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Scientific Investigations Report 2020–5015

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

Cover. U.S. Geological Survey employee performing a topographic survey at study site. Photograph by Robert Darner, U.S. Geological Survey.

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Contents

Figures

Tables

Conversion Factors

U.S. customary units to International System of Units

Multiply	By	To obtain
	Length	
inch (in.)	2.54	centimeter (cm)
foot (ft)	0.3048	meter (m)
	Volume	
gallon (gal)	3.785	liter (L)
	Mass	
pound, avoirdupois (lb)	0.4536	kilogram (kg)
	Area	
acre	0.004047	square kilometer (km^2)

International System of Units to U.S. customary units

Multiply	By	To obtain
	Length	
meter (m)	3.281	foot (ft)
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
	Volume	
milliliter (mL)	0.033814	ounce, fluid (fl. oz)
liter (L)	33.81402	ounce, fluid (fl. oz)
	Mass	
milligram (mg)	0.00003527	ounce, avoirdupois (oz)
gram (g)	0.03527	ounce, avoirdupois (oz)

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

Abbreviations

<	less than
>	greater than
≤	less than or equal to
BGSU	Bowling Green State University
GCLAS	Graphical Constituent Loading and Analysis System
HAB	harmful algal bloom
mgN/L	milligrams nitrogen per liter
mgP/L	milligrams phosphorus per liter
n	number
NWARS	Northwest Agricultural Research Station
NWIS	National Water Information System
NWQL	National Water Quality Laboratory
OARDC	Ohio State University Agricultural Research and Development Center
PVC	polyvinyl chloride
RPD	relative percent difference
USGS	U.S. Geological Survey

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Abstract

Manure and wastewater from large livestock operations have the potential to negatively affect surface water and groundwater, including the eutrophication of surface waters and harmful algal blooms. In the Western Lake Erie Basin, where there is a high density of animal agriculture, harmful algal blooms have been attributed, in part, to phosphorus loading from dairy manure and fertilizer applications. Liquid lagoon manure produced by dairy operations typically has low nutrient concentrations and high-water content, so transportation costs are high relative to the value of the nutrients when applied to fields. Treatment systems are needed to transform manure into a dewatered product that is more economical to transport greater distances and that slows and (or) reduces the release of nutrients in soil, allowing nutrients to remain available for crop growth.

This study was designed to pilot test a treatment solution in the Western Lake Erie Basin. The U.S. Geological Survey and Bowling Green State University field tested a dewatering treatment process (coagulant/polymer mixture) for dairy manure at pilot-scale test plots at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station. Automatic samplers were used to collect samples during 13 baseline and 9 post-manure application rainfall events that resulted in substantial surface runoff and (or) tile flow from October 2015 through early November 2017. Results are reported for three test plots that received liquid lagoon manure (raw manure) and three test plots that received polymer-treated manure (treated manure).

Nutrient concentrations and flow volumes in surface runoff and tile flow were determined in baseline and post-manure application rainfall events. Nutrient concentration ranges are reported for 9 baseline and 9 post-manure application events as follows: dissolved reactive phosphorus, less than (<) 0.013−2.16 milligrams per liter (mg/L); nitrate plus nitrite, filtered, 0.32−77 mg/L; ammonia, filtered, <0.05−2.6 mg/L; total phosphorus, <0.01−12.8 mg/L; and total nitrogen, 1.49−77.2 mg/L. Volumes are reported for 6 baseline and 9 post-manure application rainfall events. None of the post-manure application runoff volumes were significantly different by plot or by treatment type (raw manure versus treated manure).

Because concentrations alone do not reflect the true effects of different manure treatments, loads and flow-weighted mean concentrations of nutrients during post-manure application rainfall events were compared between plots with treated manure and those with raw manure. Loads of dissolved reactive phosphorus, total phosphorus, nitrate plus nitrite, and total nitrogen were calculated using the U.S. Geological Survey Graphical Constituent Loading and Analysis System. Loads of ammonia were not calculated because many of the ammonia concentrations were below the reporting limit.

During the post-manure application period, higher nitrogen loads resulted from tile flow than surface runoff. For phosphorus, the opposite was true in that higher loads resulted from surface runoff than tile flow. Combined loads (surface runoff and tile flow) of dissolved reactive phosphorus were significantly different between raw manure and treated manure plots, but there was no significant difference in combined loads of total phosphorus, nitrate plus nitrite, or total nitrogen between raw manure and treated manure plots. Flow-weighted mean concentrations were calculated for the combined loads for the post-manure application rainfall events. Flow-weighted mean concentrations of dissolved reactive phosphorus and, to a lesser extent, total phosphorus were significantly different between raw manure and treated manure plots. Flow-weighted mean concentrations of nitrate plus nitrite and total nitrogen were not significantly different between raw manure and treated manure plots. The differences in loads and flow-weighted mean concentrations between raw manure and treated manure plots indicate that dissolved reactive phosphorus was likely retained in the soil and hydrological transport was reduced for the plots amended with the treated manure as compared to raw manure. Although confirmation field testing needs to be done, these results indicate that the use of this coagulant/polymer mixture shows potential in helping to reduce flow of dissolved phosphorus from agricultural fields with applied manure.

Introduction

The livestock industry is continuing a trend towards fewer but larger confined operations (U.S. Environmental Protection Agency, 2012). Manure and wastewater from large livestock operations are a growing concern because of the potential to negatively affect water quality. Application of animal wastes on fields can cause excess nutrients to permeate soils and move into receiving waters and groundwater resources (Burkholder and others, 2007). Liquid, lagoon manure produced by dairy operations is typically dilute, the nutrient concentration is low, and the water content is high. Treatment methods to remove excess water from manure and to concentrate nutrients are needed. This will decrease the overall manure volume and weight and allow facilities to transport the waste greater distances, so nutrients are not overapplied on nearby fields. In addition, manure treatment that temporarily binds nutrients could reduce the transport from agricultural fields into surface water and groundwater and allow nutrients to remain available for crop growth.

Treatment systems that transform dairy manure into a dewatered product are needed in the Western Lake Erie Basin, where there is a high density of animal agriculture. Nutrient excesses can cause proliferation of cyanobacteria and harmful algal blooms (HABs), which may produce microcystin and other toxins. Concentrations of microcystin above World Health Organization guidelines have been observed in the Western Lake Erie Basin the last few years and in Ohio inland lakes (Ohio Environmental Protection Agency, 2019); this has been attributed, in part, to phosphorus loading from manure and fertilizer applications (Hoorman and others, 2008; Michalak and others, 2013; Ohio Department of Agriculture and others, 2013).

One promising technology for treating manure to reduce adverse environmental effects is solid-liquid separation enhanced with iron and aluminum salts, with or without the addition of natural (Garcia and others, 2007) or synthetic organic polymers (Chastain and others, 2001; DeBusk and others, 2008). Although polymer flocculants have been used in the food and municipal waste industries for years to enhance solid-liquid separation, the use of this technology for managing animal wastes is still new (Timby and others, 2004). Previous soil column and pilot-field studies in which investigators tested polymers for dewatering animal manure have been documented in Arkansas (Timby and others, 2004), South Carolina (Garcia and others, 2007), and North Carolina (Szogi and Vanotti, 2007); however, to our knowledge, this type of study has not been done in Ohio. Most of these studies were laboratory or feasibility studies, and only a few demonstrated production of a potential fertilizer material.

The U.S. Geological Survey (USGS) and Bowling Green State University (BGSU), in cooperation with the Ohio Water Development Authority, and in collaboration with The Ohio State University Agricultural Research and Development Center and the Village of Ottawa, field tested a dewatering treatment process for dairy manure. By amending manure with a coagulant/polymer mixture, the manure is dewatered yet retains nutrient-rich fertilizer in the solid phase. BGSU tested different dairy manure dewatering treatments for their abilities to slowly release nutrients in laboratory batch and soil column studies; these tests were used to identify the best treatment for field testing. For this study, data on nutrient concentrations and water volumes in surface runoff and tile flow from pilot-scale plots that received dairy manure applications were collected. The goal of the study was to demonstrate potential improved water-quality benefits of a manure dewatering treatment process in a natural field setting.

Purpose and Scope

The purpose of this report is to describe the results of pilot-scale testing of dairy manure treatments to reduce nutrient transport from land application in northwest Ohio. Data were collected during rainfall events that resulted in substantial surface runoff and (or) tile flow (hereinafter called "events") from October 2015 through early November 2017. These included 4 baseline events used for quality control and protocol development (baseline 1), 9 baseline events used for determining concentrations and volumes (baseline 2), and 9 events after manure application (post-manure events). For post-manure events, calculated loads and flow-weighted mean concentrations of nutrients from plots with polymer-treated manure were compared to those with liquid lagoon manure.

Methods of Study

This section describes the methods used for this study including those for sample collection and processing, application of the manure treatment, quality control and quality assurance purposes, laboratory analysis, and calculation of loads and flow-weighted mean concentrations of nutrients. The pilot-scale study site and equipment used also are described.

Site and Equipment Description

The pilot-scale field study was done at The Ohio State University Agricultural Research and Development Center (OARDC) Northwest Agricultural Research Station (NWARS; fig. 1). This facility was established in 1974 and consists of eight plots, arranged in two blocks of four plots each on either side of a sampling and flow monitoring building (Logan and others, 1994). Each test plot (30 feet [ft] by 100 ft each) is separated from adjacent plots by a grassed berm about 1-meter wide and has a heavy plastic barrier around the perimeter to a depth of about 1.5 meters (fig. 2*A*). The surface slope is less than (<) 1 percent. Surface runoff drains into a concrete gutter, which directs the water into a pipe that transports the runoff into the building. Subsurface flow is collected by tile, 10 centimeters in diameter, located 1 meter deep under the center of the plot, draining towards the building at a slope of 0.2 percent. Tile flow is transported into the building by a separate pipe than surface runoff. Manure was treated at the Village of Ottawa Wastewater Treatment Plant. Daily rainfall amounts were obtained from the OARDC Weather System for the Northwest Station in Custar, Ohio (The Ohio State University, 2017).

Each plot transports water through two pipes into the monitoring building—one for surface runoff and one for tile (subsurface) flow. In the building, each pipe (16 total) is fitted with an L-shaped polyvinyl chloride (PVC) flow director that empties into a tipping bucket (fig. 2*B*). The 16 tipping buckets were calibrated through an inlet to the PVC flow director and were set to tip when filled with 600–800 milliliters (mL) plus or minus (±) 10 mL, depending on the capacity of the bucket. A discharge line from each flow director was attached to a refrigerated automatic sampler (fig. 2*C*). Communicating through a modem, a digital I/O port expander (SDM–1O16, Campbell Scientific, Inc., Logan, Utah) coordinated the operation of each tipping bucket and automatic sampler. A data logger (CR850, Campbell Scientific) contained in a fiberglass box recorded the time and number of tips every 5 minutes and recorded when sample bottles were filled.

Sampling and Sample Processing

The refrigerated automatic samplers, each with 14 bottles, were numbered from 1 through 16 (fig. 3). Samples were collected during rainfall events that resulted in substantial surface runoff and (or) tile flow. The volume required before the first sample was collected for each event was initially set to 30 liters (L) for surface runoff and 15 L for tile flow; this could be adjusted based on antecedent field conditions. A program was written that adjusted the number of tips required for a sample to be collected (after the first sample) based on a change in flow rate. For small events, 30- and 15-L intervals were usually maintained throughout the event. For larger events, larger volume intervals would be collected, some as large as 500 L. When a sample was triggered to be collected, the line was rinsed first, then each bottle filled to

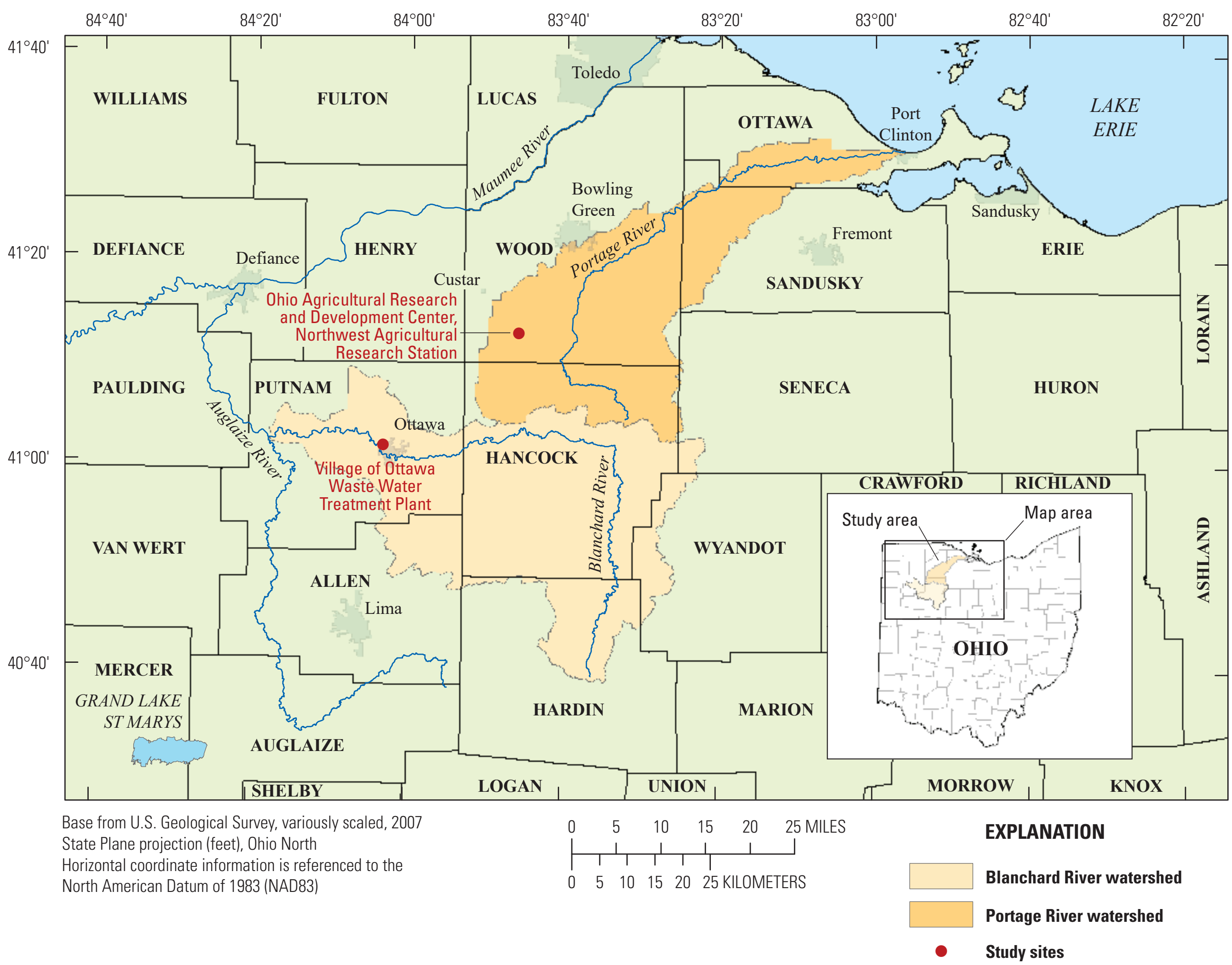

Figure 1. Location of the study area, northwest Ohio.

Photographs by Robert Darner, U.S. Geological Survey

Figure 2. The, *A*, agricultural test plots separated by grassed berms with a central sampling and volume monitoring building; *B*, polyvinyl chloride flow directors and tipping buckets for measuring volume; and *C*, 16 automatic samplers to collect water samples for nutrient analyses.

approximately 600 mL. For small events (less than 6 bottles were full), all sample bottles with adequate water were processed and analyzed. For larger events, one-third to two-thirds of the bottles with water were processed and analyzed; the bottles were selected for processing to provide adequate temporal and volume coverage over the event hydrograph.

Bottle contents were processed on site within 72 hours of collection for subsequent nutrient analyses. Samples for ammonia (filtered), nitrate plus nitrite (filtered), dissolved reactive phosphorus, total nitrogen, and total phosphorus were processed and preserved as per standard USGS methods (Wilde and others, 2004, chap. A5). A 4-layer, 0.45-micron, 25-millimeter diameter syringe filter (Tisch Scientific,

Sampler number	Tile (T) or surface (S)	Plot number		Sampler number	Tile (T) or surface (S)	Plot number
01	T	1	CENTRAL WALKWAY	16	T	8
02	S			15	S	
03	T	2		14	T	7
04	S			13	S	
05	S	3		12	S	6
06	T			11	T	
07	S	4		10	S	5
08	T			09	T	
			DOOR			

Figure 3. The numbering system for agricultural test plots and automatic samplers in the central building at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station.

GD17034) was used to collect 10–20 mL of sample for filtered nutrients. Unfiltered samples for total nutrients (125 mL) were preserved with 1.0 mL of 1:7 sulfuric acid. All samples were stored on ice immediately after processing and for transport to the laboratory.

Manure Treatment and Data Collection

The data-collection period was from October 28, 2015, through November 9, 2017, and included 22 events in which nutrient samples were collected (table 1 and fig. 4). From October 2015 through early May 2017, baseline samples were collected at the NWARS. The original intention was to identify any baseline differences in nutrient concentrations and flow conditions between plots as the plots had not been used for 3 years prior to this study. After the first few events, however, it was noted that there were both flow and water-quality differences among the plots. The project team decided to delay the application of manure for 1 year to improve the similarity of the plots in terms of surface runoff and tile flow. On May 23, 2016, the plots were tilled, berms redefined, and animal burrows were removed. On October 12, 2016, the plots were chisel-plowed to break up soil and further restore uniform grading. There were no farming activities in 2016. Samples were segregated into two baseline periods based on the first attempt to redefine the plots. The first baseline period (baseline 1, October 2015–February 2016) included four events that occurred before the plots were first redefined (identified as B1.1–B1.4). Data from baseline 1 were used to collect quality-control data and establish sampling and analysis protocols. Data from the second baseline period (baseline 2, June 2016 through early May 2017) were used to determine baseline nutrient concentrations for 9 events (B2.1–B2.9) and baseline volumes for the 6 events that occurred after the October 12, 2016, grading (B2.4–B2.9). Baseline volumes were approved and reported for all events; however, baseline volumes before the grading on October 12, 2016, were not used in this report because runoff and flow from the plots were not considered to be uniform.

Prior to and during the baseline period, BGSU tested different dairy manure dewatering treatments for their abilities to promote separation of solids and liquids, sequester agricultural nutrients from water, and slowly release nutrients when mixed with soil over time. Extensive laboratory tests under local soil conditions using dairy manure indicated that, of the materials tested, an aluminum-based coagulant commonly used in drinking water and wastewater applications (Aqua Hawk 1010P, Hawkins Water Treatment Group, Roseville, Minnesota) and a cationic based flocculant/polymer (Zetag 8816, Hawkins Water Treatment Group) performed best by the criteria of (1) separating solids and water, (2) reducing the nutrient concentration in the filtrate water, and (3) slowing the release of nutrients in soil relative to untreated manure.

On May 9, 2017, liquid manure was obtained from a local dairy. Mean nutrient and solid levels in the liquid lagoon dairy manure were as follows: dissolved reactive phosphorus, 160 milligrams phosphorus per liter (mgP/L); ammonia, 970 milligrams nitrogen per liter (mgN/L); nitrate plus nitrite, 38 mgN/L; total phosphorus, 320 mgP/L; total nitrogen, 1,200 mgN/L; and total solids, 30 mg/L. On May 9–17, 2017, 4,000 gallons of liquid manure were treated at the Ottawa Wastewater Treatment Plant with Aqua Hawk 1010P

Table 1. Timeline and sample information from 16 automatic samplers during rainfall events that resulted in substantial surface runoff and (or) tile flow and farming activities at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17.

[B, baseline; BGSU, Bowling Green State University; USGS, U.S. Geological Survey; QC, quality-control; --, no samples were collected; ex, except; NA, not applicable; PM, post-manure application]

Event number	Event dates (month day, year)	Bottles filled	Tile flow occurred, sampler number	Surface runoff occurred, sampler number	Runoff type	Analyzing agency	Data use
			Baseline 1				
B1.1	October 28–29, 2015	59	1[a], 3	All	Surface, mainly	BGSU, USGS	QC and establish protocols
B1.2	November 27–29, 2015	23	--	All ex 4	Surface	BGSU, USGS	QC and establish protocols
B1.3	December 26, 2015–January 4, 2016	151	All	All	Surface and tile	USGS	QC and establish protocols
B1.4	January 30–Feb 6, 2016	93	All	All ex 4,12	Tile, mainly	BGSU, USGS	QC and establish protocols
NA	May 23, 2016	Plots were tilled and berms redefined					
			Baseline 2				
B2.1	June 22–23, 2016	51	All ex 16	All ex 10[b]	Surface and tile	BGSU	Concentrations for filtered constituents
B2.2	August 12–16, 2016	42	All[a]	All ex 10[b]	Surface	BGSU	Concentrations
B2.3	September 17–18, 2016	60	3, 6, 9, 11, 14[b]	All ex 10[b]	Surface, mainly	BGSU	Concentrations except for total phosphorus
NA	October 12, 2016	Plots were chisel-plowed to break up soil and restore the grading					
B2.4	October 20–21, 2016	38	3, 6[a], 9	All ex 4, 10[b]	Surface, mainly	BGSU	Concentrations and volumes
B2.5	January 2–7, 2017	141	All	All	Surface and tile	BGSU, USGS	Concentrations and volumes
B2.6	January 10–15, 2017	195	All	All	Surface and tile	BGSU, USGS	Concentrations and volumes
B2.7	February 6–10, 2017	61	All	All ex 4[b]	Surface and tile	BGSU	Concentrations and volumes
B2.8	April 3–16, 2017	120	All	All ex 4[b]	Surface and tile	BGSU	Concentrations and volumes
B2.9	May 4–8, 2017	208	All	All ex 4[b]	Surface and tile	BGSU, USGS	Concentrations and volumes
NA	May 18, 2017	Manure applied					
			Post-manure application				
PM.1	May 19–22, 2017	57	All ex 8	All ex 4[b]	Surface and tile	BGSU, USGS	Post-manure loads
NA	May 26, 2017	Plots were tilled to incorporate manure					
PM.2	May 26–27, 2017	15	All[a]	All ex 4[b]	Surface and tile	BGSU	Post-manure loads
PM.3	May 28–29, 2017	15	1, 3, 6, 9, 14[a]	All ex 4[b]	Surface and tile	BGSU	Post-manure loads
NA	June 1, 2017	Plots were planted with corn					

Table 1. Timeline and sample information from 16 automatic samplers during rainfall events that resulted in substantial surface runoff and (or) tile flow and farming activities at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17.—Continued

[B, baseline; BGSU, Bowling Green State University; USGS, U.S. Geological Survey; QC, quality-control; --, no samples were collected; ex, except; NA, not applicable; PM, post-manure application]

Event number	Event dates (month day, year)	Bottles filled	Tile flow occurred, sampler number	Surface runoff occurred, sampler number	Runoff type	Analyzing agency	Data use
Post-manure application—Continued							
PM.4	June 13–14, 2017	137	All	All ex 4[b]	Surface and tile	BGSU, USGS	Post-manure loads
PM.5	June 23–24, 2017	118	All	All ex 4[b]	Surface and tile	BGSU, USGS	Post-manure loads
PM.6	July 7, 2017	96	All ex 8	All ex 4[b]	Surface, mainly	BGSU, USGS	Post-manure loads
PM.7	July 10–11, 2017	210	All	All ex 4[b]	Surface and tile	BGSU, USGS	Post-manure loads
PM.8	August 1–10, 2017	210	All	All ex 4[b]	Surface and tile	BGSU, USGS	Post-manure loads
PM.9	November 4–10, 2017	213	All	All	Surface and tile	BGSU, USGS	Post-manure loads
NA	November 13, 2017	Corn was harvested					

[a]Small volumes were recorded for this sampler (s) (<10 liters).

[b]Sampler volumes were disparate from other sampler volumes so were suspected to be erroneous.

coagulant and Zetag 8816 polymer and allowed to filter and dry on sand beds. Samples of the solid manure cake portion left on the sand beds were analyzed for total phosphorus and nitrogen. Mean nutrient levels were as follows: total phosphorus, 0.0013 grams of phosphorus per gram of treated solid cake; and total nitrogen, 0.0049 grams of nitrogen per gram of treated solid cake. On May 18, 2017, manure was applied to the surface of the test plots. Calculations were done to ensure the amount of total phosphorus in the total volume of liquid lagoon manure (raw) and total weight in coagulant-treated manure (treated) were consistent. Amounts applied were as follows:

- Plots 1 and 2 did not receive manure treatment and results were not used in the study after early work to establish protocols because of equipment malfunctions and (or) atypical runoff or flow volumes;
- Plots 4, 6, and 8 each received 800 gallons (3,000 L) of raw manure. This equates to a rate of 11,400 gallons per acre. The mean total phosphorus application of raw manure per plot was 970,000 milligrams (mg) of phosphorus. This amount was chosen to simulate the maximum allowable application of liquid manure. The maximum amount allowed by Nutrient Management Code 590 (U.S. Department of Agriculture, 2013) is 910 gallons (3,400 L). Due to the small size of the plots and the structure of the manure applicator, 3,000 L was the amount applied; and
- Plots 3, 5, and 7 each received 1,400 pounds (640,000 grams) of treated manure. The mean total phosphorus application of treated manure per plot was 830,000 mg of phosphorus. The mass of treated manure was planned based on laboratory analysis of solid, treated manure to match the mass of phosphorus being applied on the raw manure plots. However, when the solid samples from the actual pilot test were analyzed, they contained lower amounts of phosphorus.

To simulate typical farming practices, the plots were to be tilled after manure application to incorporate manure; however, 4 days of rain after manure application (totaling just over 1 inch) resulted in the plots being too wet for tilling. On May 26, 2017, the plots dried out and were tilled to incorporate the manure, and on June 1, the plots were planted with corn. On June 2, the plots were sprayed with pesticides (Corvus; Atrazine 4L; 2,4-D Ester lv4; Mad Dog+; and Choice). Samples were collected for nine events during the post-manure period (PM.1–PM.9). On November 13, 2017, corn was harvested from two rows (103 ft long) down the center of each

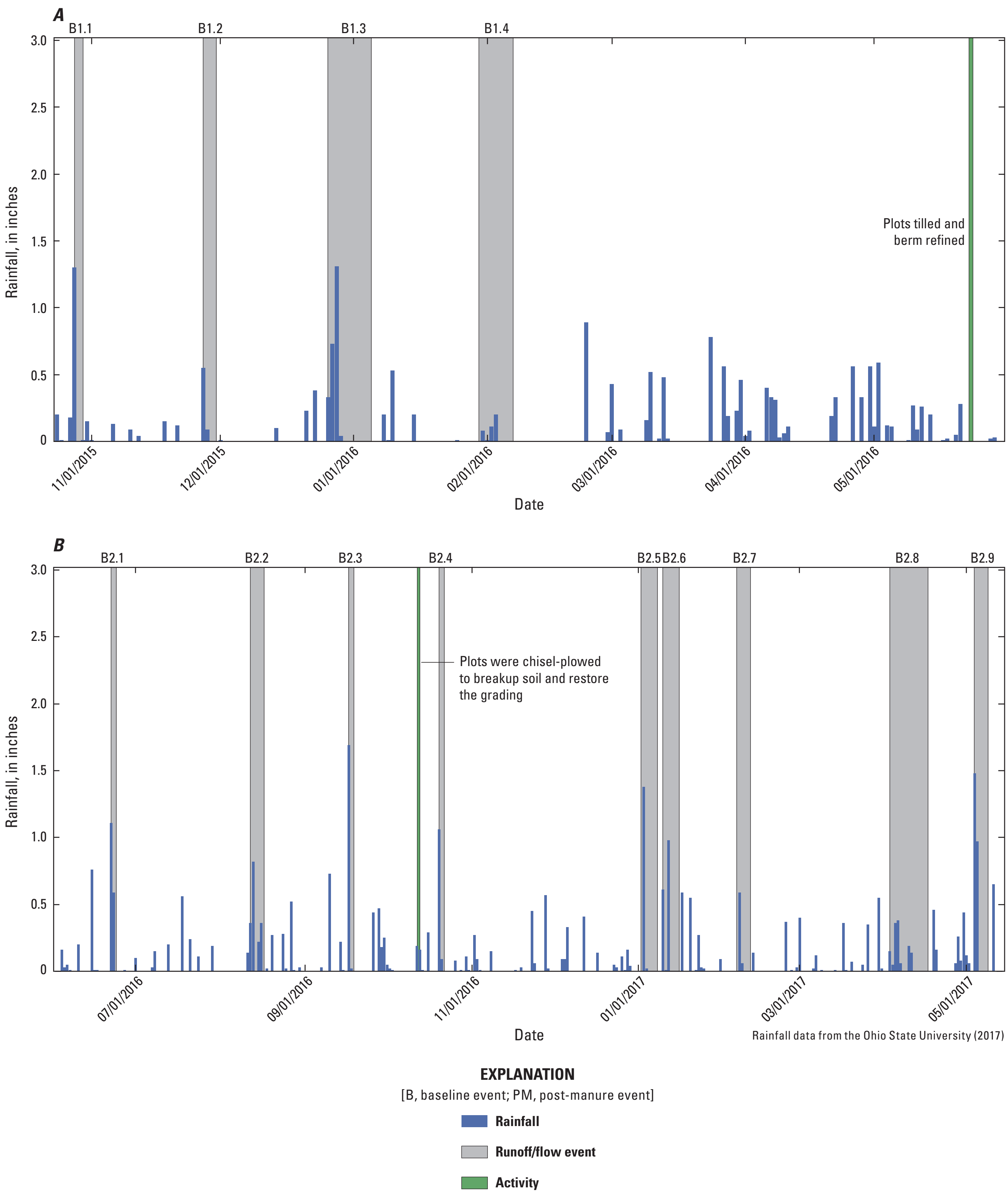

Figure 4. Time-series plots of, *A*, baseline 1, *B*, baseline 2, and *C*, post-manure application events that resulted in substantial surface runoff and (or) tile flow, rainfall amounts, and farming activities at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17.

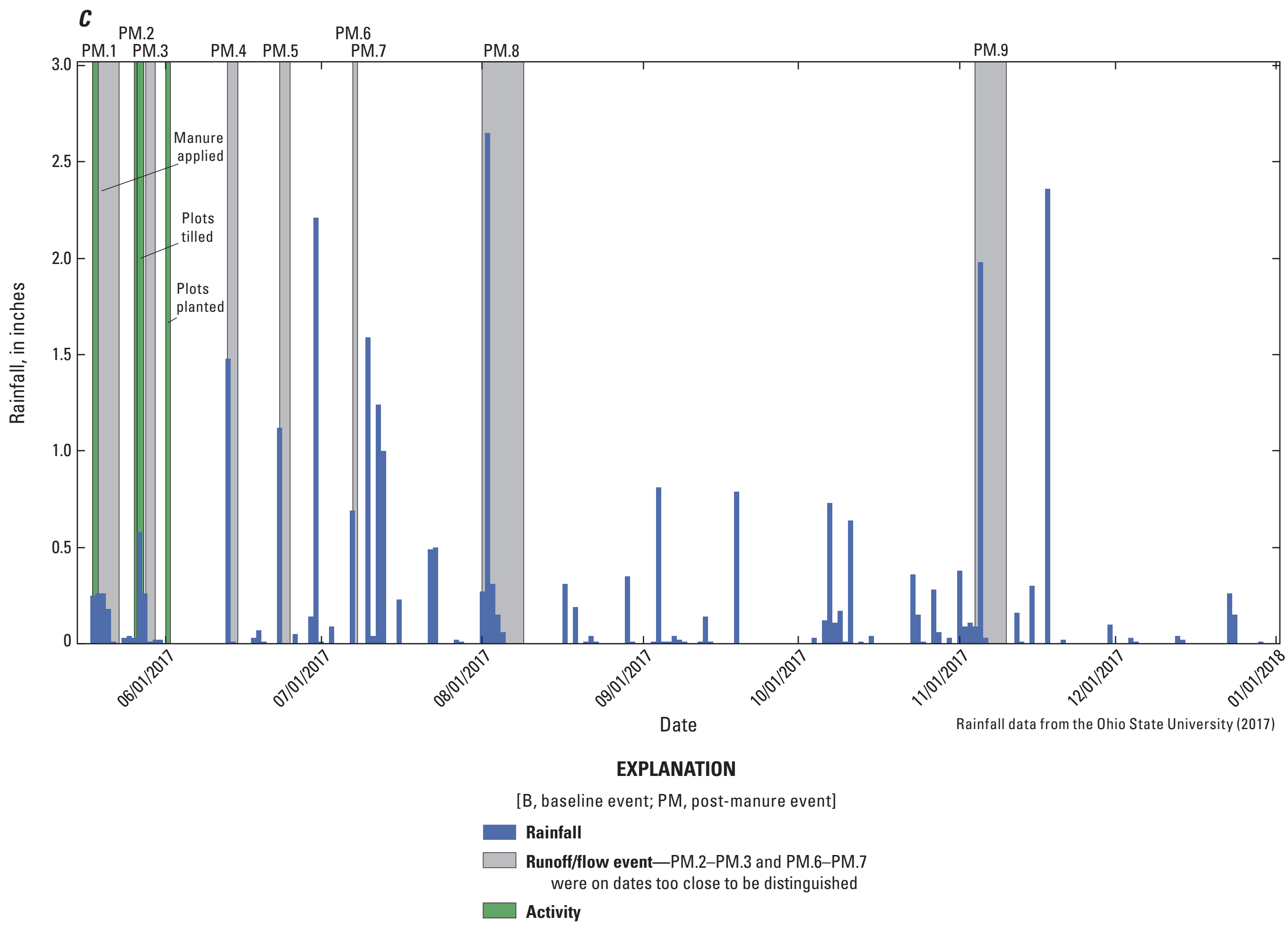

Figure 4. Time-series plots of, *A*, baseline 1, *B*, baseline 2, and *C*, post-manure application events that resulted in substantial surface runoff and (or) tile flow, rainfall amounts, and farming activities at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17—Continued

plot and was used to calculate yield in bushels per acre. The corn obtained from harvesting was weighed and measured for moisture content.

Field Quality Assurance and Quality Control

Quality-assurance and quality-control objectives are to (1) ensure that procedures are done consistently with established methods and that data are collected and reported in a complete and accurate manner, (2) identify any sampling and analytical bias, and (3) quantify the variability of the measurement process.

To address number 1, sampling, processing, and documentation procedures were distributed to all personnel. A quality-assurance project plan was written at the start of the project, approved by all parties, and updated as needed. To provide concise field instructions, a so-called “cheat sheet” was distributed to field personnel (appendix 1). To address quality-assurance and quality-control objectives 2 and 3, sampling crews collected field blanks, filter blanks, source solution blanks, and replicates throughout the sampling period. Blanks were processed using inorganic-free water provided by the USGS. Field blanks were used to determine whether contamination was introduced by the entire sampling process, including carry over from the intake line, sample bottles, filtering, laboratory bottles, and laboratory procedures. To collect a field blank, the intake line was disconnected from the flow director and placed in a graduated cylinder containing 500 mL of blank water. The 500 mL of blank water was first pumped through the sample line to rinse the line and discarded. A second aliquot of 500 mL of blank water was then passed through the intake line to the automatic sampler and collected and analyzed. Filter blanks were 25 mL of inorganic blank water filtered through the syringe/filter system and processed in the same manner as a regular sample. Source solution blanks were prepared by pouring blank water directly into the sample bottle or bag at the field site. Sampling crews collected replicates by processing and analyzing two subsamples from the same 1-L sample bottle for filtered and total nutrients.

Laboratory Analyses and Associated Laboratory Quality Control

Surface runoff and tile flow samples for nutrient analyses (table 2) were transported to the BGSU laboratory by car or to the USGS National Water Quality Laboratory (NWQL) in Denver, Colorado, by overnight delivery. During most baseline 1 events, samples were analyzed by BGSU for total and filtered nutrients with some split samples shipped to and analyzed by the USGS NWQL to complete the USGS laboratory approval process; for one event in December 2015 (B1.3), the USGS analyzed all samples due to lack of available BGSU staff. Availability of staff and size of event dictated which laboratory analyzed samples during the baseline 2 and post-manure period. For most baseline 2 and post-manure events, BGSU collected and analyzed samples for filtered nutrients, and the USGS collected samples to be analyzed by the USGS NWQL for total (unfiltered) nutrients.

To quantify the nutrient content of both the raw and treated manure that would be applied during the pilot test, samples of both types of manure were analyzed by BGSU. Raw, liquid lagoon manure was analyzed for both filtered and total nutrients. The raw manure was diluted by a factor of 100 and then syringe filtered for analysis for dissolved reactive phosphorus, ammonia, and nitrate plus nitrite (table 2). Raw (liquid) and treated (solid) manure samples were digested using the alkaline persulfate method and then analyzed for total phosphorus and total nitrogen (table 2).

The USGS NWQL has a documented quality management system, is certified by four state agencies, and takes part in laboratory proficiency testing and audits (for more information, visit https://nwql.usgs.gov/Public/quality.shtml). BGSU participates in the USGS Standard Reference Sample program (for more information, visit https://bqs.usgs.gov/srs/), analyzes certified wastewater samples with each analytical run, and was approved through the USGS laboratory approval process. Data were examined to identify outliers including calculating the ratios of filtered to total nitrogen or phosphorus. To confirm results, reruns were requested for outliers and (or) for filtered to total ratios greater than 1.2. Erroneous values were marked with a data-quality indicator of “Q” in the USGS database, indicating “reviewed and rejected.”

Data Management and Calculations of Loads and Flow-Weighted Mean Concentrations of Nutrients

Water volume and nutrient concentration data for approved periods of record are available through the USGS National Water Information System (NWIS) public database, NWIS web (U.S. Geological Survey, 2020). USGS 15-digit site identification numbers were assigned with the first 13 digits the same (4113050834531) and the last 2 digits specific for each tipping bucket/sampler (01–16) (fig. 3). Station names for the tipping buckets with associated samplers are “OARDC NWARS test plot [1–8] subsurface [OR surface] drainage.” Continuous volume data were stored in the USGS NWIS database using parameter code 72270 (volume of water, total during measurement interval, liters) and are available through NWIS web by changing the last two digits for the designated sampler number in this link: https://waterdata.usgs.gov/oh/nwis/uv?site_no=411305083453101. Discrete nutrient analyses are stored in the USGS NWIS database for water quality samples, available on NWIS web by entering the site identification number (https://nwis.waterdata.usgs.gov/oh/nwis/qwdata).

Results from quality-control samples were examined to aid in data interpretations. Concentration and relative percent differences (RPDs) between paired replicates of discrete nutrient samples were examined to quantify variability (eq. 1). If both replicates had concentrations below detection, RPDs were not calculated. RPD was calculated as follows:

$$RPD = \frac{|R_A - R_B|}{\left(\frac{R_A + R_B}{2}\right)} \times 100 \qquad (1)$$

where

R_A is the concentration in replicate A, and

R_B is the concentration in replicate B.

Detections in the blanks were compared to event flow samples. If a flow sample result was less than or equal to (≤) two times the detection in the associated blank, a remark code “V” for “value affected by contamination” was added to the result in the NWIS database for any qualifying samples for the event for that constituent.

For post-manure events, nutrient loads were calculated using the USGS Graphical Constituent Loading and Analysis System (GCLAS; Koltun and others, 2006). For this study, GCLAS was used to estimate concentrations for periods between measured concentrations during events based on the relation between nutrient concentrations and flow. Loads of dissolved reactive phosphorus, nitrate plus nitrite, total nitrogen, and total phosphorus were estimated for individual events. Loads of ammonia were not calculated because more than one-half of values for post-manure samples were below the reporting limit. GCLAS assigns censored values their censoring level when plotting or using the values to compute loads (Koltun and others, 2006), so the reporting limit was substituted for dissolved reactive phosphorus and total phosphorus concentrations below detection. Event loads calculated for events with sample concentrations below the reporting limit were reported as less than the total calculated load. Flow-weighted mean concentrations were calculated for each sampler in each event by dividing the total load by the total volume. Flow-weighted mean concentrations were calculated per plot for each event by dividing the sum of the total loads for both samplers associated with that plot (surface runoff and tile flow) by the sum of the volumes for both samplers. By substituting the reporting limit for samples with concentrations

Table 2. Laboratory method information for nutrients analyzed by the U.S. Geological Survey National Water Quality Laboratory and Bowling Green State University in surface runoff and tile flow samples from eight test plots at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17.

[USGS, U.S. Geological Survey; NWIS, National Water Information System; mg/L, milligram per liter; EPA, U.S. Environmental Protection Agency]

Constituent	NWIS parameter code	USGS National Water Quality Laboratory		Bowling Green State University, Department of Chemistry	
		Method and NWIS method code	Reporting limit (mg/L)	Method and NWIS method code	Reporting limit (mg/L)
Ammonia, filtered, as nitrogen	00608	Colorimetry and salicylate-hypochlorate (Fishman, 1993), SHC02	0.01	Colorimetry, alkaline phenol and hypochlorite (auto phenate), EPA–103–A, EPA 350.1 (EPA, 1993), CL015	0.29, 0.05[a]
Nitrate plus nitrite, filtered, as nitrogen	00631	Colorimetry with enzyme reduction-diazolization (Patton and Kryskalla, 2011), RED01	0.04	Colorimetry, cadmium reduction, EPA–114–A, EPA 353.2 (EPA, 1993), CDR06	0.31
Dissolved reactive phosphorus	00671[b]	Colorimetry with phosphomolybdate (Fishman, 1993), PHM01	0.004	Colorimetry, acidic molybdate, EPA–118–A, EPA 365.3, (EPA, 1993), 00119	0.013
Total nitrogen, unfiltered	62855	Alkaline persulfate digestion (Patton and Kryskalla, 2003), AKPO1	0.05	Colorimetry,alkaline persulfate digestion (EPA, 1993; Patton and Kryskalla, 2003), AKPO1	0.31
Total phosphorus, unfiltered	00665	Colorimetry, EPA 365.1 (EPA, 1993), CL021	0.004	Colorimetry by discrete analyzer, alkaline-persulfate digestion, (EPA, 1993; Patton and Kryskalla, 2003), PSF03	0.01

[a]Reporting limit was lower after April 1, 2017.

[b]NWIS parameter code is for dissolved orthophosphate, as phosphorus.

below detection, resultant loads and flow-weighted means may be biased high. However, the overall effect on the resultant total event load or flow-weighted mean is minor.

Because the volume, load, and flow-weighted mean concentration data were not normally distributed (based on Shapiro-Wilk tests; Helsel and Hirsch, 2002), nonparametric statistical tests were used. The Kruskal-Wallis test (Helsel and Hirsch, 2002) was used to determine if there were differences in median values of volume and flow-weighted mean concentration among the six test plots. If the Kruskal-Wallis test determined that at least one median differed from the others, a Dunn's multiple comparison test (which includes a Bonferroni adjustment for the pair-wise significance levels; Dunn, 1964) on all possible pairs of plots was done to determine differences between any of the test plots. The Wilcoxon rank-sum test (Helsel and Hirsch, 2002) was used to identify any significant differences in median values of volume and flow-weighted mean concentration by manure treatment type (values for the three treated manure plots were compared to values for the three raw manure plots). The Wilcoxon signed-rank test was used to identify if the median of differences between paired observations equaled zero. Specifically, for each event, loads were summed for all plots by manure treatment type, and the test was run on the differences per event to determine if one treatment type had greater nutrient loads than the other treatment type. For statistical tests, a significance level of $\alpha=0.05$ was used.

Quality-Control Measures of Bias and Variability

To characterize bias, sampling crews collected 9 field blanks, 4 filter blanks, and 3 source solution blanks for 5 nutrient constituents (table 3). There were no detections in any of the BGSU-analyzed field blanks. There were detections, however, for every constituent in USGS-analyzed field blanks because reporting limits were lower than reporting limits for BGSU-analyzed samples. There were no detections in the filter or source solution blanks analyzed by BGSU or by the USGS.

It was not necessary to qualify any nitrate plus nitrite or total nitrogen results, as concentrations in surface runoff and tile flow samples were greater than (>) two times the concentrations of detections in field blanks. For the other constituents, if a surface runoff or tile flow sample result was ≤ two times the associated field blank concentration during that event, the environmental sample result was qualified in the database. There were three detections of dissolved reactive phosphorus in USGS field blank samples, ranging from 0.005 to 0.009 mg/L (table 3); as a result, five USGS samples from the B2.6 event were qualified. For ammonia, the two detections in USGS samples (0.01 and 0.02 mg/L) were considerably less than BGSU reporting limits (<0.29 or <0.05 mg/L). Five USGS samples for ammonia in the B1.4 event and 10 USGS samples in the B2.6 event were qualified. Because USGS samples were not used to calculate loads of filtered nutrients, blank detections of dissolved reactive phosphorus and ammonia did not affect loading calculations. That was not the case for total phosphorus. Total phosphorus was detected in 6 out of 8 USGS field blanks in concentrations ranging from 0.009 to 0.092 mg/L (table 3). These detections caused several sample results to be qualified as "V" for "value affected by contamination." These qualified values were examined and used in subsequent data analysis only if the value fit with surrounding data points.

To characterize variability, replicate samples were collected. BGSU collected and analyzed 35 or 38 replicates per filtered nutrient and the USGS collected and analyzed 21 replicates for total nutrients (table 4). For BGSU replicate pairs, median RPDs for ammonia, nitrate plus nitrite, and dissolved reactive phosphorus ranged from 0.0–1.3 percent. Ammonia was only detected in five replicate pairs with a maximum RPD of 4.5 percent. For nitrate plus nitrite, RPDs were <20 percent for all but two replicate pairs, in which the RPDs were 25 percent (18 and 14 mg/L) and 29.8 percent (0.690 and 0.511 mg/L). For dissolved reactive phosphorus, the highest RPD (65.1 percent) resulted from two low concentrations (0.055 and 0.028 mg/L). For USGS-collected replicate pairs, median RPDs for total phosphorus and nitrogen were both 3.8 percent. Only one sample for total phosphorus showed a large difference in terms of concentration (2.88 and 1.52 mg/L) and RPD (61.8 percent). Similarly, only one sample for total nitrogen showed a large difference in terms of concentration (12.9 and 9.95 mg/L) and RPD (25.8 percent).

Sampling Events and Concentrations of Nutrients in Surface Runoff and Tile Flow Samples

Rainfall amounts, soil moisture conditions, and time of year varied among events. Two events were surface only, four were mainly surface, one was mainly tile, and the remainder were various degrees of surface and tile (table 1). The mainly tile event (B1.4) occurred in winter when the ground was likely frozen so that water may have infiltrated through fissures in the subsurface. The surface-only or surface-mainly events likely occurred following small amounts of rainfall and (or) when the ground was dry due to lack of antecedent rain. If all bottles filled, a total of 224 bottles would be collected (16 samplers with 14 bottles per sampler). At least 200 bottles were collected during 4 events (B2.9, PM.7, PM.8, and PM.9), and the fewest number of bottles filled (15) occurred during 2 small events in May 2017 (PM.2 and PM.3). Equipment malfunctions resulted in missing data for sampler 10 during B2.1–B2.4 and for sampler 4 during B2.4–PM.8.

Concentrations of dissolved reactive phosphorus, total phosphorus, nitrate plus nitrite, total nitrogen, and ammonia are presented for samples collected for events during

Table 3. Quality-control field blank, filter blank, and source solution blank data for nutrients, 2015–17.

[B, baseline; BGSU, Bowling Green State University; <, less than; USGS, U.S. Geological Survey; --, not done; NA, not applicable; PM, post-manure application; detections in blanks are bolded and italicized]

Event	Collecting/ analyzing agency	Concentration, in milligrams per liter				
		Dissolved reactive phosphorus	Ammonia	Nitrate plus nitrite	Total phosphorus	Total nitrogen
Field blanks						
B1.1	BGSU	<0.013	<0.29	<0.31	<0.013	<0.31
B1.1	USGS	<0.004	<0.01	***0.115***	<0.004	***0.16***
B1.2	BGSU	<0.013	<0.29	<0.31	--	--
B1.2	USGS	***0.005***	<0.01	***0.097***	***0.012***	***0.15***
B1.4	BGSU	<0.013	<0.29	<0.31	--	--
B1.4	USGS	***0.007***	***0.01***[a]	***0.100***	***0.022***[b]	***0.016***
B2.6	BGSU	<0.013	<0.29	<0.31	--	--
B2.6	USGS	***0.009***[a]	***0.02***[a]	<0.04	***0.092***[a]	***0.39***
B2.8	BGSU	<0.013	<0.29	<0.31	--	--
B2.9	USGS	--	--	--	***0.009***	***0.08***
PM.1	USGS	--	--	--	***0.061***[a]	<0.05
PM.7	USGS	--	--	--	***0.04***[b]	***0.126***
PM.9	USGS	--	--	--	<0.05	<0.25
Filter blanks						
B1.4	USGS	<0.004	<0.01	<0.04	NA	NA
B2.5[c]	BGSU	<0.013	<0.29	<0.31	NA	NA
B2.8	BGSU	<0.013	<0.29	<0.31	NA	NA
B2.9	USGS	<0.004	<0.01	<0.04	NA	NA
Source solution blanks						
B2.9	USGS	--	--	--	<0.004	<0.05
PM.7	USGS	--	--	--	<0.004	<0.05
PM.9	USGS	--	--	--	<0.05	<0.25

[a]Several results from this event were qualified because runoff sample results were less than two times the concentration measured in the field blank.

[b]One result from this event was qualified because runoff sample result was less than two times the concentration measured in the field blank.

[c]Two blanks were collected and analyzed on this date.

baseline 2 and post-manure application periods (fig. 5) for raw (orange shading; plots 4, 6, and 8) and treated (gray shading; plots 3, 5, and 7) manure plots. Manure was not applied to plots 1 and 2, and they are not included in any subsequent data analysis. Flows from plot 1 tile (sampler 1) were substantially higher than those from the other tile samplers (data not shown). The sampler for plot 2 surface (sampler 4) often failed to trigger and (or) insufficient water was collected. Investigators were unable to identify the causes of atypical volumes for samplers 1 and 4.

Dissolved Reactive and Total Phosphorus Concentrations

Dissolved reactive phosphorus was detected above the reporting limit (0.013 mg/L) in all surface runoff samples and in 58.6 and 75.2 percent of baseline and post-manure tile flow samples, respectively (fig. 5*A*). In two tile samplers, baseline median concentrations were less than the reporting limit (plots 3 and 7). In surface runoff samples, the four highest concentrations were from plot 8 (2.05–2.16 mg/L). In tile flow samples, the two highest concentrations were from plots 8 and 6 (0.489 and 0.343 mg/L, respectively). Concentrations of dissolved reactive phosphorus in tile flow samples from raw manure plots (plots 4, 6, and 8) post-manure application were noticeably higher than those in those same plots during

Table 4. Summary statistics for quality-control split replicates for nutrient samples collected and analyzed by Bowling Green State University or the U.S. Geological Survey, 2015–17.

[mg/L, milligram per liter; RPD, relative percent difference; <, less than; %, percent]

Constituent	Reporting limit (mg/L)	Number of sample pairs	Number of samples with at least one detection	Range of differences (mg/L)	Median RPD	Maximum RPD
Bowling Green State University						
Ammonia nitrogen	<0.29, <0.053	35	5	0–0.013	0.5%	4.5%
Nitrate plus nitrite nitrogen	<0.31	38	38	0–4	0%	29.8%
Dissolved reactive phosphorus	<0.013	35	20	0–0.036	1.3%	65.1%
U.S. Geological Survey						
Total phosphorus	<0.004	21	21	0–1.36	3.8%	61.8%
Total nitrogen	<0.05	21	21	0–3.4	3.8%	25.8%

the baseline period; this difference was not evident for surface runoff samples. In addition, concentrations of dissolved reactive phosphorus were generally an order of magnitude higher in surface runoff than in tile flow samples.

Total phosphorus was detected above the BGSU reporting limit (0.01 mg/L) in all surface runoff samples, all post-manure tile flow samples, and in 94.9 percent of baseline tile flow samples (fig. 5B). The highest median total phosphorus concentrations were measured in surface runoff during the post-manure period. Concentrations of total phosphorus in samples when detected ranged from 0.01 to 12.8 mg/L.

Nitrate Plus Nitrite and Total Nitrogen Concentrations

Nitrate plus nitrite was detected above the reporting limit (0.31 mg/L) in all samples (fig. 5C). The lowest concentration (0.32 mg/L) was measured in three surface runoff samplers post-manure application (plots 6, 8, and 7). The five highest concentrations (54–77 mg/L) were measured in tile flow from plot 8 (raw manure) post-manure application. Wide ranges of nitrate plus nitrite concentrations were measured in baseline surface runoff samples and in post-manure tile and surface runoff samples; however, relatively narrow concentration ranges were measured in baseline tile flow samples.

Total nitrogen was detected above the reporting limit (0.05 mg/L) in all samples (fig. 5D); the lowest concentration (1.49 mg/L) was from plot 8 (surface, raw manure) during the post-manure application period. The five highest concentrations (65.1–77.2 mg/L) were measured in tile flow from plot 8, the same plot where the highest nitrate plus nitrite concentrations were found. As was also found for nitrate plus nitrite, wide ranges of total nitrogen concentrations were found in post-manure tile and surface runoff samples and in baseline surface runoff samples. Relatively narrow concentration ranges of total nitrogen were found in baseline tile flow samples.

Ammonia Concentrations

During baseline 2 events, there were two reporting limits for ammonia—0.29 mg/L before April 2017 and 0.05 mg/L thereafter (table 2; fig. 5E). This change occurred before the last two baseline events (B2.8 and B2.9); hence, some of the baseline ammonia data points are below the higher reporting limit. Ammonia was detected in 14.2 and 67.0 percent of baseline and post-manure surface runoff samples, respectively, due to the lower reporting limit of the latter. In tile flow samples, ammonia was not detected in any baseline samples but was detected in 19.0 percent of post-manure samples. When ammonia was detected, concentrations ranged from 0.05 to 2.6 mg/L.

Water Volumes

Volumes of surface runoff and tile flow in plots 3−8 are reported for 6 baseline 2 (B2.4–B2.9) and 9 post-manure events (table 2.1) and summarized in boxplots (fig. 6). The highest median volumes were found during the baseline period in tile samplers because of a large event in May 2017 (B2.9) when the ground was saturated (fig. 6). During the post-manure period, volumes of surface runoff in plots with raw manure (orange background) and treated manure (gray background) were similar, with median volumes ranging from 170 to 500 L. For tile flow, median volumes ranged from 240 to 940 L with the highest median and maximum volumes coming from two of the treated manure plots (plots 3 and 5). None of the post-manure volumes were significantly different by plot, as determined by the Kruskal-Wallis test (p-value=0.46 for tile; p-value=0.95 for surface) or by manure treatment type, as determined by the Wilcoxon rank-sum test (p-value=0.10 for tile; p-value=0.76 for surface). The volume data from plots 3−8 collected during the post-manure period were used to calculate loads and flow-weighted mean concentrations of surface runoff and tile flow to compare treated manure to raw manure plots.

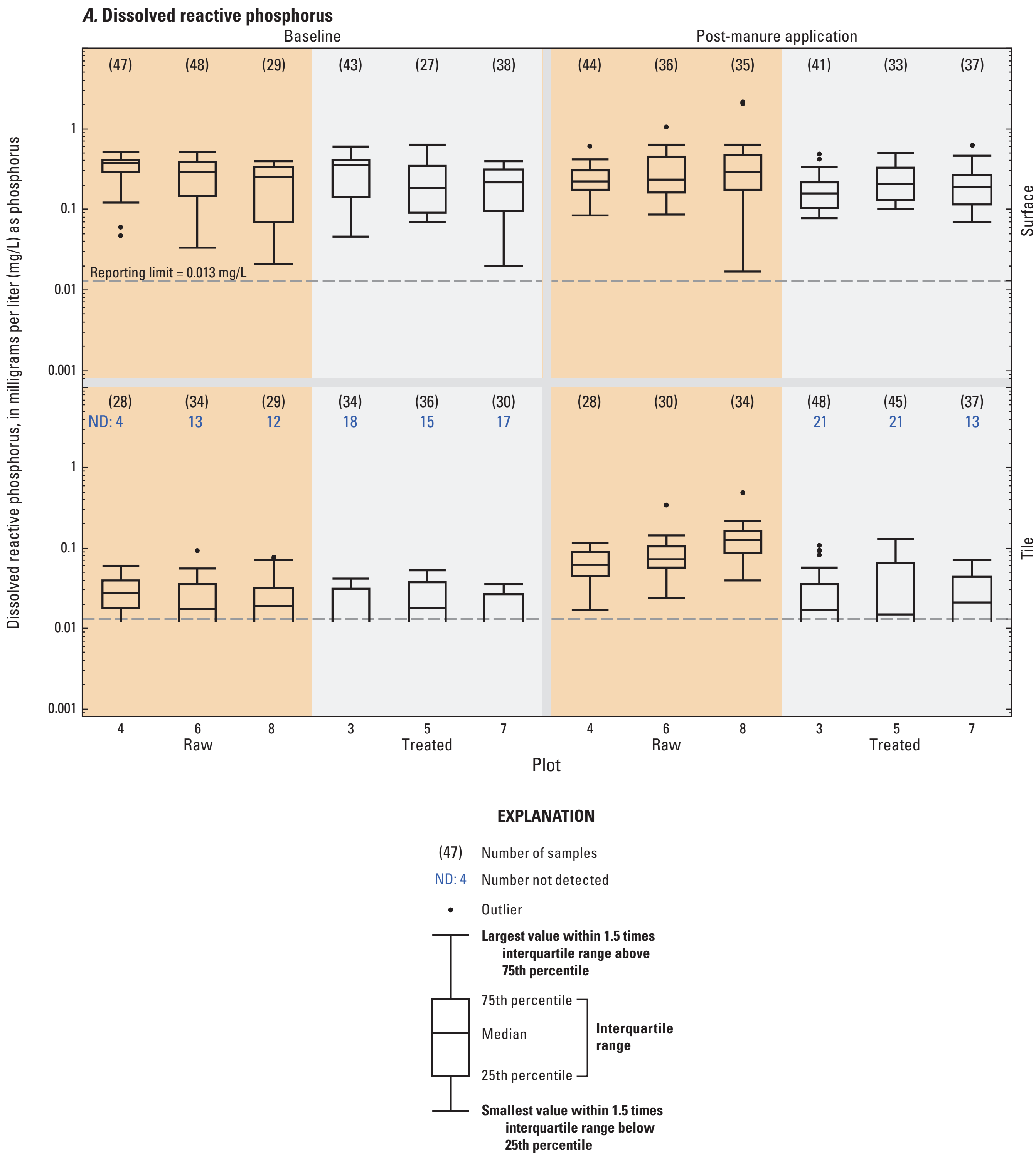

Figure 5. Concentrations of nutrients in samples from surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during baseline 2 and post-manure periods in 2016–17. *A*, dissolved reactive phosphorus. *B*, total phosphorus. *C*, nitrate plus nitrite. *D*, total nitrogen. *E*, ammonia.

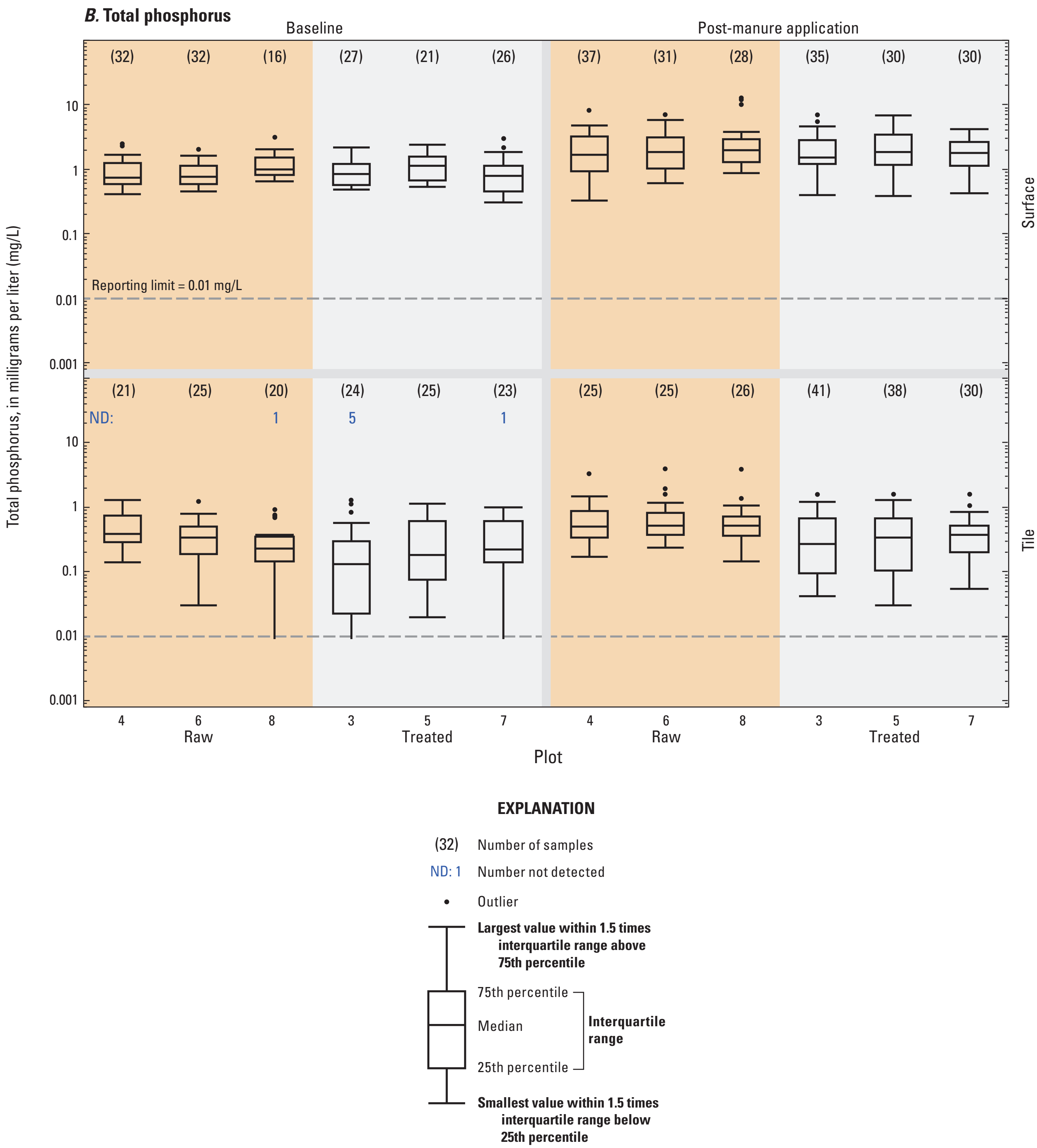

Figure 5. Concentrations of nutrients in samples from surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during baseline 2 and post-manure periods in 2016–17. *A*, dissolved reactive phosphorus. *B*, total phosphorus. *C*, nitrate plus nitrite. *D*, total nitrogen. *E*, ammonia.—Continued

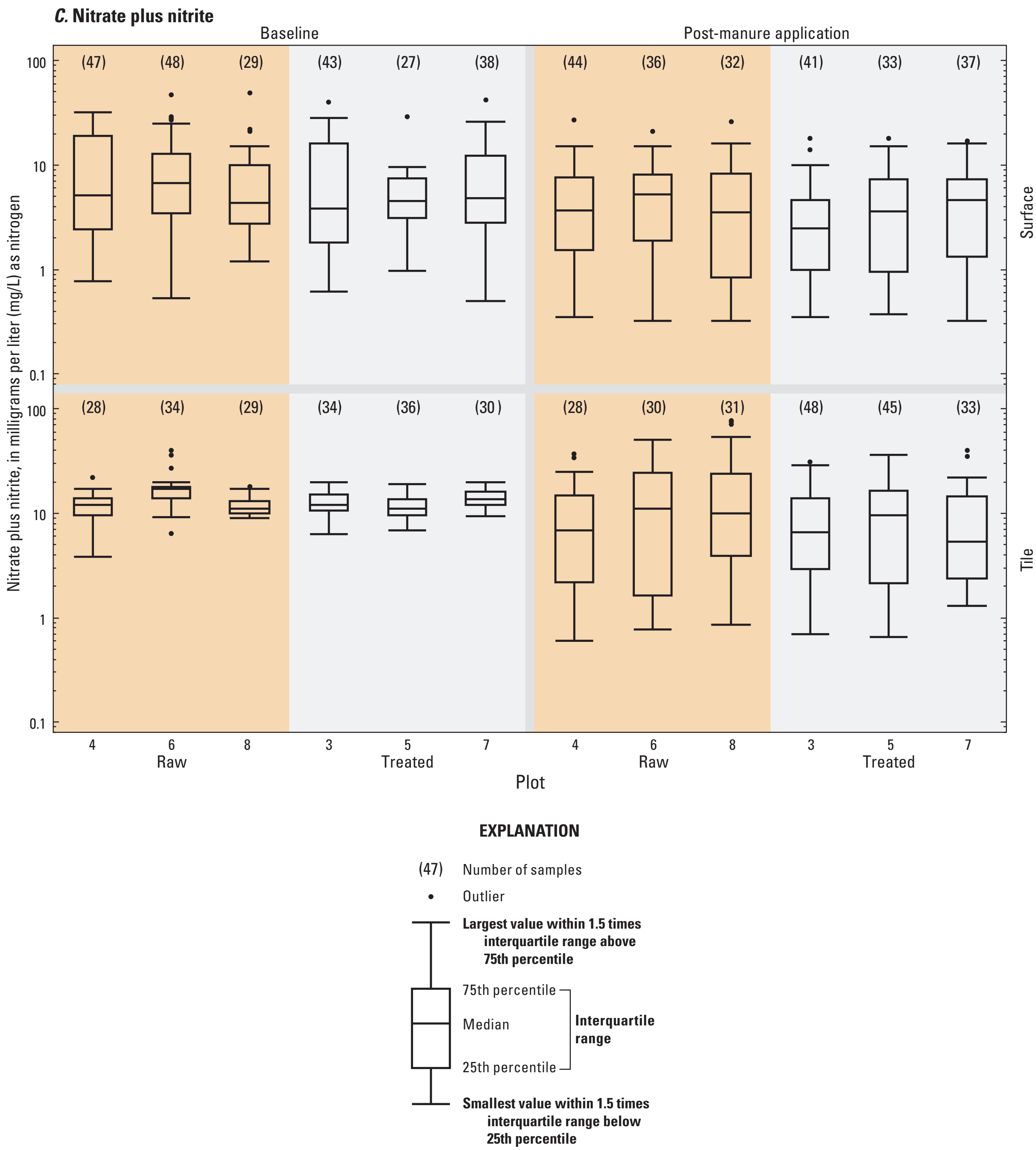

Figure 5. Concentrations of nutrients in samples from surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during baseline 2 and post-manure periods in 2016–17. *A*, dissolved reactive phosphorus. *B*, total phosphorus. *C*, nitrate plus nitrite. *D*, total nitrogen. *E*, ammonia.—Continued

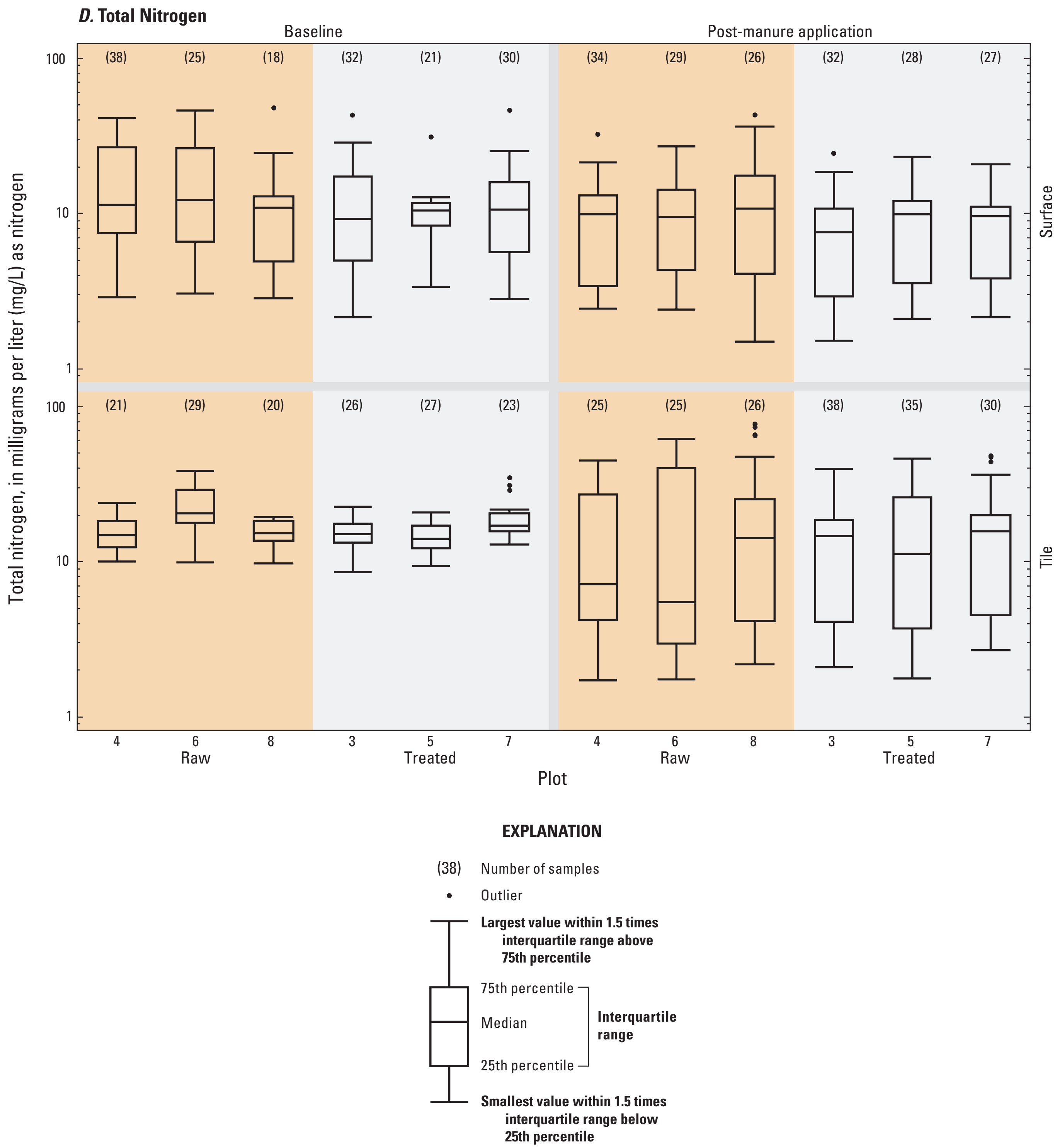

Figure 5. Concentrations of nutrients in samples from surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during baseline 2 and post-manure periods in 2016–17. *A*, dissolved reactive phosphorus. *B*, total phosphorus. *C*, nitrate plus nitrite. *D*, total nitrogen. *E*, ammonia.—Continued

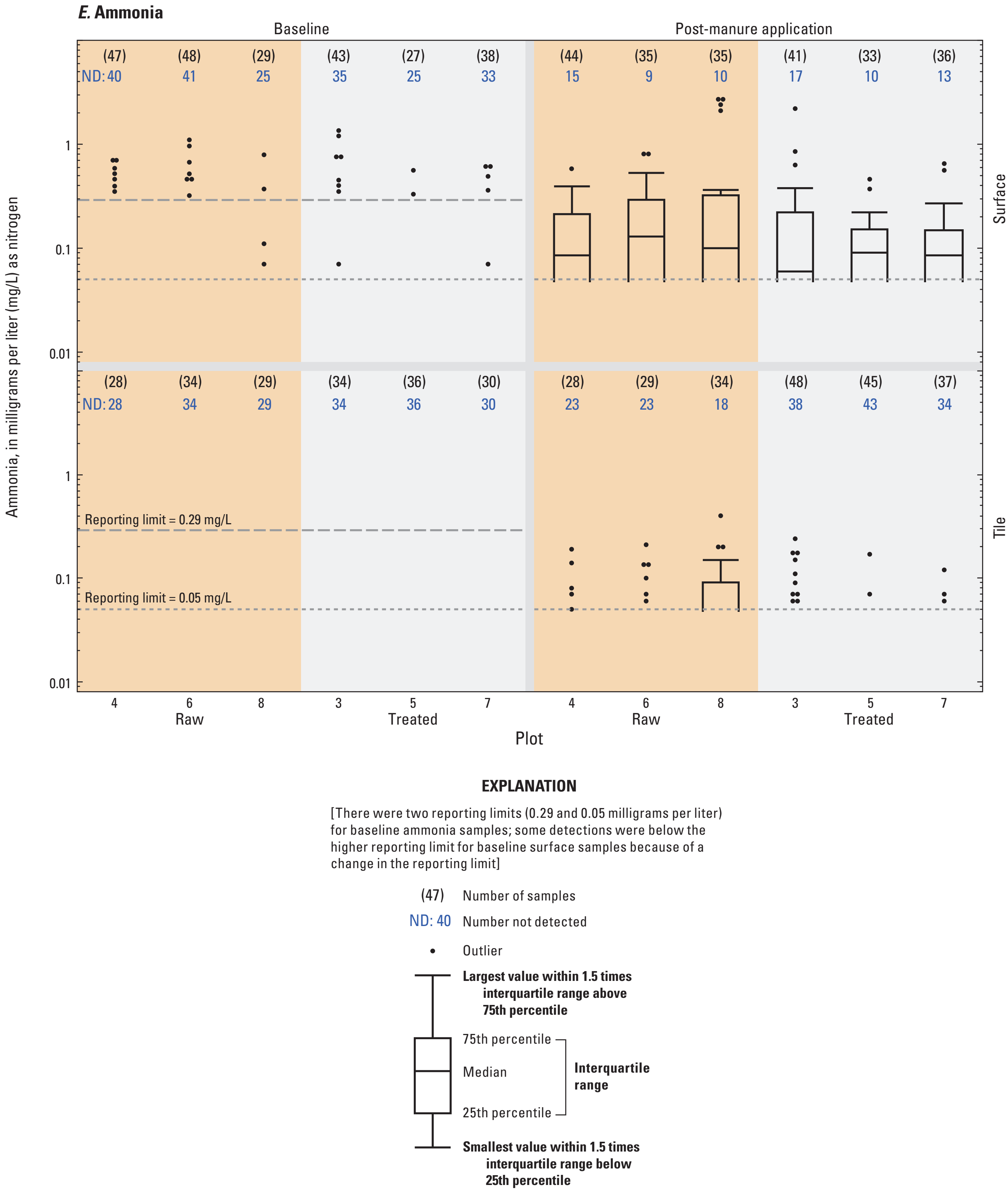

Figure 5. Concentrations of nutrients in samples from surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during baseline 2 and post-manure periods in 2016–17. *A*, dissolved reactive phosphorus. *B*, total phosphorus. *C*, nitrate plus nitrite. *D*, total nitrogen. *E*, ammonia.—Continued

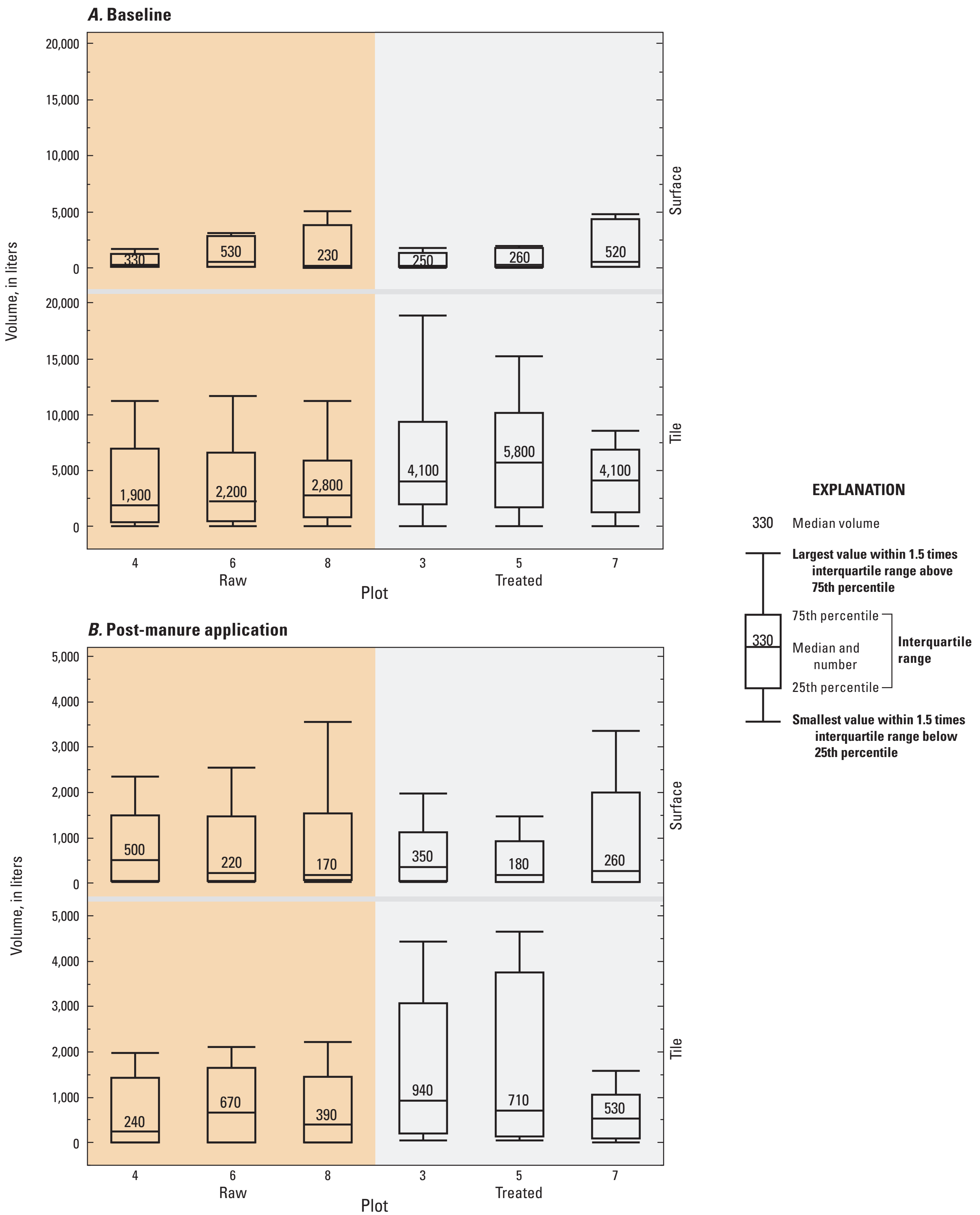

Figure 6. Total volumes of surface runoff and tile flow from plots with raw manure and treated manure at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station in 2016–17. *A*, six baseline rainfall events. *B*, nine post-manure application rainfall events.

Comparisons of Nutrient Loads and Flow-Weighted Mean Concentrations from Raw Manure and Treated Manure Plots

Concentrations of nutrients alone do not reflect the true effects of different manure treatments. Instead, post-manure loads and flow-weighted mean concentrations of dissolved reactive phosphorus, total phosphorus, nitrate plus nitrite, and total nitrogen were used to quantify amounts of nutrients that may be released from the manure and compare treated manure to raw manure plots. Ammonia was not included because of the large number of samples below the minimum reporting limit that led to incomplete load and flow-weighted mean concentration estimates. Nutrient loading provides information on the quantity of nutrients entering a system during a given period; in this case, loads were calculated per event. Whereas loads provide data on amounts of nutrients per event, flow-weighted mean concentrations provide a measure that normalizes constituent delivery values. Flow-weighted mean concentrations were calculated to account for differences in flow among the plots during the same event and in the same plot for different rain events. For example, if two plots had similar constituent loads during a rain event but one of the plots had a greater flow volume, the plot with greater flow will have more dilution and, therefore, lower constituent concentrations than the plot with lower flow volume. Therefore, to compare the relative water quality of different plots across the entire pilot test period, a flow-weighted mean concentration was calculated for each rain event by dividing the plot load by the flow volume.

Loads

Loads were computed for four constituents in 12 samplers from 3 raw manure and 3 treated manure plots for each of the 9 post-manure events (table 2.2). The nine loads were summed for each plot and presented in separate bars for surface runoff and tile flow (fig. 7). Combined loads (surface and tile loads added together) are presented in figures 8 and 9.

Loads from Surface Runoff and Tile Flow

Loads of four constituents are shown in bar graphs to qualitatively compare contributions between surface runoff and tile flow (fig. 7). For dissolved reactive phosphorus and total phosphorus, higher loads resulted from surface runoff (black bars) than from tile flow (gray bars); this was especially evident in plot 7 (figs. 7*A*–*B*). In contrast, for nitrate plus nitrite and for total nitrogen, higher loads resulted from tile flow than from surface runoff (fig. 7*C*–*D*). This is consistent with the different fate and transport mechanisms for phosphorus and nitrogen constituents. Nitrate is widely found in groundwater, due to its high solubility, mobility, and easy displacement by water (Follett, 1995a). Phosphorus in tile flow is lower than in surface runoff because phosphorus is sorbed onto soils from infiltrating water as it moves through phosphorus-deficient subsoils (Follett, 1995b).

Combined Loads

Combined loads (surface and tile added together) for nine post-manure events are shown in step graphs by date for each nutrient constituent (fig. 8). Loads from raw manure plots are indicated with solid lines and those from treated manure plots are indicated with dotted lines; the nine post-manure events are indicated with gray vertical lines. There was a long period with little-to-no surface runoff or tile flow from August through November 2017, indicated by long horizontal lines between PM.8 and PM.9 events. For dissolved reactive phosphorus (fig. 8*A*), combined loads from raw manure plots were consistently higher than those from treated manure plots. Two of the raw manure plots (plots 6 and 8) contributed the highest loads of nitrate plus nitrite (fig. 8*C*). In contrast, for total phosphorus and total nitrogen, load contributions were similar for raw manure and treated manure plots (figs. 8*B* and 8*D*). For both dissolved reactive phosphorus and total phosphorus, a large proportion of the loading came from the later events between July 10 and November 9, 2017 (PM.7, 8, and 9). In contrast, for nitrate plus nitrite and total nitrogen, the loads increased by more even increments than phosphorus throughout the season.

Combined loads are shown in bar graphs to qualitatively and statistically compare contributions between treated manure and raw manure plots; summed loads for each group of three plots by treatment are included above the bars (fig. 9). For dissolved reactive phosphorus, total phosphorus, and nitrate plus nitrite, summed event loads were higher for raw manure than treated manure plots; the opposite was found for total nitrogen. The RPDs for summed raw minus summed treated loads were as follows: dissolved reactive phosphorus, 34 percent; total phosphorus, 1.3 percent, nitrate plus nitrite, 7.8 percent; and total nitrogen, −6.5 percent.

For statistical analysis, loads from the three plots for each manure treatment type were summed for each event. The Wilcoxon signed-rank test was used to evaluate the differences in summed loads for the nine events. A significant difference between the manure treatment types was found for dissolved reactive phosphorus (p-value<0.01) but not for total phosphorus (p-value=0.50), nitrate plus nitrite (p-value=0.43), or total nitrogen (p-value=0.36). Calculations were done to ensure that the significant difference in dissolved reactive phosphorus loads between raw manure and treated manure plots was valid, given that somewhat disparate amounts of manure were applied to the two types of plots (table 5). Though the initial amounts of phosphorus applied to the three plots receiving raw manure (2,900,000 mg) were higher than those receiving treated manure (2,500,000 mg), the percentage of total phosphorus coming off the plots as dissolved reactive phosphorus was higher from raw manure plots (0.26 percent) than the percentage that came off the treated manure plots (0.22 percent). The opposite, however, was found for total phosphorus (table 5).

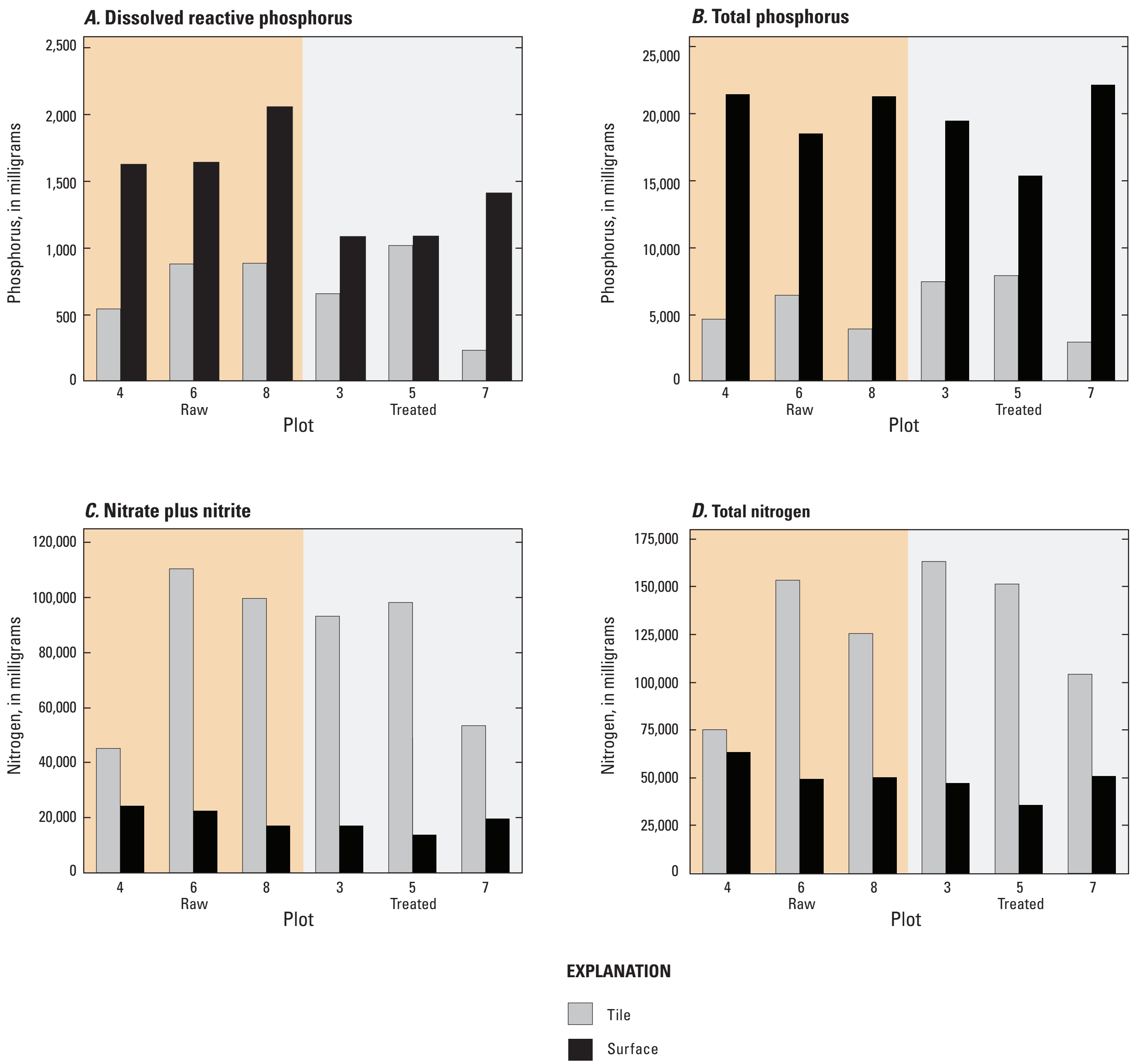

Figure 7. Loads in surface runoff and tile flow from plots with raw manure and treated manure at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during nine post-manure application rainfall events in 2017. *A*, dissolved reactive phosphorus; *B*, total phosphorus; *C*, nitrate plus nitrite; *D*, total nitrogen.

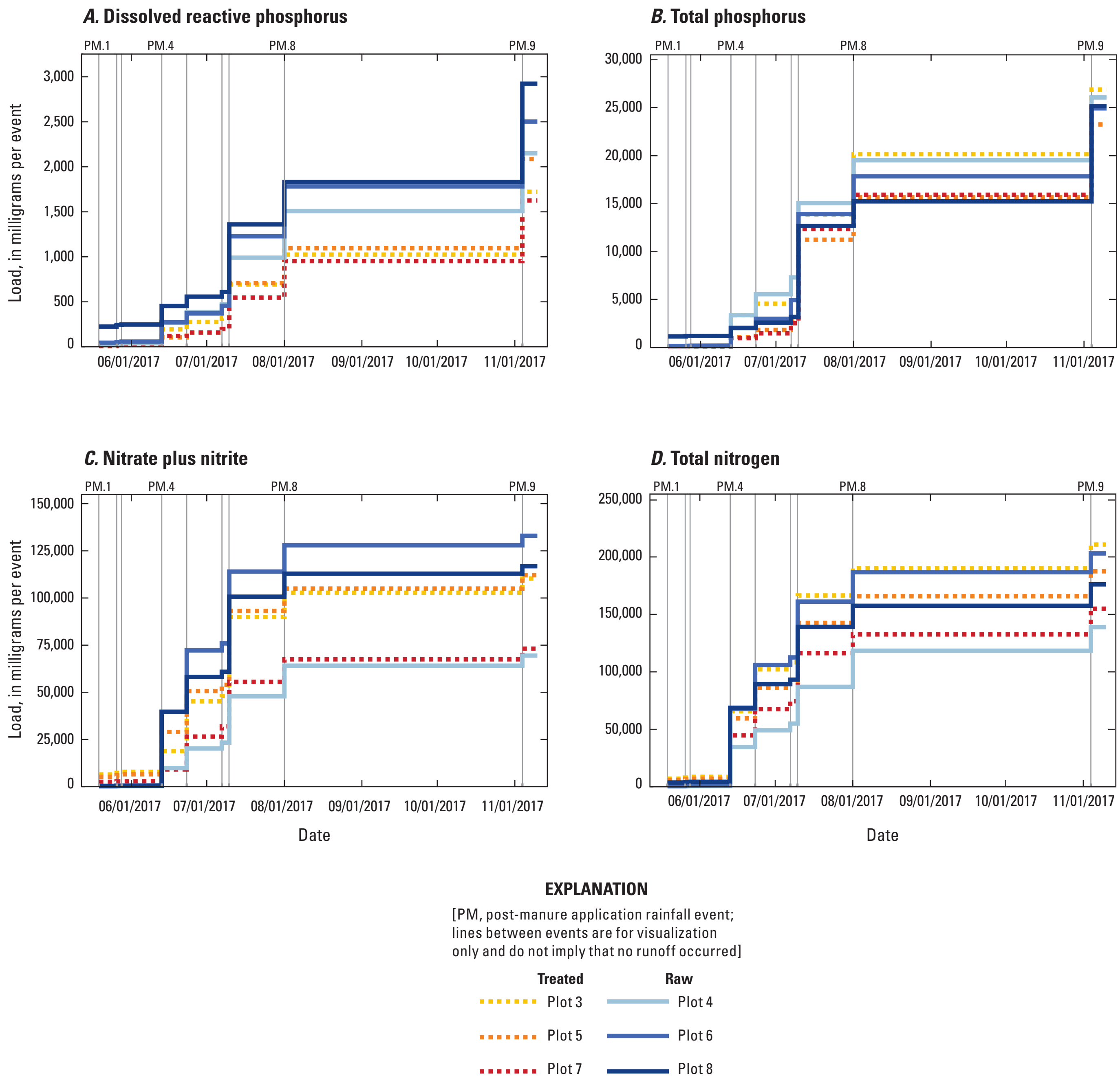

Figure 8. Step graphs by date for combined loads (surface runoff and tile flow) in samples from plots with raw manure and treated manure at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during nine post-manure application rainfall events in 2017. *A,* dissolved reactive phosphorus; *B,* total phosphorus; *C*, nitrate plus nitrite: *D,* total nitrogen.

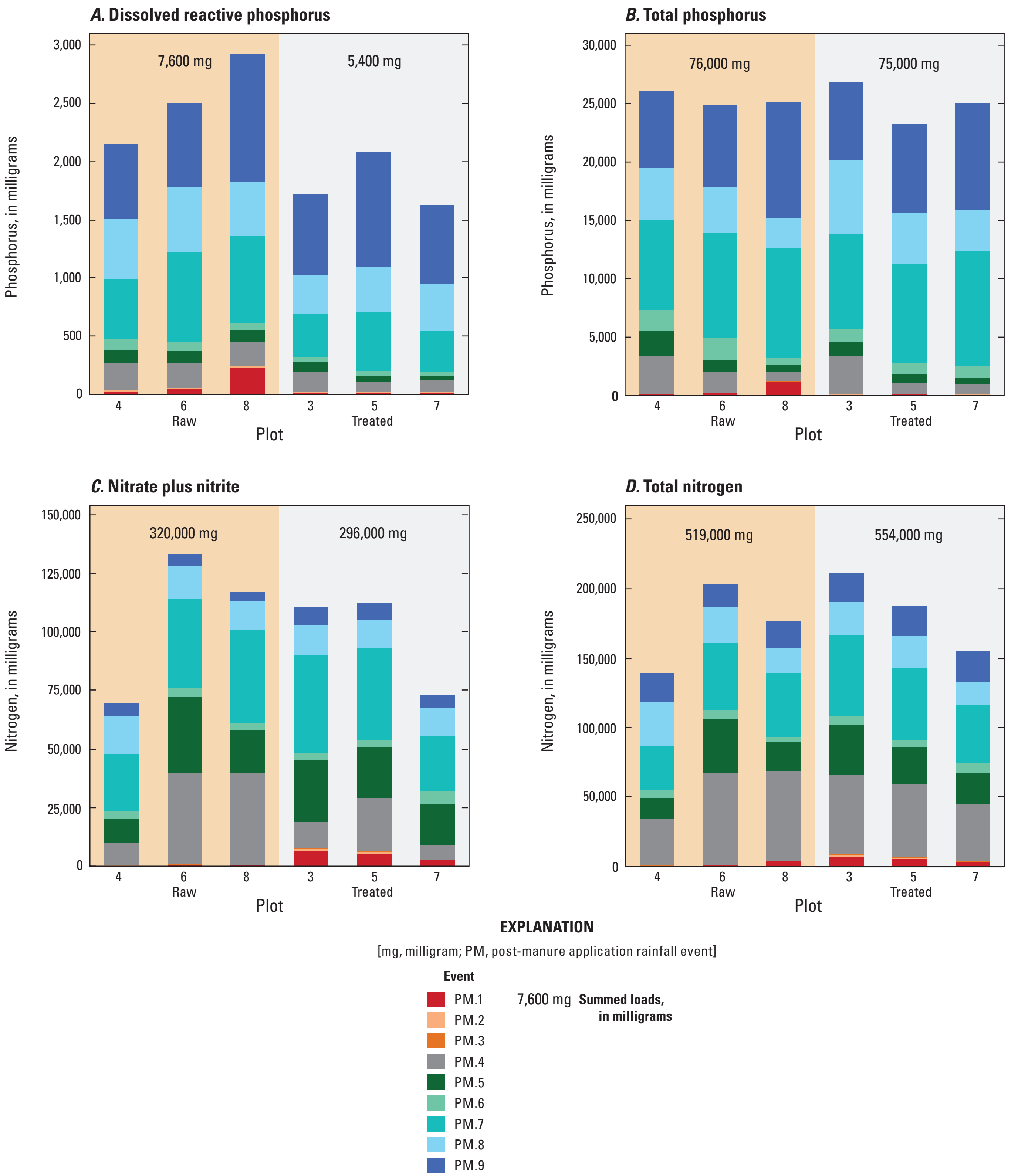

Figure 9. Combined loads (surface runoff and tile flow) in samples from plots with raw manure and treated manure at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during nine post-manure application rainfall events (PM.1–PM.9) in 2017. *A*, dissolved reactive phosphorus; *B*, total phosphorus; *C*, nitrate plus nitrite; *D*, total nitrogen.

Table 5. Total phosphorus applied to 3 raw and 3 treated manure plots and percentages of dissolved reactive phosphorus and total phosphorus in combined surface runoff and tile flow.

Type of application or load	Raw manure	Treated manure
Total phosphorus applied to each plot, in milligrams	970,000	830,000
Total phosphorus applied to three plots, in milligrams	2,900,000	2,500,000
Dissolved reactive phosphorus load from three plots, in milligrams of phosphorus	7,600	5,400
Total phosphorus applied that was measured in dissolved reactive phosphorus load from three plots, in percent	0.26	0.22
Total phosphorus load from three plots, in milligrams of phosphorus	76,000	75,000
Total phosphorus applied that was measured in total phosphorus load from three plots, in percent	2.60	3.00

Flow-Weighted Mean Concentrations

Flow-weighted mean concentrations for each nutrient constituent were computed for surface runoff or tile flow (table 2.3) and for combined surface runoff and tile flow (table 2.4) for each of the nine post-manure events. Combined (surface runoff and tile flow) values for the three raw manure plots (orange shading) and three treated manure plots (gray shading) are shown in boxplots (fig. 10). The *p*-values shown on the graphs result from Kruskal-Wallis multiple comparison tests for differences in flow-weighted means by plot, and Wilcoxon rank-sum tests for differences in flow-weighted means by manure treatment (raw versus treated). For examination by plot, flow-weighted means (number [*n*]=9) for each of the six plots were compared; for examination by treatment, flow-weighted means (n=27) for the three raw manure plots were compared to flow-weighted means (n=27) for the three treated manure plots. Dissolved reactive phosphorus was the only constituent where there was a significant difference (p-value<0.0001) in flow-weighted mean concentrations by plot. The letters at the top of figure 10*A* are used to show the results of a Dunn's multiple comparison test for all possible pairs of test plots. Test plots that share the same letter have sample populations that are not significantly different at the α=0.05 level of significance. Two treated manure plots (plots 3 and 5) had significantly lower medians than all three of the raw manure plots. Flow-weighted mean concentrations of dissolved reactive phosphorus and total phosphorus were significantly different (p-value<0.0001 and p-value<0.01, respectively) by manure treatment type (figs. 10*A* and 10*B*). Flow-weighted mean concentrations of nitrate plus nitrite (fig. 10*C*) and total nitrogen (fig. 10*D*) were not significantly different by treatment type.

Corn Yields

After the last event in November 2017, the corn was harvested and corn yields from the test plots were calculated. Based on the weight of corn obtained from the two rows harvested, a yield of bushels per acre was calculated (table 6). Mean yields from plots with treated manure was 3 percent lower than plots treated with raw manure, and yield from plots with no manure was 10 percent lower than plots treated with raw manure. Statistical analysis of yield differences was not possible because of the small number of data points; however, 3 percent is likely within the range of random variation, whereas 10 percent may exceed that random variation.

Table 6. Corn yields from Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station test plots, November 2017.

Treatment	Plot	Yield, in bushels per acre[a]	Mean yield
Control/no manure	1	126	132
	2	139	
Treated manure	3	140	141
	5	128	
	7	156	
Raw manure	4	134	145
	6	155	
	8	148	

[a]Harvested corn weight was corrected for moisture content above 15 percent.

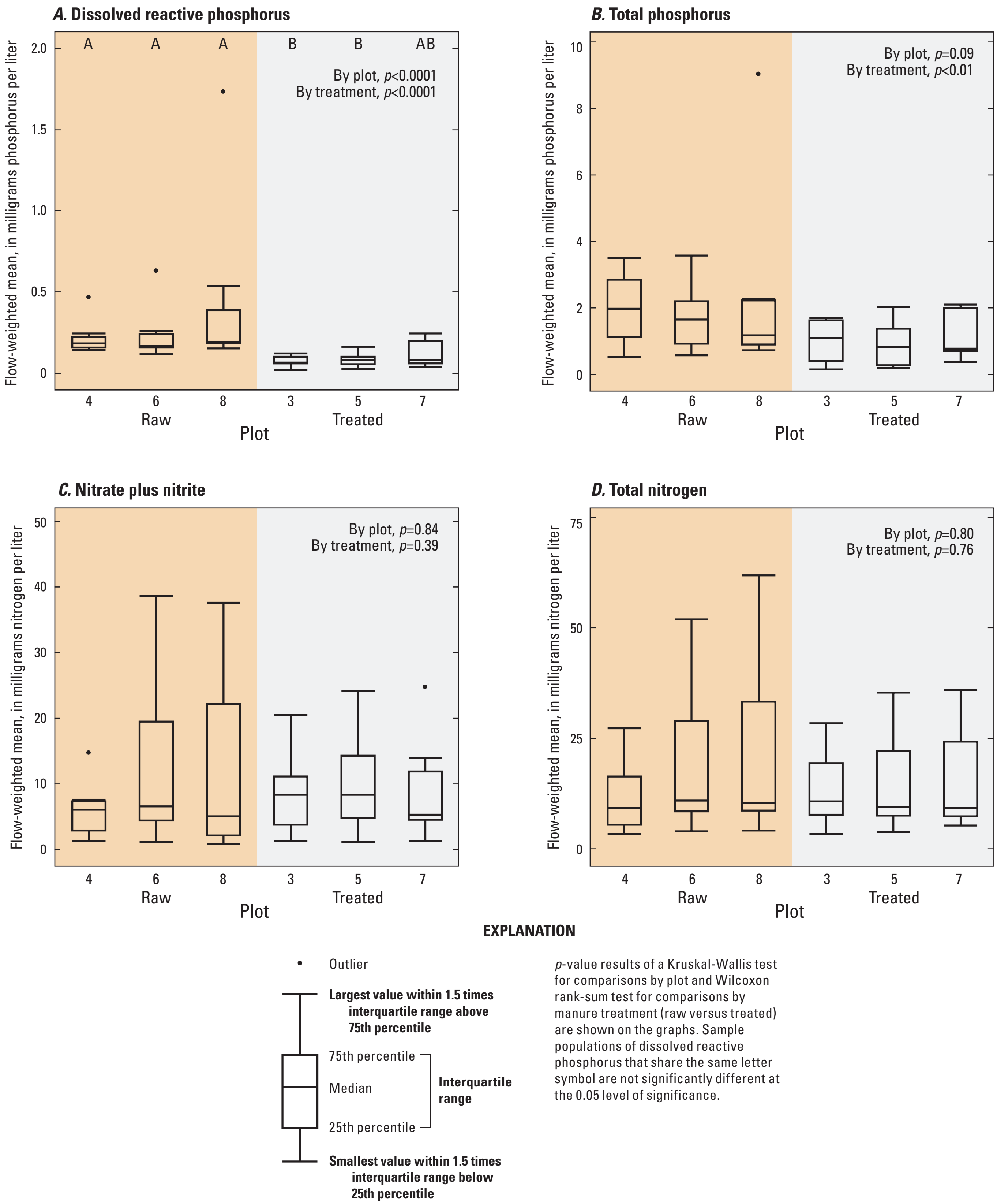

Figure 10. Combined flow-weighted mean concentrations (surface runoff and tile flow) in samples from plots with raw manure and treated manure at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station during nine post-manure application rainfall events (PM.1–PM.9) in 2017. *A*, dissolved reactive phosphorus; *B*, total phosphorus; *C*, nitrate plus nitrite; *D*, total nitrogen.

Summary and Conclusions

Nutrient excesses from large livestock operations are a growing concern because of the potential to cause eutrophication of surface waters from runoff and permeate soils to reach groundwater resources. In the Western Lake Erie Basin where there is a high density of animal agriculture, phosphorus loading to Lake Erie from dairy manure and fertilizer applications is a likely contributor to harmful algal blooms (HABs). One option to help alleviate nutrient loads is to treat manure to reduce the water content and slow and (or) reduce the release of nutrients when applied to soils. Manure treatment may enable more economical transport to fields for application and slow the transport of nutrients to surface water and groundwater. A promising technology, tested during this study, is solids-liquid separation enhanced with iron and aluminum salts with the addition of organic polymers.

A pilot-scale field study at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station was done to estimate the performance of treated manure (coagulant/polymer mixture) relative to liquid lagoon (raw) manure in a natural field setting. Samples were collected and analyzed for concentrations of nitrogen and phosphorus constituents that resulted from surface runoff and tile flow from pilot-scale test plots. Tipping buckets and refrigerated automatic samplers contained in a central building were used for collection of volume and water-quality data. Treated manure was applied to three plots and raw manure to another three plots. Data were collected during rainfall events that resulted in substantial surface runoff and (or) tile flow—13 baseline events and 9 events after manure application (post-manure events). The first four baseline events (baseline 1) were used to establish protocols and collect initial quality-control samples. Samples from the next 9 baseline events (baseline 2) and 9 post-manure events were used to report nutrient concentrations from the 6 plots; samples from 6 baseline and all post-manure events were used to report flow volumes. Loads and flow-weighted mean concentrations were calculated and used to compare surface runoff and tile flow from three treated manure to three raw manure test plots during the nine post-manure events.

Samples were retrieved from the automatic samplers and analyzed for dissolved reactive phosphorus, total phosphorus, nitrate plus nitrite, total nitrogen, and ammonia. To characterize bias and variability, quality-control samples were collected and analyzed throughout the sampling period. Concentrations for baseline 2 and post-manure events were as follows: Dissolved reactive phosphorus, less than (<) 0.013 to 2.16 milligrams per liter (mg/L); total phosphorus <0.01 to 12.8 mg/L; nitrate plus nitrite, 0.32 to 77 mg/L; total nitrogen, 1.49 to 77 mg/L; and ammonia, <0.05 to 2.6 mg/L.

Water volumes in surface runoff and tile flow were reported from six baseline 2 and nine post-manure events. There were no significant differences in volumes of surface runoff or tile flow by plot or by manure treatment type for the nine post-manure events. Baseline 2 volumes were not used for statistical tests. The volumes measured during the post-manure period were used to calculate loads and flow-weighted mean concentrations of dissolved reactive phosphorus, nitrate plus nitrite, total phosphorus, and total nitrogen; these were used to compare flow from treated manure plots to flow from raw manure plots. Ammonia was not included because of the large number of samples below the minimum reporting limit.

For nitrogen during the post-manure period, higher loads resulted from tile than surface runoff. For phosphorus, the opposite was true in that higher loads resulted from surface runoff than tile flow. This is consistent with a different fate and transport mechanism for phosphorus and nitrogen constituents. Combined loads (surface and tile) were used in subsequent data analysis. In terms of timing of combined loads, a large proportion of the phosphorus loading came from the last three events. For nitrogen, however, the loads increased by more even increments than phosphorus throughout the season. Combined loads of dissolved reactive phosphorus were significantly different by manure treatment type; loads were higher for the raw manure treated plots than for the treated manure plots. Combined loads of total phosphorus, nitrate plus nitrite, or total nitrogen were not significantly different by manure treatment type.

Combined (surface runoff and tile flow) flow-weighted mean concentrations were also examined. Combined flow-weighted mean concentrations of dissolved reactive phosphorus were significantly different by plot and by manure treatment type. Flow-weighted mean concentrations of total phosphorus were significantly different by treatment type but not by plot. Flow-weighted mean concentrations of total nitrogen and nitrate plus nitrite were not significantly different by plot or by treatment type.

The purpose of treating the liquid dairy manure with a coagulant and cationic polymer was to dewater the manure and sequester nutrients in a solid matrix that retained more nutrients in soil, better matching the rate of assimilation by crops. In theory, this would reduce the amount of free nutrients in the soil, which is susceptible to hydrological transport. Degradation of the matrix in the soil releases nutrients throughout the growing season instead of it all being available at the time of application. Differences in loads and flow-weighted mean concentrations between raw manure and treated manure plots indicate that dissolved reactive phosphorus was likely retained in the soil and hydrological transport was reduced for the plots amended with the treated manure as compared to raw manure. HABs in the Western Lake Erie Basin are promoted more by dissolved phosphorus than total phosphorus so reduction in dissolved phosphorus transport may be more effective for reducing the risk of HAB formation. In addition, corn yields from plots with treated and raw manure were in a similar range. The use of this coagulant/polymer mixture, therefore, shows promise in helping to reduce dissolved forms of phosphorus loading into waters of the Western Lake Erie Basin and thus reducing the risk of HABs. Additional field tests are needed to reproduce and confirm these results and to investigate the transportation cost savings that the treatment provides; these studies are in progress. Future studies could focus on scaling-up testing from the pilot to the full-scale field level.

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Appendix 1. Sample Processing Cheat Sheet

This document was used in the field and served as concise step-by-step instructions for collecting and processing samples from 16 automatic samplers at The Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17. Samples are processed within 72 hours of automatic sampler collection.

1. Label bags and USGS sample bottles. Label a set of Whirl-pak bags for filtered nutrients and 125-mL clear polyethylene bottles for total nutrients with the sampler number and the order in the sampler (i.e. sampler 2, bottle 7 would be “2-7”).

2. Rinse bottles 2X with deionized water (DIW). The labeled USGS bottles, if not pre-rinsed, are rinsed 2 times with DIW.

3. Total nutrients. Shake the 1-L bottle. Pour a small amount of sample into the WCA bottle to rinse the bottle. Shake the 1-L bottle again and fill the WCA bottle to the shoulder. Acidity with 1 mL 4.5 N H_2SO_4 and place the acid sticker on the WCA bottle. Chill the WCA bottle.

4. Filtered nutrients. Process the filtered sample. Remove 20 mL using a syringe, insert a filter into the Luer-lock, and filter the sample into a Whirl-pak bag.

5. OPTIONAL USGS. Filtered nutrient analysis by the USGS is done for QC purposes or at the request of BGSU. Use a 125-mL brown polyethylene bottle that has been rinsed 2X with DIW. Using a syringe, remove approximately 20 mL from the 1-L bottle to rinse the syringe; discard the rinse. Remove a second 20-mL aliquot and insert a filter into the Luer-lock. Filter 5-10 mL through the filter into the brown bottle, rinse the bottle with this small amount of water, and discard the rinse water. Resume filtering and immediately chill the brown bottle. You need to provide at least 10–20 mL in the brown bottle.

6. Save water for pH and spC. From **each sampler** composite the water from two of the Isco bottles into a 500-mL bottle (staggered, for example bottles #1 and #7 OR #5 and #11). Measure the pH and specific conductance of each within 24 hours of sample collection.

7. Discard the water from the rest of the bottles.

8. USGS paperwork (next working day is OK). Write in the sample collection date/time and your initials on the pre-printed label. Some labels need the last two digits of the site ID. Fill out an Analytical Service Request (ASR) form. To a pre-printed surface or tile ASR, enter

   a The last two digits of the Station ID

   b Begin Date and Begin Time

   c To the Station Name, enter the plot #; to the Field ID, enter the Sampler #

   d Collected by, phone number, and date Shipped

   e Specific conductance and pH values (measure and write this on the field form also).

Make a copy of the ASR to include with the samples. Ship the samples by overnight mail on ice within 72-hours of collection.

National Water Quality Laboratory, Building 95, Entrance E3, Denver Federal Center, Denver, CO 80225-0046 Phone: 303-236-3707.

Appendix 2. Data Tables

Total volume, nutrient loads, and flow-weighted mean concentrations of nutrients for 12 samplers (6 plots) during 9 post-manure (PM) rainfall events that resulted in substantial surface runoff and (or) tile flow at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17, are provided in this appendix (tables 2.1–2.4, available for download at https://doi.org/10.3133/sir20205015).

Table 2.1. Total volume, in liters, for 12 samplers during 6 baseline 2 and 9 post-manure application rainfall events that resulted in substantial surface runoff or tile flow at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2015–17.

Table 2.2. Nutrient loads, in milligrams, for 12 samplers during 9 post-manure application rainfall events that resulted in substantial surface runoff or tile flow at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2017.

Table 2.3. Flow-weighted mean concentrations of nutrients, in milligrams per liter, for 12 samplers during 9 post-manure application rainfall events that resulted in substantial surface runoff or tile flow at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2017.

Table 2.4. Flow-weighted mean concentrations of nutrients, in milligrams per liter, for 6 plots during 9 post-manure application rainfall events that resulted in substantial surface runoff and tile flow (combined) at the Ohio State University Agricultural Research and Development Center Northwest Agricultural Research Station, 2017.

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