET, filtered, µg/L (62082)	Phenol, filtered, µg/L (34466)	Tris(2-chloroethyl) phosphate, filtered, µg/L (62087)				
L61	09/24/08 1240	Lagoon	38	< 100	< 12	0.139	< 10	E4.5	< 10	100	E5.3				
L2M	09/23/08 1720	Groundwater	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L2D	09/24/08 0855	Groundwater	< 0.08	< 1	E0.1	< 0.100	< 0.1	< 0.1	1.4	< 0.2	< 0.1				
L15M	09/22/08 1415	Groundwater	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L15D	09/22/08 1000	Source solution blank	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L15D	09/22/08 1650	Groundwater	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L70D	09/23/08 1325	Field equipment blank	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L70D	09/23/08 1500	Groundwater	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L70D	09/23/08 1505	Replicate	< 0.08	< 1	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L71D	09/24/08 1035	Groundwater	< 0.08	E0.7	< 0.1	< 0.100	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1				
L72	09/24/08 1310	Groundwater	< 0.08	< 1	E0.03	< 0.100	< 0.1	< 0.1	0.8	< 0.2	< 0.1				
S7	09/23/08 1150	Surface water	E0.014	< 1	< 0.1	< 0.100	E0.1	E0.01	< 0.1	< 0.2	< 0.1				

Map number (fig. 2, table 1)	Sample date/time	Sample type	HIAs			Sterols			Pharamaceuticals		Hormones
			Tris(dichloro-isopropyl) phosphate, filtered, µg/L (62088)	Triphenyl phosphate, filtered, µg/L (62092)	3-beta-Coprostanol, filtered, µg/L (62057 – method 1)	3-beta-Coprostanol, filtered, µg/L (64512 – method 2)	beta-Stig-mastanol, filtered, µg/L (62086)	Cholesterol, filtered, µg/L (64514)	Caffeine, filtered, µg/L (50305)	Ibuprofen, filtered, µg/L (62014)	11-Ketotestosterone, filtered, µg/L (64507)
L61	09/24/08 1240	Lagoon	< 12	< 10	E170	E174	E78	26	E4.2	< 0.05	0.186
L2M	09/23/08 1720	Groundwater	< 0.1	E0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L2D	09/24/08 0855	Groundwater	E0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	0.477	< 0.002
L15M	09/22/08 1415	Groundwater	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L15D	09/22/08 1000	Source solution blank	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L15D	09/22/08 1650	Groundwater	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L70D	09/23/08 1325	Field equipment blank	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L70D	09/23/08 1500	Groundwater	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L70D	09/23/08 1505	Replicate	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L71D	09/24/08 1035	Groundwater	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
L72	09/24/08 1310	Groundwater	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002
S7	09/23/08 1150	Surface water	< 0.1	< 0.1	< 1	< 2	< 1	< 2	< 0.1	< 0.05	< 0.002

Table 3. Concentrations of organic wastewater compounds detected in environmental and quality-control samples collected from a concentrated swine feeding operation study site, Greene County, North Carolina, 2008.—Continued

[HIA, household, industrial, and agricultural-use compounds; µg/L, micrograms per liter; Numbers in parentheses are U.S. Geological Survey parameter codes; <, less than; E, estimated concentration; italics indicate quality-control samples]

Map number (fig. 2, table 1)	Sample date/time	Sample type	Hormones				Antibiotics				
			4-Androstene-3,17-dione, filtered, µg/L (64513)	Cis-androst- erone, filtered, µg/L (64515)	Estriol, filtered, µg/L (64515)	Estrone, filtered, µg/L (64521)	Epi-iso- chlorotet- racycline, filtered, µg/L (64047)	Epi-tetra- cycline, filtered, µg/L (63727)	Iso- chlorotet- racycline, filtered, µg/L (64175)	Linco- mycin, filtered, µg/L (62894)	Oxytetracycline, filtered, µg/L (61759)
LG1	09/24/08 1240	Lagoon	< 0.490	0.165	0.0877	0.804	36	0.02	52	393	55
L2M	09/23/08 1720	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L2D	09/24/08 0855	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L15M	09/22/08 1415	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L15D	09/22/08 1000	Source solution blank	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L15D	09/22/08 1650	Groundwater	E0.00042	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L70D	09/23/08 1325	Field equipment blank	< 0.0008	E0.00025	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L70D	09/23/08 1500	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L70D	09/23/08 1505	Replicate	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
L71D	09/24/08 1035	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	0.018	< 0.01
L72	09/24/08 1310	Groundwater	< 0.0008	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01
S7	09/23/08 1150	Surface water	E0.00051	< 0.0008	< 0.002	< 0.0008	< 0.01	< 0.01	< 0.01	< 0.005	< 0.01

Map number (fig. 2, table 1)	Sample date/time	Sample type	Antibiotics		Total number of analytes detected
			Sulfametha- zine, filtered, µg/L (61762)	Tetracycline, filtered, µg/L (62781)	
LG1	09/24/08 1240	Lagoon	1.15	0.572	20
L2M	09/23/08 1720	Groundwater	< 0.005	< 0.010	1
L2D	09/24/08 0855	Groundwater	< 0.005	< 0.010	4
L15M	09/22/08 1415	Groundwater	< 0.005	< 0.010	0
L15D	09/22/08 1000	Source solution blank	< 0.005	< 0.010	0
L15D	09/22/08 1650	Groundwater	< 0.005	< 0.010	1
L70D	09/23/08 1325	Field equipment blank	< 0.005	< 0.010	1
L70D	09/23/08 1500	Groundwater	< 0.005	< 0.010	0
L70D	09/23/08 1505	Replicate	< 0.005	< 0.010	0
L71D	09/24/08 1035	Groundwater	< 0.005	< 0.010	2
L72	09/24/08 1310	Groundwater	< 0.005	< 0.010	2
S7	09/23/08 1150	Surface water	< 0.005	< 0.010	4

The occurrence of cis-androsterone in the equipment blank at an estimated concentration (0.00025 microgram per liter ($\mu\text{g/L}$)) less than the RL of 0.0008 $\mu\text{g/L}$ (table 3) was not considered to be a source of contamination to the environmental samples. The lagoon sample was the only environmental sample with a detection of cis-androsterone, and the reported concentration of 0.165 $\mu\text{g/L}$ was approximately three orders of magnitude higher than the estimated result for the equipment blank. The replicate sample, obtained from monitoring well site L70D (fig. 2), was used to examine potential variability introduced during sample collection, processing, and laboratory handling. No analytes were detected above laboratory RLs for either the groundwater sample or the replicate sample collected at site L70D (table 3), which indicates that the samples were collected, processed, and analyzed under consistent conditions. Based on the results of the quality-control samples, no quality-assurance issues were apparent during the collection and processing of the environmental samples.

Lagoon Sample

The lagoon sample (site LG1) contained the highest number and concentrations of OWCs among the environmental samples (fig. 2; table 3). The 20 analytes identified in the lagoon sample represent a range of general uses and sources. The 5 detected HIA compounds include 2 fragrances (3-methyl-1H-indole and indole), 1 plasticizer (bisphenol-A), 1 disinfectant (phenol), and 1 fire retardant (tris(2-chloroethyl) phosphate). Three sterols (3-*beta*-coprostanol, *beta*-stigmastanol, and cholesterol) derived from animals or plants were detected in the lagoon sample. Caffeine, a nonprescription stimulant, was the only detected pharmaceutical compound. Four hormones were identified in the lagoon sample that were indicative of either male characteristics (11-ketotestosterone and cis-androsterone) or female characteristics (estriol and estrone). Seven common veterinary antibiotics or their degradation products were detected in the lagoon sample, including epi-chlorotetracycline, epi-tetracycline, iso-chlorotetracycline, lincomycin, oxytetracycline, sulfamethazine, and tetracycline (table 3).

Groundwater and Surface-Water Samples

Of the 20 analytes identified in the lagoon sample, only 3 of the same analytes also were identified in the groundwater or surface-water samples. The antibiotic lincomycin was detected at a concentration of 0.018 $\mu\text{g/L}$ in water from well L71D (fig. 2; table 3). The concentration of lincomycin in the

lagoon sample was 393 $\mu\text{g/L}$, which represents the highest concentration of any OWC detected during this study. At surface-water site S7, the HIA compounds 3-methyl-1H-indole (estimated concentration of 0.014 $\mu\text{g/L}$) and indole (estimated concentration of 0.01 $\mu\text{g/L}$) were detected below the analytical RLs (table 3). The lagoon sample had a concentration of 38 $\mu\text{g/L}$ for 3-methyl-1H-indole and an estimated concentration of 4.5 $\mu\text{g/L}$ for indole. The compounds 3-methyl-1H-indole and indole commonly are used as fragrances or fragrance fixatives; however, these compounds also occur naturally in feces. None of the sterols or hormones detected in the lagoon sample was detected in the groundwater or surface-water samples (table 3).

Eight analytes that were detected at low levels in the groundwater and surface-water samples were not identified in the waste-lagoon sample (table 3), which possibly reflects the higher analytical RLs associated with the lagoon sample or sources other than swine waste. The following groundwater sites had estimated concentrations of HIA compounds at or below the RLs, including well L2M (0.1 $\mu\text{g/L}$ for triphenyl phosphate), well L2D (0.1 $\mu\text{g/L}$ for benzophenone and 0.1 $\mu\text{g/L}$ for tris(dichloroisopropyl) phosphate), well L71D (0.7 $\mu\text{g/L}$ for 4-nonylphenol), and well L72 (0.03 $\mu\text{g/L}$ for benzophenone). At surface-water site S7, the HIA compound camphor had an estimated concentration of 0.1 $\mu\text{g/L}$. The HIA compound DEET (N,N-diethyl-*meta*-toluamide) had the highest detected concentrations above the analytical RL in water samples from wells L2D (1.4 $\mu\text{g/L}$) and L72 (0.8 $\mu\text{g/L}$). DEET was reported as less than the RL of 10 $\mu\text{g/L}$ in the lagoon sample (table 3).

Analysis of the pharmaceutical compounds indicated that ibuprofen, a nonprescription anti-inflammatory drug, was detected in water from well L2D at a concentration of 0.477 $\mu\text{g/L}$. Ibuprofen was reported as less than the RL of 0.05 $\mu\text{g/L}$ in the lagoon sample and at all other sites. The occurrence of both ibuprofen and DEET in the sample from well L2D is interesting in that this well is located at the hydraulically upgradient edge of the spray fields near local residences. It is unclear whether the presence of these compounds in water from well L2D reflects local agricultural and/or domestic sources.

The occurrence of hormones in groundwater and surface-water samples was limited to 4-androstene-3,17-dione, which was detected at estimated concentrations of 0.00042 $\mu\text{g/L}$ in groundwater from well L15D and 0.00051 $\mu\text{g/L}$ at surface-water site S7. The hormone 4-androstene-3,17-dione was reported as less than the RL of 0.490 $\mu\text{g/L}$ for the lagoon sample.

Summary

The analytical results for the OWCs, hormones, and antibiotics obtained in this study are based on one round of sampling that occurred following a period of below-normal precipitation and when swine waste was not being applied to the fields. Future studies designed to examine the occurrence of OWCs, hormones, and antibiotics in groundwater and surface water associated with animal-waste application fields could enhance the body of information by including sampling strategies that represent seasonal differences in rainfall, groundwater levels, streamflow, and waste applications.

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