An Evaluation of Septic Effluent Presence and Spatial Distribution in the Lindsay Creek Watershed

State of Idaho
Department of Environmental Quality
January 2019

Acknowledgments

The Idaho Department of Environmental Quality (DEQ) thanks property owners who gave DEQ property access for sampling. Ed Marugg of Public Health – Idaho North Central District provided septic repair permit records. Bill Reynolds of Nez Perce County Assessor's Office provided county geographic information systems records. Daniel Sturgis of Idaho Department of Water Resources provided helpful information about local geology. King's Thrones provided assistance with septic sampling. Barney Metz of the Lewiston Orchards Irrigation District provided access for irrigation water sampling. Mark Ritari and Todd Taruscio at Anatek Labs analyzed septic indicator samples. At DEQ, Michael Camin, Hailey Shingler, and Jason Williams performed field sampling. Kathryn Elliot and Joanna Hahn assisted analysis of ground water nitrate data. Michael Camin, Hailey Shingler, and Sujata Connell provided input on study design and report contents. Hawk stone performed data validation. Brady Johnson reviewed project R code. Kathryn Elliot, John Cardwell, Graham Freeman, and Brady Johnson provided helpful comments on a draft version of the report.

Prepared by
Jason Williams
Idaho Department of Environmental Quality
Lewiston Regional Office
1118 F St
Lewiston, ID 83501

Table of Contents

Executive Summary vi
1 Introduction 1
2 Objectives 1
3 Watershed Description 2
4 Nitrate Concerns 6
5 Artificial Sweeteners and Caffeine as Septic Markers 7
6 Methods 8
6.1 Septic System Locations 8
6.2 Septic Indicator Sampling 9
6.3 Ground Water NO_3+NO_2-N and Escherichia coli 9
6.4 Stream Discharge and NO_3+NO_2-N 10
6.5 Analytical Methods 10
7 Results 11
7.1 Septic System Locations 11
7.2 Septic Indicator Sampling 11
7.3 Ground Water NO_3+NO_2-N and Escherichia coli 14
7.4 Stream Discharge and NO_3+NO_2-N 14
8 Conclusions 15
References 18
Appendix A. Quality Assurance/ Quality Control 23

List of Tables

Table 1. Sucralose, acesulfame, and caffeine removal efficiencies reported in the literature for wastewater treatment plants (WWTP), drinking water treatment plants (DWTP) and septic systems. 8
Table 2. Nitrate and septic indicator results from 9-10-2018 sampling. 13

List of Figures

Figure 1. Lindsay Creek watershed and administrative boundaries. 4
Figure 2. Water year stream flow and nitrate plus nitrite nitrogen concentrations at the Lindsay Creek mouth, and cumulative precipitation measured at the Lewiston Airport. 5

Figure 3. Geologic strata and average nitrate concentrations at wells and springs sampled by DEQ within the Lindsay Creek NPA and Lewiston plateau ground water management area (Figure 1). Geologic strata are from Neely 2018. 5
Figure 4. Sample sites, septic parcels, and parcels where PH-INCD issued a septic repair permit..... 13
Figure 5. 2018 NO_3+NO_2-N concentrations at shallow ground water and surface water monitoring sites. See figure 4 for sample locations. 14

Abbreviations, Acronyms, and Symbols

APHA	American Public Health Association
cfs	cubic feet per second
DEQ	Idaho Department of Environmental Quality
DWTP	drinking water treatment plant
E. coli	Escherichia coli
EPA	US Environmental Protection Agency
FDA	US Food and Drug Administration
FEP	fluorinated ethylene propylene
GIS	geographic information systems
GWMA	ground water management area
H_2SO_4	sulfuric acid
HDPE	high density polyethylene
HPLC	high pressure liquid chromatography
IDWR	Idaho Department of Water Resources
IDHW	Idaho Department of Health and Welfare
PH-INCD	Public Health – Idaho North Central District
IPDES	Idaho Pollution Discharge Elimination System
LOID	Lewiston Orchards Irrigation District
MBAS	methylene blue active substances
MSL	mean sea level
MS/MS	tandem mass spectrometer
NO_2	nitrite
NO_3	nitrate
NO_3+NO_2-N	nitrate plus nitrite nitrogen
NPA	nitrate priority area
PQL	practical quantitation limit
TMDL	total maximum daily load
WWTP	wastewater treatment plant

Executive Summary

Lindsay Creek is a tributary to the Clearwater River in Nez Perce County, Idaho. Its 22 square mile watershed has 72 percent agricultural land, increasing urban development, and elevated nitrate concentrations in surface water and in the shallow Saddle Mountains aquifer. Nitrate has contributed to nuisance algal growths in Lindsay Creek, exceeds concentrations associated with human health effects in some domestic wells, and some residents have stopped using shallow private wells as a drinking water source because of health concerns. Nearly 800 parcels in the watershed (~15% of parcels) have an individual on-site septic system, so individual on-site septic systems are one potential nitrate source. However, the presence and distribution of septic effluent in ground water and surface water, and its relationship to nitrate was not clear; previous efforts to test for septic inputs yielded inconclusive results. Septic systems, soil, fertilizers, livestock, and storm water discharges are all potential nitrate sources.

This study used artificial sweeteners and caffeine as markers of septic effluent in the Lindsay Creek Watershed. The Idaho Department of Environmental Quality (DEQ) tested for two artificial sweeteners and caffeine in ground water, surface water, and in one septic system. These septic indicator chemicals were used to evaluate the presence and spatial distribution of septic effluent, and its relationship to nitrate in the Lindsay Creek Watershed.

Artificial sweeteners acesulfame and sucralose, which are unique to human wastes and are not completely removed by septic systems, were detected at 4 of 5 stream sites; in 4 of 5 domestic wells; in 1 of 1 springs; and in 1 of 1 septic systems. Sweeteners were detected downstream of areas with high septic density, and not at locations with no suspected septic inputs. These patterns suggest acesulfame and sucralose are reliable indicators of septic effluent in the watershed, and indicate septic effluent is present in Lindsay Creek and the Saddle Mountains aquifer, including some domestic wells.

Caffeine was detected at two stream sites and one well drawing from the Saddle Mountains aquifer. Because caffeine is completely removed by most properly-functioning septic systems, caffeine detections indicate inputs from poorly-functioning septic systems at some locations. This conclusion is supported by local public health district records; caffeine was detected downstream of areas with a recent history of septic failures. Results suggest caffeine is a reliable marker for effluent from poorly-functioning septic systems in the Lindsay Creek watershed, and demonstrate such effluent has entered Lindsay Creek and the Saddle Mountains aquifer.

Nitrate concentrations were high (7.1-10.3 mg N/L) where septic indicators were detected, and much lower (< 1.14 mg N/L) where they were not. Patterns suggest septic effluent contributes to elevated nitrate concentrations observed in Lindsay Creek and in the Saddle Mountains aquifer. However, the relative (percent) contribution of septic effluent to nitrate contamination is beyond the scope of this study and merits further investigation.

1 Introduction

In rural areas of Idaho, many homes and buildings use on-site septic systems to treat wastewater. Septic systems treat wastewater generated from bathroom, laundry, kitchen, and cleaning activities. Septic systems typically include a one or two-chamber underground septic tank and a drainfield with underground piping that extends from the septic tank into soil. Wastewater enters the septic tank, where solids settle to the bottom and are digested by anaerobic bacteria. Accumulated sludge must be pumped out periodically. Effluent from the septic tank is further purified in the drainfield by bacteria, chemical adsorption to soil particles, and various chemical reactions. Properly designed, located, constructed, and maintained septic systems remove or greatly reduce concentrations of many chemicals by the time wastewater exits the drainfield.

By design, septic systems discharge treated wastewater to ground water, and therefore are a potential source of nutrients, bacteria, and other pollutants to ground water and surface water. If they are not properly designed, installed, or maintained, septic systems can discharge water with elevated concentrations of nutrients, bacteria, and other pollutants. In addition, as development and septic system density increases, the collective output from many properly functioning septic systems can become a significant source of pollutants to ground water or surface water.

Nitrate is one chemical of concern discharged by septic systems. Nitrate is a form of nitrogen, an element essential for plant and animal growth. When discharged into ground water, nitrate can enter wells people use as a drinking water source. Consuming water with nitrate-nitrogen concentrations greater than 10 mg N/L can increase health risks for infants, people of poor health, and the elderly. Infants younger than six months of age are especially sensitive to nitrate poisoning, which may result in serious illness or death. The illness occurs when nitrate (NO_3) is converted to nitrite (NO_2) in the body. Nitrite reduces the amount of oxygen in blood, causing shortness of breath and blueness of the skin (often called blue baby syndrome or methemoglobinemia). Adults in poor health and the elderly can also be susceptible to health problems from short-term nitrate exposure. In surface water, nitrate can also contribute to nuisance algal growths and affect aquatic life.

In addition to septic systems, nitrogen in soil, nitrogen-based fertilizers, waste from livestock and wildlife, and urban runoff are all common nonpoint sources of nitrate to ground water and surface water. Identifying the presence and relative contribution of different nitrate sources can help prioritize pollutant reduction efforts when limited resources are available. However, because there are many potential nitrate sources in most watersheds, identifying the presence or contribution of specific nitrate sources in ground water and surface water can be difficult.

2 Objectives

This study used artificial sweeteners and caffeine to evaluate the presence/absence, and spatial distribution of septic effluent in the Lindsay Creek Watershed, where nitrate concentrations are elevated in ground water and surface water, and septic systems are one of several potential nonpoint nitrate sources. This study had four objectives:

1. Test for the presence/absence of septic effluent in surface water and ground water in the Lindsay Creek watershed using artificial sweeteners acesulfame and sucralose.
2. Test for the presence/absence of effluent from poorly-functioning septic systems in surface water in the Lindsay Creek watershed using caffeine.
3. Use available information about septic system distribution and septic system repair permits issued by Public Health – Idaho North Central District to evaluate the reliability of acesulfame, sucralose, and caffeine as septic effluent markers in the Lindsay Creek watershed.
4. Document the spatial distribution of septic effluent and its relationship to NO_3+NO_2-N in surface water and ground water in the Lindsay Creek watershed.

3 Watershed Description

Lindsay Creek is a tributary to the Clearwater River in Nez Perce County, Idaho (Figure 1). The headwaters of Lindsay Creek begin within residential developments and farmland at approximately 1,800 feet above mean sea level (MSL). At its mouth (750 feet above MSL), Lindsay Creek flows through a tunnel drain in the Clearwater Levee and then into the Clearwater River. The main stem of Lindsay Creek is a third order stream with typical flows of 1-2 cubic feet per second (cfs) in summer and 5-7 cfs in spring at the mouth (Figure 2). Several first and second order tributary segments have lower flows and some tributary segments go dry during summer.

Water in Lindsay Creek comes from precipitation, ground water, and irrigation inputs. Average annual precipitation from 1981-2010 in Lewiston, ID was 12.31 inches (NOAA 2018). Precipitation occurs primarily during fall, winter, and spring, with very limited summer precipitation. Lindsay Creek receives ground water inputs from springs associated with basalt canyon wall rock fall and ground water seepage from basalt canyon walls. Ground water inputs are thought to be substantial, but percent ground water contribution to stream flow has not been quantified. A portion of the watershed also receives irrigation inputs. The Lewiston Orchards Irrigation District (LOID) conveys water from Sweetwater and Webb creeks into Mann Reservoir (also called ‘Reservoir A’, Figure 1). The water is delivered to the LOID service area through a pipe system; residents use it to water lawns, for winter livestock watering, and for fire protection. LOID water may enter ground water or surface water after being used within the LOID service area. Mann Reservoir does not discharge directly into Lindsay Creek, but a wetland near the reservoir outlet and Lindsay Creek headwaters suggests some limited under flow seepage may occur.

Land uses within the watershed include non-irrigated agriculture, small ranches and livestock operations, residential developments, and industrial businesses. Seventy-two percent of watershed area is non-irrigated agricultural land (USGS 2018). A 2002 watershed farming practices survey indicated crops included wheat, barley, legumes, alfalfa, and fallow (NPSWCD 2002). Approximately 18 percent of watershed area is developed, and 4 percent of the land surface is impervious material (USGS 2018). Portions of the City of Lewiston are served by the

city storm water system. Natural drainage ways in the Lewiston Orchards and within portions of the City of Lewiston convey ephemeral storm water into tributaries of Lindsay Creek. Most parcels within city boundaries are connected to the City of Lewiston sewer line system. Many city parcels in the Lewiston Orchards area and all parcels outside city boundaries use septic systems; approximately 800 parcels in the watershed (~15% of parcels) have septic systems. There are no known nitrate sources within the watershed that could be defined as a point source requiring an Idaho Pollution Discharge Elimination System (IPDES) discharge permit.

There are multiple aquifer systems below the Lindsay Creek watershed (Figure 3). The Saddle Mountains Aquifer spans 0-250 ft below the ground surface, and is composed of multiple layers of fractured basalt. The Saddle Mountains Aquifer is likely the primary source of springs discharging into Lindsay Creek. A sedimentary interbed called the Sweetwater formation sits below the Saddle Mountains Aquifer and separates it from a second lower fractured basalt aquifer, the Wanapum aquifer. The Saddle Mountains and Wanapum aquifers are perched aquifers thought to be recharged through precipitation and infiltration of water from the land surface (Neely 2018). The permeability of the Sweetewater formation and amount of downward hydrologic communication between the Saddle Mountains and Wanapum basalt aquifers is not clear (Daniel Sturgis, IDWR, personal communication, 11/14/18). At a depth greater than approximately 700 ft below the ground surface, there is a deep Grande Ronde regional aquifer within basalt flows of the Grande Ronde Formation (Ralston 2017). This aquifer is recharged primarily by the Snake River and Clearwater River and is the source of many regional municipal water supply wells (Ralston 2017).

The Saddle Mountains, Wanapum, and Grande Ronde aquifers are all used by Lindsay Creek watershed residents for drinking water and irrigation. Idaho Department of Water Resources (IDWR) documented declining water levels in wells drawing from the Saddle Mountains aquifer and subsequently designated the Lindsay Creek Ground Water Management Area (GWMA), which was later expanded and renamed the Lewiston Plateau GWMA. The GWMA requires that new wells be completed in the deep regional Grande Ronde aquifer, and requires well casing to seal off water from the Saddle Mountains and Wanapum aquifer (IDWR 2015, Daniel Sturgis IDWR, personal communication 1-14-2019). DEQ observed nitrate concentrations that exceed the U.S. Environmental Protection Agency's maximum contaminant level and Idaho ground water quality standard (10 mg N/L) for protection of human health in some wells completed in the Saddle Mountains aquifer (DEQ 2009). In response, DEQ established the Lindsay Creek Nitrate Priority Area (NPA) (DEQ 2008; DEQ 2014). NPAs are areas where additional monitoring and ground water quality improvement are needed to protect human health and the environment. DEQ uses ground water nitrate monitoring results to delineate and prioritize NPAs. Additional information about NPA delineation and ranking is available in the 2014 Nitrate Priority Area Delineation and Ranking Process document (DEQ 2014).

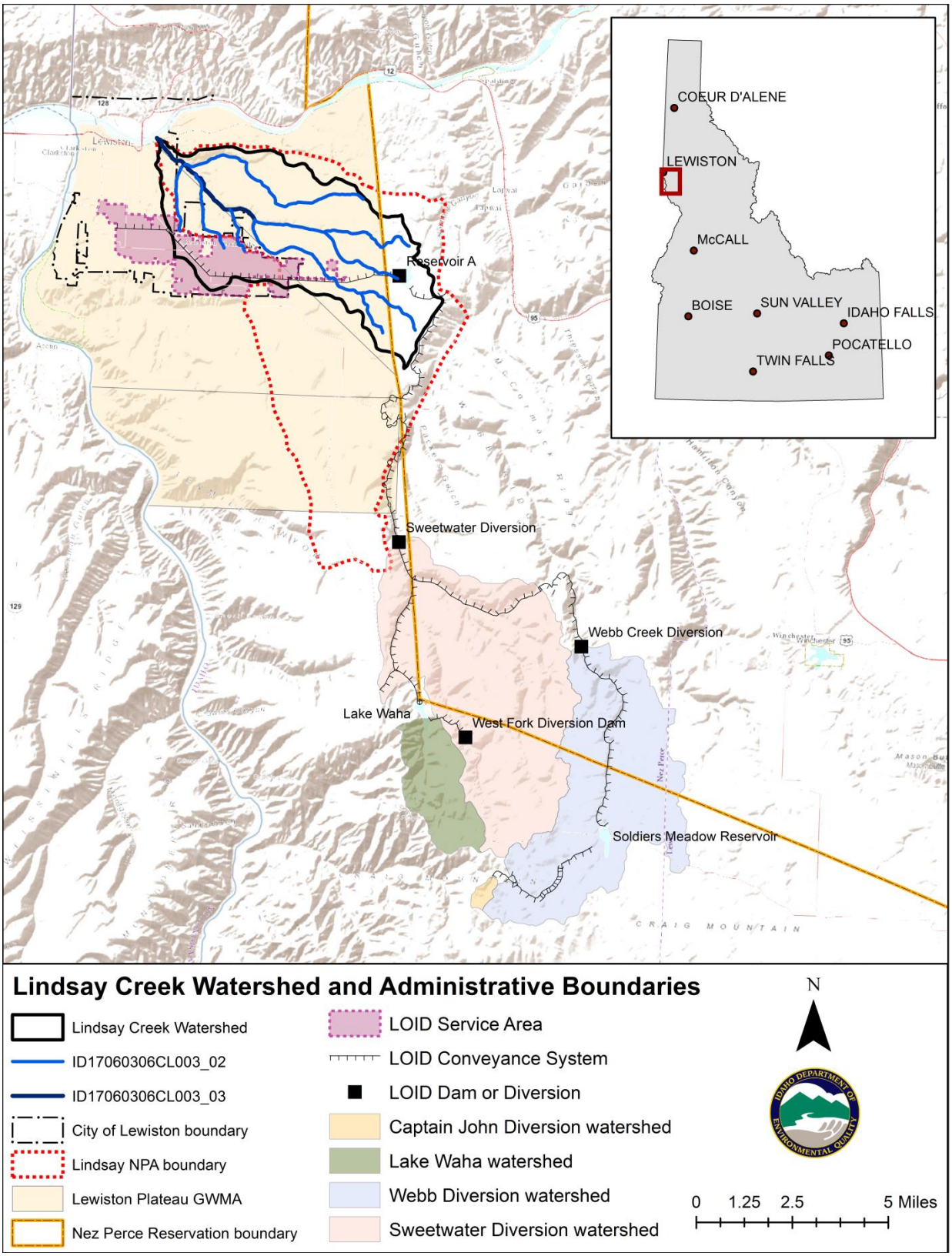

Figure 1. Lindsay Creek watershed and administrative boundaries.

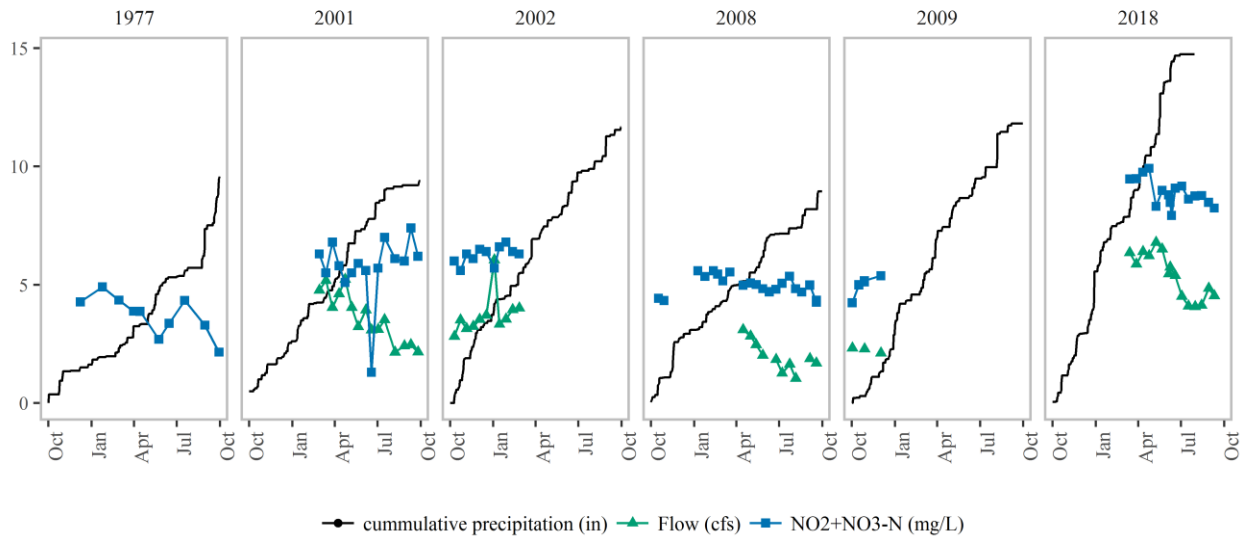

Figure 2. Water year stream flow and nitrate plus nitrite nitrogen concentrations at the Lindsay Creek mouth, and cumulative precipitation measured at the Lewiston Airport.

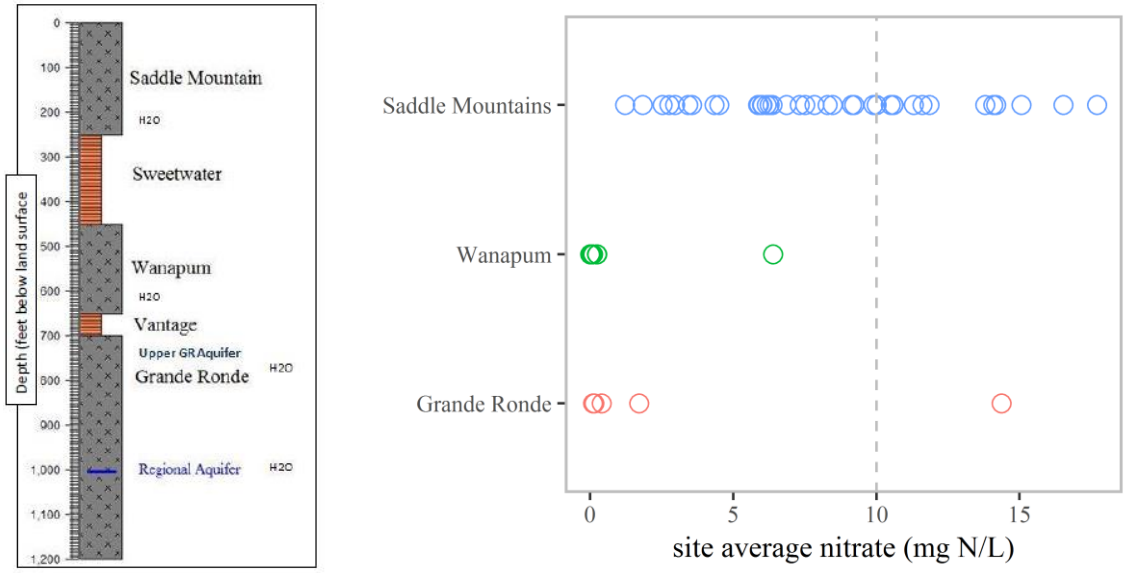

Figure 3. Geologic strata and average nitrate concentrations at wells and springs sampled by DEQ within the Lindsay Creek NPA and Lewiston plateau ground water management area (Figure 1). Geologic strata are from Neely 2018.

4 Nitrate Concerns

In 1978, the Idaho Department of Health and Welfare Division of Environmental Quality identified elevated nutrient concentrations in Lindsay Creek and observed associated nuisance algal growths (IDHW 1978). DEQ subsequently placed Lindsay Creek on Idaho's list of waters impaired under the Clean Water Act (Idaho's §303(d) list). In 2007, DEQ developed a Total Maximum Daily Load (TMDL) for nitrite plus nitrate nitrogen (NO_3+NO_2-N) in Lindsay Creek. A TMDL specifies maximum inputs of a pollutant from all sources that can occur while sill meeting state water quality standards. The Lindsay Creek TMDL attributed all loading to nonpoint sources and did not delineate contributions from different potential nonpoint sources (DEQ 2007). The TMDL stated "Elevated nutrient levels in Lindsay Creek appear to originate within the watershed and from ground water springs…" and that the TMDL was "developed to initiate protective ground water quality management actions…" (DEQ 2007).

In 2008, DEQ designated the Lindsay Creek Ground Water Nitrate Priority Area (NPA) (DEQ 2008; DEQ 2014) (Figure 1) to better characterize and address elevated nitrate in ground water. NPAs are areas where DEQ develops ground water quality improvement plans, conducts regular ground water nitrate monitoring, and water quality improvement is needed to protect human health and the environment (DEQ 2014). Since 2008, when DEQ designated Lindsay Creek NPA and began regular NPA monitoring, NO_3+NO_2-N concentrations ranged from 0.01-21 mg N/L and exceeded 10 mg N/L at least once in 15 out of 31 (48%) of wells sampled by DEQ. A draft ground water management plan for the Lindsay Creek NPA was developed in 2009; it recommended more public education about nitrate health risks and sources, as well as voluntary implementation of agricultural and residential best management practices to reduce nitrate inputs to ground water (DEQ 2009).

Several water quality improvement projects have been implemented in the watershed, but nitrate concentrations have remained high (Figure 2; Figure 3). An agricultural implementation plan detailing best management practices for reducing nutrient inputs from agriculture was developed in 2008 (NPSWCD 2008). Funds from two Clean Water Act Section 319 Nonpoint Source Management Program grants ($493,534 total) were used for nutrient reduction projects on several properties (PCEI 2012; NPSWCD 2016). In 2018, the City of Lewiston extended a sewer line into the eastern Lewiston Orchards area, and some residences previously served by individual on-site septic systems are now served by city sewer.

Potential nonpoint nitrate sources include organic nitrogen in soil, fertilizers, livestock, pets, wildlife, municipal storm water discharges, and septic systems. Septic systems have been cited as a potentially significant nitrate source. IDHW hypothesized septic systems were a source of stream nutrient contamination as far back as 1978 (IDHW 1978). In 2018, the City of Lewiston installed a sewer trunk line to extend sewer service into the eastern Lewiston Orchards, and cited elevated nitrate as one justification for the extension project (City of Lewiston URA 2017).

Previous DEQ efforts to evaluate septic contributions to surface water and ground water yielded inconclusive results. In 2005, DEQ tested for caffeine, methylene blue active substances (MBAS) and chlorine at several surface water sites; all three parameters can be used as indicators of septic inputs. Results were below the laboratory practical quantitation limit (PQL) (DEQ 2007). In 2017, DEQ again tested for the presence of caffeine in outflow of a culvert on Burrell Avenue draining the eastern Lewiston Orchards where septic systems are prevalent;

caffeine was detected (0.0346-0.0567 µg/L, PQL = 0.025 µg/L), suggesting potential septic inputs at this one site (DEQ 2017).

In 2005, DEQ also evaluated nitrogen stable isotope ratios (δ15N) in surface water. Some nitrate sources have distinct δ15N signatures that can assist source identification. However, δ15N did not clearly differentiate potential sources. Results indicated nitrate could derive from organic and inorganic nitrogen in soil, human waste, or animal waste (DEQ 2007). Considering the Lindsay Creek watershed includes ranches and livestock operations and nitrogen and oxygen isotopes are not able to differentiate human and animal waste sources (Xue et al. 2009; Fenech et al. 2012; Nikolenko et al. 2017), nitrogen and oxygen stable isotopes likely will not help delineate septic contributions.

5 Artificial Sweeteners and Caffeine as Septic Markers

Sucralose (brand name Splenda®) and acesulfame are artificial sweeteners used in some foods, beverages (diet sodas), and medicines. The US Food and Drug Administration (FDA) approved acesulfame for use as a non-nutritive sweetener in specific types of foods and beverages in 1988, and as a general purpose sweetener and flavor enhancer in 2003 (FDA 1988; FDA 2003). FDA does not allow acesulfame to be used as a sweetener in meat and poultry food products (21 CFR 172.800). USDA approved sucralose for use as a non-nutritive sweetener in 15 food categories in 1998 and as a general purpose sweetener in 1999 (FDA 1998, FDA 1999).

These sweeteners are frequently used as indicators of human wastewater inputs to surface water or ground water because they are unique to human waste sources and do not easily break down (Lange et al. 2012; Lim et al. 2017). Acesulfame and sucralose are not naturally-occurring and are unique to human waste. After being ingested, these compounds are not readily adsorbed or metabolized in the human body, and are excreted after little or no breakdown (Lange et al. 2012). Wastewater treatment plants and septic systems also do not completely remove acesulfame and sucralose from wastewater (Table 1). Water treatment typically removes less than 50% of sucralose in wastewater (Table 1). Reported acesulfame removal is consistently less than 100%, but is highly variable (Table 1). In some cases, sweeteners appear to accumulate in wastewater treatment plants or septic systems, yielding greater concentrations in effluent than influent (see negative removal efficiencies in Table 1). Because they are not completely removed by wastewater treatment, detection of these sweeteners in water samples strongly suggests wastewater or septic effluent is present. Sweeteners have been used in local (Robertson et al. 2016, Spolestra et al. 2017), state (Silvanima et al. 2018), and national (Bernot et al. 2016) assessments of wastewater inputs to ground water and surface water.

Caffeine is a stimulant present in many beverages (coffee, tea, sodas, energy drinks) foods (chocolate), and medicines. Properly-functioning septic systems remove caffeine very efficiently. Schaider et al. (2017) reviewed studies that reported caffeine concentrations in both septic systems (tank liquid or effluent) and downstream water (drainfield leachate or effluent from an alternative system) and calculated caffeine removal efficiencies (effluent concentration / influent concentration x 100). Median removal efficiencies were 99% for septic drainfields (N=15) and 93% for alternative systems (N=17). Other studies not included in Shaider et al. (2017) also reported 100% caffeine removal efficiencies (Yang et al. 2016; Yang et al. 2017a). Detection of

caffeine in water samples therefore suggests effluent from poorly functioning septic systems is present. Caffeine is widely used as a septic effluent marker in studies evaluating wastewater impacts on water quality (Buerge et al. 2003; Lim et al. 2012; Schaider et al. 2017).

Together, artificial sweeteners and caffeine can provide useful information about septic system inputs to ground water and surface water. Artificial sweeteners are markers of septic effluent and indicate where effluent inputs are present. Detection of caffeine further suggests inputs from one or more poorly-functioning septic systems.

Table 1. Sucralose, acesulfame, and caffeine removal efficiencies reported in the literature for wastewater treatment plants (WWTP), drinking water treatment plants (DWTP) and septic systems.

			Removal (%)		
Source	**Site Type**	**N**	**Sucralose**	**Acesulfame**	**Caffeine**
Subedi & Kannan (2014)	WWTP	2	1.6	-54	
Li et al. (2018)	WWTP	5	-3.0 to 30		
Castronovo et al. (2017)	WWTP	13		57-97	
Qi et al. (2015)	WWTP	5	6 to 31		91-100
Schaider et al. (2017)	WWTP	13			43-100
Ryu et al. (2014)	WWTP	1	24	4	
Thomas & Foster (2005)	WWTP	3			99-100
Scheurer et al. (2015)	WWTP	1		54	
Cardenas et al. (2016)	WWTP	1		92	100
Yang et al. (2017)	WWTP	9	< 0 to 50		> 90
Mawhinney et al. (2011)	DWTP	13	0 to 48		
Hoque et al. (2014)	aerated sewage lagoon	1	-330 to -83[b]		
Yang et al. (2016)	septic system	1	0		100
Yang et al. (2017a)	septic system	3	45-85		100
Schaider et al. (2017)	septic system	15			34-100
Robertson et al. (2016)[a]	septic system	5		-4 to 93	

[a]removal efficiencies calculated here using concentrations in tank liquid and in ground water below or downstream from infiltration beds from Table S1.
[b]values indicate range across seasons for one system
N indicates the number of sites where removal efficiencies were reported in the study.

6 Methods

6.1 Septic System Locations

Geographic information systems (GIS) records provided by Nez Perce County (Bill Reynolds, personal communication, April 3, 2018) were used to map locations of parcels with septic systems, and estimate the number of parcels with a septic system in the watershed. Nez Perce County indicated their GIS record accurately reflect septic system distribution within the Lewiston city boundaries, but may not be up to date for some areas outside city boundaries (Bill Reynolds, personal communication, April 5, 2018). It was not possible to map the exact location of each septic system; location data for each septic system are not available. County GIS data

were used to estimate the number of septic systems upstream from each stream sample point, assuming there is only one system per parcel.

Idaho North Central Public Health district (PH-INCD) provided addresses and parcel numbers for properties where PH-INCD issued a septic repair permit in the Lewiston area between 2011 and November 2018 DEQ (Ed Marugg, personal communication November 16, 2018). PH-INCD issues repair permits when PH-INCD determines a septic system is malfunctioning and needs repairs. DEQ mapped locations of these parcels to determine the number and location of parcels within Lindsay Creek watershed boundaries where PH-INCD confirmed septic failures (Figure 4). DEQ also evaluated if there is a history of septic failure upstream of where septic indicators were detected.

6.2 Septic Indicator Sampling

On September 10, 2018, septic indicator samples were collected at 5 stream sites, 5 domestic wells, 1 spring, and from the LOID irrigation water distribution system (Figure 4, Table 2). Samples were collected following procedures described in a project quality assurance project plan (DEQ 2018; DEQ 2018a; DEQ 2018b). Samples for acesulfame, sucralose, and caffeine were collected into 1 L glass amber bottles, placed on ice in the field, and delivered to the lab the same day. Stream water samples were grab samples collected from area within the stream channel that carries the greatest portion of flow (the thalweg). Well and spring samples were collected from a faucet, sample port, or spring as close to the source as possible before any treatment system. A portable water quality meter was used to measure pH, specific conductance, dissolved oxygen, and temperature at each sample location while allowing water to run for several minutes. Samples were collected after these parameters stabilized.

A one-compartment septic system that served a three bedroom residence in the eastern Lewiston Orchards was sampled on October 30, 2018. The goal of septic sampling was simply to confirm septic indicator compounds are present in septic systems within the watershed. The septic system had a 1000 gallon concrete tank, with 144 ft long, 3 ft wide drainfield. The system was installed in 2013 and the drainfield was repaired and expanded in 2014. The property owner hooked up their property to the Lewiston sewer line extension one week prior to DEQ sampling. DEQ obtained permission from the property owner and collected samples from the septic tank before it was pumped dry and decommissioned. Clean fluorinated ethylene propylene (FEP) tubing was lowered through the scum layer and a peristaltic pump was used to pump liquid from the septic tank through the FEP tubing and a short piece of silicone tubing into sample bottles.

Quality control samples included two field blanks and two field duplicates. A field blank was collected during surface water sampling by pouring deionized water into a sample container in the field. A septic field blank was collected by passing deionized water through sample tubing prior to pumping water for septic samples. A field duplicate was collected the spring site, and at the septic sampling site. A detailed summary of project data quality is provided in Appendix A.

6.3 Ground Water NO_3+NO_2-N and *Escherichia coli*

On September 10, 2018, ground water NO_3+NO_2-N samples were collected at each well and the spring site concurrently with septic indicator samples. Samples were collected using methods

described above (Section 6.2). In addition, NO_3+NO_2-N was sampled in one well (# 1225) and one spring (# 5000) twice per month March through September 2018 concurrently with surface water sampling (section 6.4) to compare seasonal NO_3+NO_2-N patterns in shallow ground water and surface water (Figure 4). Well and spring samples were collected as described above. All NO_3+NO_2-N samples were collected into clean high density polyethylene (HDPE) bottles, preserved with H_2SO_4, and placed on ice in the field. *Escherichia coli* (*E. coli*) samples were also collected from well and spring sites sampled 9/10/2018. *E. coli* samples were preserved with sodium thiosulfate in the field and analyzed at Anatek Labs within 24 hours of collection. Ground water data collected in 2018 were combined with older ground water NO_3+NO_2-N and NO_3-N data collected by DEQ (DEQ 2008; DEQ 2009; DEQ 2014) to plot aquifer nitrate patterns (Figure 3). NO_3+NO_2-N and NO_3-N were plotted together, because available data suggests nitrite (NO_2-N) is a very small fraction of NO_3+NO_2-N in ground water. IDWR staff helped identify the aquifer wells and springs with nitrate data draw water from (Daniel Sturgis, IDWR, December 26, 2018, personal communication).

6.4 Stream Discharge and NO_3+NO_2-N

On September 10, 2018, stream discharge was measured and NO_3+NO_2-N samples were collected concurrently with septic indicator samples at each stream site. In addition, discharge and NO_3+NO_2-N were measured twice per month from March through September 2018, at 6 stream sites, including the 5 stream sites where septic indicator samples were collected (Figure 4). Stream discharge was measured using a portable electromagnetic velocity meter and the velocity-area method. Water samples were grab samples collected from the thalweg. All NO_3+NO_2-N samples were collected into clean high density polyethylene (HDPE) bottles, preserved with H_2SO_4, and placed on ice in the field. Methods and results for 2018 stream discharge and NO_3+NO_2-N sampling are documented in detail elsewhere (DEQ 2018c). Stream data collected in 2018 were combined with stream discharge and NO_3+NO_2-N data previously collected by the Idaho Department of Environmental Quality (DEQ) (IDHW 1978, DEQ 2007) to plot inter-annual patterns (Figure 2).

6.5 Analytical Methods

All samples were analyzed at Anatek Labs in Moscow, ID. NO_3+NO_2-N was analyzed using colorimetric automated cadmium reduction (EPA 353.2) with a practical quantitation limit (PQL) of 0.1-1 mg N/L. *E. coli* was analyzed using American Public Health Association (APHA) method 9223B within 24 hours of collection; the PQL was 1 mpn/100 mL. For ground water and surface water samples, acesulfame, sucralose, and caffeine were analyzed using high pressure liquid chromatography (HPLC) coupled to a tandem mass spectrometer (MS/MS) by following a modified version of EPA 8321A. Samples were extracted following a procedure outlined by Kokotou et al (2013) in which samples are extracted using a solid phase cartridge (Waters Oasis HLB SPE) in order to reduce detection limits for the analytes of interest. Samples were extracted within seven days of sample collection and analyzed within 14 days. Isotopically labeled acesulfame, sucralose, and caffeine were used as internal standards to account for matrix effects. PQLs for acesulfame, sucralose, and caffeine were 0.04 µg/L, 1 µg/L and 0.01 µg/L, respectively. For septic liquid, samples were initially analyzed using a 10x dilution with no solid phase extraction because of difficulty passing septic liquid samples through the cartridges. Direct

injection analysis yielded PQLs of 5 µg/L for acesulfame, 10 µg/L for caffeine, and 100 µg/L for sucralose. Because sucralose was initially not detected in septic liquid with a PQL of 100 µg/L, samples were also subsequently analyzed for sucralose without dilution yielding a PQL of 25 µg/L.

7 Results

7.1 Septic System Locations

Nez Perce County Assessor GIS records indicated there were 780 parcels in the Lindsay Creek watershed with a septic system (15% of watershed parcels). Septic parcels were most dense in residential developments connected to Lapwai Road near North Fork Lindsay Creek and in the eastern Lewiston Orchards (Figure 4). PH-INCD issued 21 septic repair permits within the Lindsay Creek watershed between 2011 and November 2018. Four permits were issued during 2017-2018. Fourteen permits were issues for parcels in the Lewiston Orchards, and five were issued for parcels draining into NF Lindsay Creek (along Lapwai Road or in the Pheasant Trails and Viewpointe developments (Figure 4).

7.2 Septic Indicator Sampling

Artificial sweeteners were detected in septic liquid, ground water, and surface water. Aceslufame and sucralose were detected in septic liquid (Table 2). At least one sweetener was detected in 4 out of 5 domestic wells, 4 out of 5 locations in Lindsay Creek, and in the one spring sampled (Table 2, Figure 4). Acesulfame was detected in all sampled ground water sites drawing from Saddle Mountains aquifer (sites 5000, 1225, 1253, 2655, 533) (Table 2). Sucralose was also detected in one well drawing from the Saddle Mountains aquifer (site 2655) (Table 2). Sweeteners were not detected in a well drawing from the Grande Ronde aquifer (site 2022) or in LOID irrigation water. The acesulfame concentration in septic liquid was 40-1200 times greater than that observed in surface and ground water (Table 2). The sucralose concentration in septic liquid was 10-60 times greater than that observed in surface and ground water (Table 2).

Artificial sweeteners were detected at all 4 stream sites downstream of septic systems (Figure 4), and not at locations where septic inputs were not expected. Both acesulfame and sucralose were detected at the two stream sites with the highest upstream septic density (LC3, LC4; Table 2). Acesulfame and sucralose were not detected at a headwater stream segment (LC6). The headwaters segment is within a septic parcel (Figure 4), but the stream sample site was upstream of the septic system drainfield. Water at this stream site comes from a wetland with no known septic inputs. Sweeteners were also not detected in the LOID irrigation pipe system, which draws water from Mann Reservoir.

Caffeine was detected in septic liquid, one domestic well and at the two stream sites with the highest upstream septic density (LC3, LC4) (Table 2). These two stream sites also have a history of upstream septic failures based on PH-INCD records, including recently—PH-INCD issued two septic repair permits upstream of site LC3 in 2018, and two septic repair permits upstream of site LC4 in 2017 (Figure 4). The caffeine concentration in septic liquid was 6,000-9,000 times greater than that observed in surface water and ground water.

The total nitrogen concentration in septic liquid, calculated as the sum of NO_3+NO_2-N and total kjeldahl nitrogen (NH_3-N plus organic N) was 82.6 mg N/L. NO_3+NO_2-N in septic liquid was less than the laboratory practical quantitation limit (0.1 mg N/L). Low or non-detect NO_3+NO_2-N concentrations are common in septic liquid; nitrogen is typically present primarily as ammonia and organic nitrogen due to anaerobic (low oxygen) conditions in septic tanks. Ammonia is typically oxidized and converted to nitrate in septic drainfields.

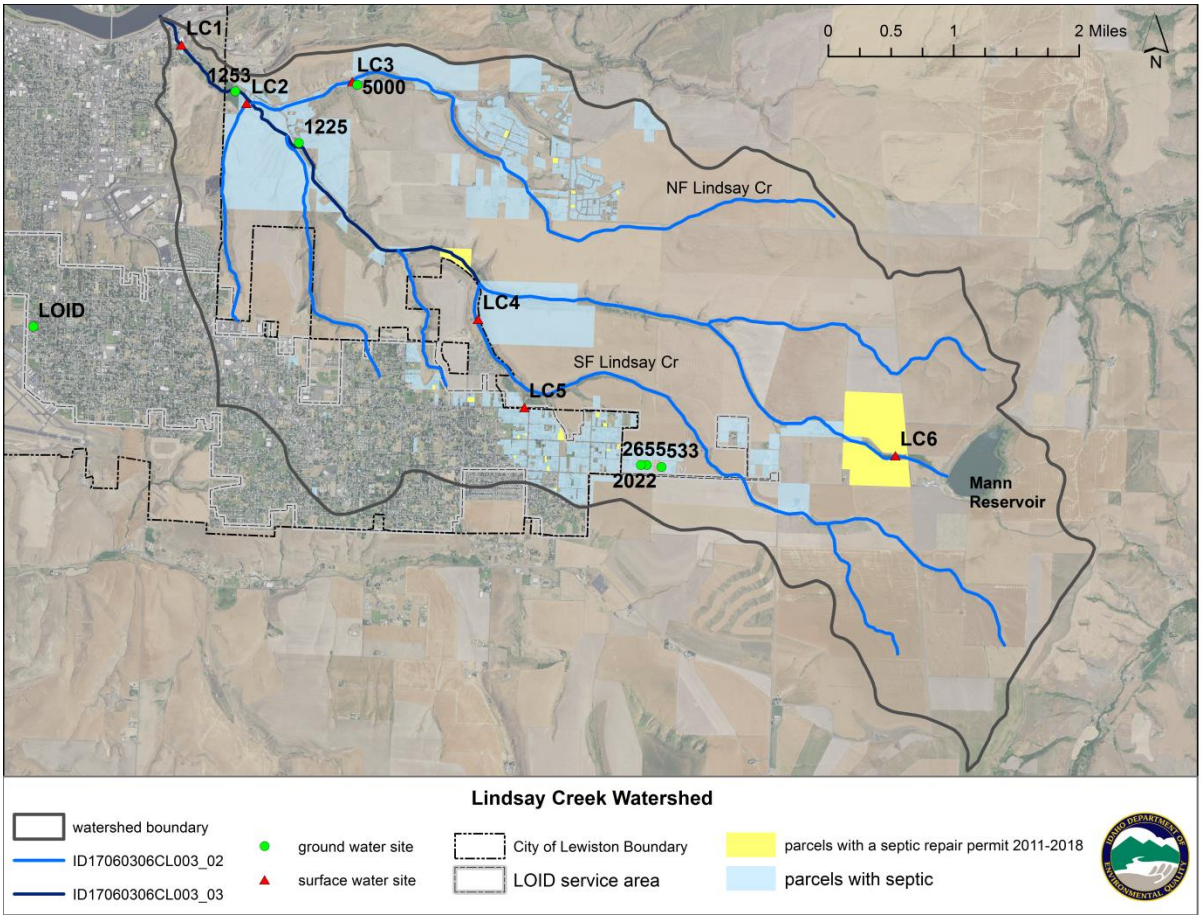

Figure 4. Sample sites, septic parcels, and parcels where PH-INCD issued a septic repair permit.

Table 2. Nitrate and septic indicator results from 9-10-2018 sampling.

Site	Upstream Septics (#, density mi^2)	Depth (ft)	NO_3+NO_2-N (mg N/L)	Caffeine (µg/L)	Sucralose (µg/L)	Acesulfame (µg/L)
Surface Water						
LC1 (mouth)	780, 3.6	--	8.24	ND	ND	0.141
LC2 (Gun Club Creek)	5, 4.3	--	8.92	ND	ND	0.0535
LC3 (NF Lindsay Creek)	271, 69	--	9.8	0.0167	1.32	0.265
LC4 (SF Lindsay Creek)	360, 70	--	10	0.0132	7.56	0.0644
LC5 (Burrell Ave culvert)	--	--	--	--	--	--
LC6 (headwaters)	0	--	1.14	ND	ND	ND
LOID distribution system	0	--	0.185	ND	ND	ND
Ground Water						
5000 (spring)	--	0	9.26	ND	ND	0.506
1225 (well)	--	16	7.15	ND	ND	0.404
1253 (well)	--	56	10.3	0.0189	ND	0.0857
2655 (well)	--	200	10.2	ND	1.15	1.22
533 (well)	--	225	9.88	ND	ND	0.202
2022 (well)	--	950	ND	ND	ND	ND
Septic System	--	--	ND*	119	80	51.3

Notes: LC5 was dry on 9-10-2018. All wells except 2022 are completed in the Saddle Mountains aquifer. ND indicates results less than the practical quantitation limit (0.1 mg N/l for nitrate, 0.04 µg/L for acesulfame, 1 µg/L for sucralose, and 0.01 µg/L for caffeine). *The total nitrogen concentration in septic liquid, calculated as NO_3+NO_2-N + total kjeldahl nitrogen (TKN) was 82.6 mg N/L.

7.3 Ground Water NO_3+NO_2-N and *Escherichia coli*

During September 2018 sampling, NO_3+NO_2-N concentrations ranged from 7.17-10.3 mg N/L at ground water sites drawing from the Saddle Mountains aquifer and was below the reporting limit at the one sampled site drawing from the Grande Ronde aquifer (Table 2). Based on data collected by DEQ in the Lewiston basin between 1988-2018, nitrate nitrogen concentrations are generally much greater in the Saddle Mountains aquifer than in the Wanapum or Grand Ronde aquifers, although a limited number of sites drawing from the Wanapum and Grande Ronde aquifers have been sampled (Figure 3). From March through September 2018, NO_3+NO_2-N concentrations and seasonal patterns at one spring and one shallow well drawing from the Saddle Mountains aquifer were similar to those observed in Lindsay Creek (Figure 5). *E. coli* was not detected at any of the ground water sites sampled in 2018.

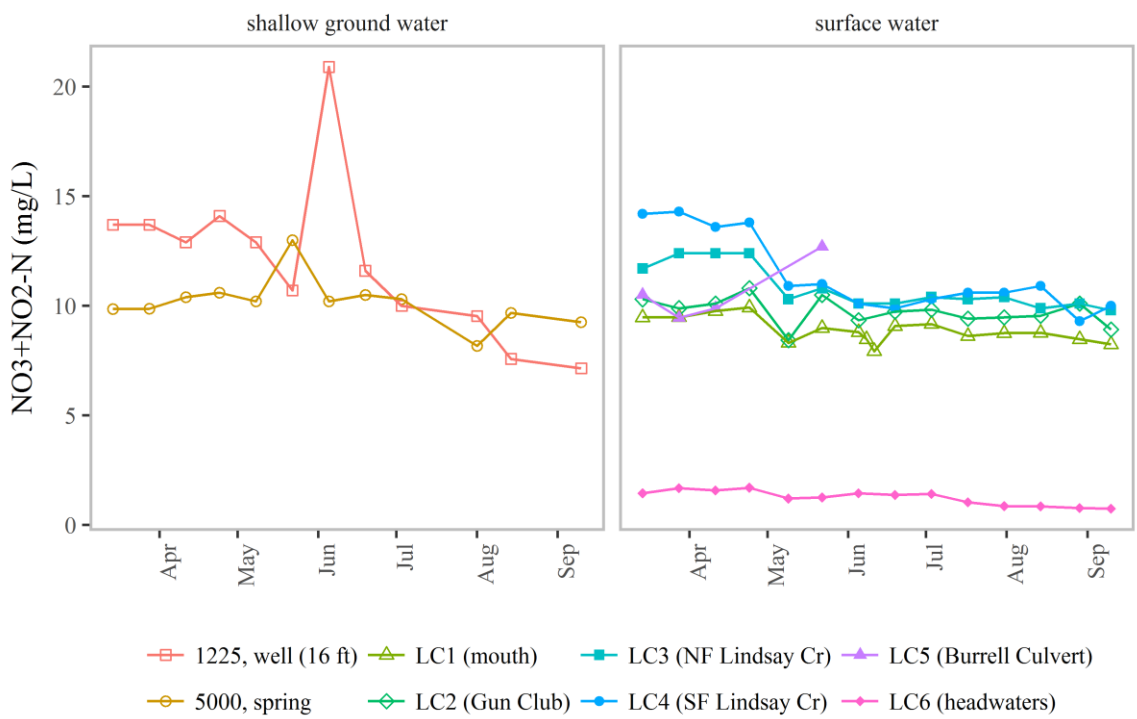

Figure 5. 2018 NO_3+NO_2-N concentrations at shallow ground water and surface water monitoring sites. See figure 4 for sample locations.

7.4 Stream Discharge and NO_3+NO_2-N

Between March and September 2018, stream discharge ranged from 4.1 to 6.8 cubic feet per second (cfs) at the Lindsay Creek mouth (LC1), and was ≤ 1 cfs at all monitored tributary sites. Discharge was greatest in May and decreased through September. In 2018, stream NO_3+NO_2-N ranged from 7.93 to 9.92 at the mouth (Figure 2), and 0.75 to 14.3 mg N/L across all monitoring sites.

Across years, stream NO_3+NO_2-N ranged from 1.3-9.92 mg N/L at the mouth. Stream discharge and NO_3+NO_2-N were substantially higher in 2018 than in previous years (Figure 2). Inter-annual variation in precipitation appears to affect stream NO_3+NO_2-N. At the mouth, discharge and NO_3+NO_2-N were greater in water years with greater winter precipitation and stream flow (Figure 2). Greater than normal winter precipitation in 2018, combined with greater than normal precipitation in water year 2017 likely contributed to higher than normal NO_2+NO_3-N in 2018 than previous years.

In 2018, NO_3+NO_2-N concentrations were elevated at sites where one or more septic indicators were detected, and were much lower or not detected at locations where septic indicators were not detected (Table 2). NO_3+NO_2-N concentrations were consistently high March-September 2018 at stream sites LC3 and LC4, where all three septic indicators were detected (Table 2, Figure 4, and Figure 5).

8 Conclusions

Septic Effluent is Present in Lindsay Creek and the Saddle Mountains Aquifer

Acesulfame and sucralose are useful indicators of human wastewater because they are unique to human waste sources and do not completely break down in human digestive systems, wastewater treatment plants, or septic systems (Lange et al. 2012). Individual on-site septic systems are likely the primary source of these compounds in the Lindsay Creek watershed. Other potential significant sources, such as landfills or effluent from wastewater treatment plants are not present. Within the Lindsay Creek watershed, acesulfame and sucralose were detected in a septic system, at surface water locations downstream from septic systems, and in shallow ground water within areas served by septic systems (Table 2, Figure 4). Sweeteners were not detected at three locations where septic inputs would not be expected: at headwaters stream site, in irrigation water, and in deep ground water (Table 2, Figure 4). These patterns suggest acesulfame and sucralose are reliable indicators of septic effluent in the watershed, and that indicate septic effluent is present in Lindsay Creek and in ground water collected from domestic wells.

It is not surprising that septic effluent is present in ground water and surface water. Septic systems are designed to discharge effluent to subsurface soils and therefore are a source of water to the shallow Saddle Mountains aquifer in the Lindsay Creek watershed. The Saddle Mountains aquifer is also a source of ground water inputs to Lindsay Creek through springs and the hyporheic zone of gaining stream segments, so septic effluent would be expected to enter Lindsay Creek. This study confirmed that septic effluent is present in Lindsay Creek and the Saddle Mountains Aquifer and documented its spatial distribution.

Effluent From Poorly-functioning Septic Systems is Present in Lindsay Creek and the Saddle Mountains Aquifer

Because properly-functioning septic systems remove caffeine very efficiently (Section 5), caffeine results (Table 2, Figure 4) suggest effluent from one or more poorly functioning septic systems has entered Lindsay Creek and the Saddle Mountains Aquifer. Caffeine was detected in

septic liquid, at two stream sites, and in one shallow well (Table 2). The two stream sites (LC3, LC4) are both downstream of areas with high septic density (~70 septics/square mile) and a recent history of septic failure based on PH-INCD records (Figure 4). In 2017, caffeine was also detected in outflow of a culvert draining the Lewiston Orchards (LC5) (0.0346-0.0567 µg/L, PQL = 0.025 µg/L), where septic density is high and PH-INCD has issued septic repair permits (DEQ 2017). Caffeine was not detected at other surface water and ground water locations, including those where septic inputs would not be expected and where there are no records of upstream septic failures (LC6, LOID water, a well drawing from Grande Ronde aquifer). These results suggest caffeine is a reliable marker for effluent from poorly-functioning septic systems in the Lindsay Creek watershed, and demonstrate such effluent has entered Lindsay Creek and the Saddle Mountains aquifer.

Nitrate Concentrations Are High Where Septic Effluent is Present, and Low Where it Is Not

In surface water, NO_3+NO_2-N concentrations were 8-10 mg N/L at stream sites where one or more artificial sweeteners were detected, and were < 0.8 mg N/L at sites where sweeteners were not detected (Table 2). Throughout 2018, NO_3+NO_2-N concentrations were consistently highest at the two stream sites (LC3, LC4) where all three septic indicators were detected (Figure 5). In ground water, NO_3+NO_2-N concentrations were 7.15-10.3 mg N/L at sites where one or more sweeteners were detected, and were below detection in a well drawing from the Grande Ronde aquifer where sweeteners were not detected (Table 2). These patterns suggest septic effluent contributes to elevated NO_3+NO_2-N concentrations observed in Lindsay Creek and in the Saddle Mountains aquifer.

The Relative Contribution of Septic Effluent to Nitrate Contamination is Not Clear and Merits Further Investigation

It is not possible to estimate the percent of NO_3+NO_2-N in ground water or surface water that comes from septic effluent using data collected in this study. Other studies have tested for correlations between NO_3+NO_2-N and sweeteners to assess if septic effluent is a primary factor controlling NO_3+NO_2-N concentrations (Spolestra et al. 2017). Here, NO_3+NO_2-N concentrations were not correlated with acesulfame concentrations, but the sample size (12 sites) was too small to robustly test for a correlation. It was not possible to test for a correlation between sucralose and NO_3+NO_2-N because sucralose was only detected at 3 sample sites. Collecting additional sweetener and NO_3+NO_2-N data would help test for correlations more robustly. However, estimating the percent of NO_3+NO_2-N in ground water or surface water that comes from septic effluent would likely require ground water flow and watershed modeling.

It is also not possible to reliably estimate the percent of ground water or surface water that comes from septic effluent using data collected in this study. Some studies have divided artificial sweetener concentrations in water samples by average local concentrations in septic liquid or septic effluent to estimate the percent septic effluent in ground water or surface water (Spolestra et al. 2017). In this study, only one septic system was sampled; many more would need to be

sampled to estimate average acesulfame and sucralose concentrations within the watershed. Sweetener concentrations in septic systems vary based on the diet and household products used by residents. Percent sweetener removal achieved by septic systems is also variable (Table 1), and is likely affected by system design, location, construction, and maintenance. Sweetener concentrations reported in the literature could be used for these calculations, but may not be representative of those in the Lindsay Creek area due to regional differences in diet, use of household products, local geology, or other factors.

References

Bernot, Melody J., Jesse C. Becker, Jason Doll, and Thomas E. Lauer. 2016. "A National Reconnaissance of Trace Organic Compounds (TOCs) in United States Lotic Ecosystems." *Science of The Total Environment* 572 (December): 422–33. *https://doi.org/10.1016/j.scitotenv.2016.08.060*.

Buerge, Ignaz J., Thomas Poiger, Markus D. Müller, and Hans-Rudolf Buser. 2003. "Caffeine, an Anthropogenic Marker for Wastewater Contamination of Surface Waters." *Environmental Science & Technology* 37 (4): 691–700.

Cardenas, M.A.R., Ali, I., Lai, F.Y., Dawes, L., Their, R., Rajapaske, J. 2016. "Removal of micropollutants through a biological wastewater treatment plant in a subtropical claimate, Queensland-Australia". *Journal of Environmental Health Science & Engineering*. 2016. 14: 14.

Castronovo, Sandro, Arne Wick, Marco Scheurer, Karsten Nödler, Manoj Schulz, and Thomas A. Ternes. 2017. "Biodegradation of the Artificial Sweetener Acesulfame in Biological Wastewater Treatment and Sandfilters." *Water Research* 110 (March): 342–53. https://doi.org/10.1016/j.watres.2016.11.041.

City of Lewiston Urban Renewal Agency (URA). 2017. Urban Renewal Plan for Revenue Allocation Area #5 Each Orchards Sewer. https://www.cityoflewiston.org/filestorage/848/6585/RAA_5_-_EOS_Plan_as_of_11-17-17.pdf

DEQ (Idaho Department of Environmental Quality). 2007. *Lindsay Creek Watershed Assessment and Total Maximum Daily Loads*. Lewiston, ID: Lewiston Regional Office. *http://www.deq.idaho.gov/media/454943-_water_data_reports_surface_water_tmdls_lindsay_creek_lindsay_creek_entire_final.pdf*

DEQ (Idaho Department of Environmental Quality). 2008. *2008 Nitrate Priority Area Delineation and Ranking Process*. December 2008. *http://www.deq.idaho.gov/media/471611-ranking_2008.pdf*

DEQ (Idaho Department of Environmental Quality). 2009. *Lindsay Creek Nitrate Priority Area: Ground Water Quality Management Plan (Draft).* December 2009. *http://www.nezperceswcd.org/Portals/2/DynamicDocs/Publications//Lindsay%20Creek%20Nitrate%20Priority%20Area%20-%20Ground%20Water%20Quality%20-%202009.pdf*

DEQ (Idaho Department of Environmental Quality). 2014. *2014 Nitrate Priority Area Delineation and Ranking Process*. July 2014. *http://www.deq.idaho.gov/media/1117845/nitrate-priority-area-delineation-ranking-2014.pdf*

DEQ (Idaho Department of Environmental Quality). 2017. Lewiston Orchards Surface Water Monitoring Report. August 2017. *http://www.deq.idaho.gov/media/60180509/lewiston-orchards-sw-report-0817.pdf*

DEQ (Idaho Department of Environmental Quality). 2018. *Quality Assurance Project Plan: Lewiston Regional Office TMDL 5 Year Review Monitoring Plan for Lindsay Creek 2018*. Lewiston, ID: Lewiston Regional Office.

DEQ (Idaho Department of Environmental Quality). 2018a. *Field Sampling Plan: Lindsay Creek TMDL Ground Water Sampling*. Lewiston, ID: Lewiston Regional Office.

DEQ (Idaho Department of Environmental Quality). 2018b. *Lindsay Creek TMDL Review QAPP Addendum: Lindsay Creek Septic Tracer Study*. Lewiston, ID: Lewiston Regional Office.

DEQ (Idaho Department of Environmental Quality). 2018c. *Lindsay Creek Surface Water Quality Monitoring Report: 2018*. Lewiston, ID: Lewiston Regional Office. *http://www.deq.idaho.gov/media/60182476/lindsay-creek-surface-water-quality-monitoring-report-2018.pdf*

FDA (US Food and Drug Administration). 1988. *Food Additives Permitted for Direct Addition to Food For Human Consumption; Acesulfame Potassium*. 21 CFR Part 172. *https://www.fda.gov/downloads/Food/IngredientsPackagingLabeling/FoodAdditivesIngredients/UCM404328.pdf*

FDA (US Food and Drug Administration). 1998. *Food Additives Permitted for Direct Addition to Food for Human Consumption; Sucarlose.* 21 CFR Part 172. *https://www.gpo.gov/fdsys/pkg/FR-1998-04-03/pdf/98-8750.pdf*

FDA (US Food and Drug Administration). 1999. *Food Additives Permitted for Direct Adddition to Food for Human Consumption; Sucralose*. 40 CFR Part 172. *https://www.gpo.gov/fdsys/pkg/FR-1999-08-12/pdf/99-20888.pdf*

FDA (US Food and Drug Administration). 2003. *Food Additives Permitted for Direct Addition to Food for Human Consumption; Acesulfame Potassium*. 21 CFT Part 172. *https://www.gpo.gov/fdsys/pkg/FR-2003-12-31/pdf/03-32101.pdf*

Fenech, C., Rock, L., Nolan, K., Tobin, J., Morrissey, A. 2012. "The potential for a suite of isotope and chemical markers to differentiate sources of nitrate contamination: A review." *Water Research* 46: 2023-2041.

Hoque, E.M., Cloutier,C., Arcieri, C., McInnes, M., Sultana T., Murray, C., Vanrolleghem, P.A., Metcalfe, C.D. 2014. "Removal of Selected Pharmaceuticals, Personal Care Products and Artificial Sweetener in an Aerated Sewage Lagoon." *Science of The Total Environment* 487 (July): 801–12. https://doi.org/10.1016/j.scitotenv.2013.12.063.I

Kokotu, M., Thomaidis N.S. 2013. "Determination of eight artificial sweeteners in wastewater by hydrophilic interaction liquid chromatography-tandem mass spectrometry." *Analytical Methods* 5: 3825-3833.

IDHW (Idaho Department of Health and Welfare). 1978. *Lindsay Creek Water Quality Status Report*. Report No. WQ-34. August 1978. *http://www.nezperceswcd.org/Portals/2/DynamicDocs/Publications//Lindsay%20Creek%20Water%20Quality%20Status%20Report%20-%201977.pdf*

IDWR (Idaho Department of Water Resources). 2015. *Final Order Adopting Ground Water* Management Plan. https://idwr.idaho.gov/files/legal/orders/2015/20150325-Final-Order-Adopting-Lewiston-Plateau-GW-Management-Plan.pdf

Lange, F.T., Scheurer, M., Brauch, H. 2012. "Artificial Sweeteners—a Recently Recognized Class of Emerging Environmental Contaminants: A Review". *Analytical and Bioanalytical Chemistry* 403 (9): 2503–18. *https://doi.org/10.1007/s00216-012-5892-z*.

Li, S., Ren, Y., Fu, Y., Gao, X., Wu, CJG, Ren, H., Geng, J. 2018. "Fate of Artificial Sweeteners through Wastewater Treatment Plants and Water Treatment Processes." *PLOS ONE* 13 (1): e0189867. https://doi.org/10.1371/journal.pone.0189867.

Lim, F., Ong, S., Hu, J., Lim, F.Y., Ong, S.Y., Hu, J. 2017. "Recent Advances in the Use of Chemical Markers for Tracing Wastewater Contamination in Aquatic Environment: A Review." *Water* 9 (2): 143. *https://doi.org/10.3390/w9020143*.

Mawhinney, D.B., Young, R.B., Vanderford, B.J., Borch, T., Snyder, S. 2011. "Artificial Sweetener Sucralose in U.S. Drinking Water Systems." *Environmental Science and Technology* 45(20): 8716-8722. https://doi.org/10.1021/es202404c.

Neely, K.W. 2018. Update of Hydrogeologic Conditions for the Lewiston-Lapwai area, Idaho 2012-2018. https://idwr.idaho.gov/files/groundwater-mgmt/201811-Lewiston-Lapwai-Groundwater-Resources-Update.pdf

Nikolenko, W., Jurado, A., Borges, A., Knoller, K., Brouyere, S. 2017. "Isotopic composition of nitrogen species in groundwater under agricultural areas: A review." *Science of the Total Environment* 621: 1415-1432.

NPSWCD (Nez Perce Soil & Water Conservation District). 2002. *Lindsay Creek Farming Practices Survey*. March 2002. *http://www.nezperceswcd.org/Portals/2/DynamicDocs/Publications//Lindsay%20Creek%20Farming%20Practices%20Survey%20-%202002.pdf*

NPSWCD (Nez Perce Soil & Water Conservation District). 2008. *Lindsay Creek Watershed Total Maximum Daily Load Implementation Plan for Agriculture*. April 2008. *http://www.deq.idaho.gov/media/454994-_water_data_reports_surface_water_tmdls_lindsay_creek_lindsay_creek_ag_imp_plan.pdf*

NPSWCD (Nez Perce Soil & Water Conservation District). 2016. *Lindsay Creek Water Quality Improvement Project Grant S472 Final Report*. January 2016.

NOAA (National Oceanic and Atmospheric Administration). 2018. Climate Data Online. *https://www.ncdc.noaa.gov/cdo-web/* . Lewiston Nez Perce Co Airport USW00024149. Checked December 2018.

PCEI (Palouse Clearwater Environmental Institute). 2012. *PCEI Invoice and Final Report: Lindsay Creek Riparian Animal Management Project Walton, Cowger, Neilson, and Canyon Crest Estates Sites*. January 2012.

Qi, W., Singer, H., Berg, M., Müller, B., Pernet-Coudrier, B., Liu, H., Qu, J. 2015. "Elimination of Polar Micropollutants and Anthropogenic Markers by Wastewater Treatment in Beijing, China." *Chemosphere* 119: 1054–61. https://doi.org/10.1016/j.chemosphere.2014.09.027.

Ralston, D. 2017. *Hydrologic Analysis of the Eastern and Southern Portions of The Lewiston Plateau Ground Water Management Area, Idaho: Progress Report and Request for Second Year Funding*. April 2017. https://idwr.idaho.gov/files/groundwater-mgmt/201704-Hydrogeologic-Analysis-of-Eastern-and-Southern-Lewiston-Plateau-GWMA.pdf

Robertson, W. D., D. R. Van Stempvoort, J. W. Roy, S. J. Brown, J. Spoelstra, S. L. Schiff, D. R. Rudolph, S. Danielescu, and G. Graham. 2016. "Use of an Artificial Sweetener to Identify Sources of Groundwater Nitrate Contamination." *Groundwater* 54 (4): 579–87. https://doi.org/10.1111/gwat.12399.

Ryu, J, Oh, J., Snyder, S.A., Yoon, Y. 2014. "Determination of Micropollutants in Combined Sewer Overflows and Their Removal in a Wastewater Treatment Plant (Seoul, South Korea)." *Environmental Monitoring and Assessment* 186 (5): 3239–51. https://doi.org/10.1007/s10661-013-3613-5.

Schaider, L.A., Rodgers, K.M., Rudel, R.A. 2017. "Review of Organic Wastewater Compound Concentrations and Removal in Onsite Wastewater Treatment Systems." *Environmental Science & Technology* 51 (13): 7304–17. *https://doi.org/10.1021/acs.est.6b04778*.

Scheurer, M., Heb, S., Luddeke, F., Sacher, F., Gude, H., Loffler, H., Gallert, Cl. 2015. "Removal of micropollutants, facultative pathogenic and antibiotic resistant bacteria in a full-scale retention soil filter receiving combined sewer overflow." *Environmental Science Processes & Impacts*. 2015. 17: 186.

Silvanima, J., Woeber, A., Sunderman-Barnes, S., Copeland, R., Sedlacek, C., Seal, T. 2018. "A Synoptic Survey of Select Wastewater-Tracer Compounds and the Pesticide Imidacloprid in Florida's Ambient Freshwaters." *Environmental Monitoring and Assessment* 190 (7): 435.

Spolestra, J., Senger, N.D., Schiff, S.L., 2017. "Artificial Sweeteners Reveal Septic System Effluent in Rural Groundwater." *Journal of Environmental Quality*. 46: 1434-1443.

Subedi, Bikram, Keshava Balakrishna, Ravindra K. Sinha, Nobuyoshi Yamashita, Vellingiri G. Balasubramanian, and Kurunthachalam Kannan. 2015. Mass Loading and Removal of Pharmaceuticals and Personal Care Products, Including Psychoactive and Illicit Drugs

and Artificial Sweeteners, in Five Sewage Treatment Plants in India. *Journal of Environmental Chemical Engineering* 3 (4, Part A): 2882–91. *https://doi.org/10.1016/j.jece.2015.09.031*.

Thomas, P.M., Foster, G.D. 2005. "Tracking Acidic Pharmaceuticals, Caffeine, and Triclosan Through the Wastewater Treatment Process." *Environmental Toxicology and Chemistry* 24 (1): 25. https://doi.org/10.1897/04-144R.1.

USGS (US Geological Survey). 2018. "StreamStats." *https://water.usgs.gov/osw/streamstats/*.

Xue, D., Botte, J., De Baets, B., Accoe, F., Nestler, A., Taylor, P., Van Cleemput, O., Berglund, M., Boeckx, P. 2009. "Present Limitations and future prospects of stable isotope methods for nitrate source identification in surface and groundwater." *Water Research*. 43: 1159-1170.

Yang, Y., Toor, G.S., Wilson, C.P, Williams, C.F. 2016a. "Septic Systems as Hot-Spots of Pollutants in the Environment: Fate and Mass Balance of Micropollutants in Septic Drainfields." *Science of The Total Environment* 566–567: 1535–44. *https://doi.org/10.1016/j.scitotenv.2016.06.043*.

Yang, Y., Liu, W.R., Liu, Y.S., Zhao, J.L., Zhang, Q.Q., Zhang, M., Zhang, J.N., Jiang, Y.X., Zhang, L.J., Ying, G.G. 2017. "Suitability of Pharmaceuticals and Personal Care Products (PPCPs) and Artificial Sweeteners (ASs) as Wastewater Indicators in the Pearl River Delta, South China." *Science of The Total Environment* 590–591 (July): 611–19. https://doi.org/10.1016/j.scitotenv.2017.03.001.

Yang, Y., Toor, G., Wilson, C.P, Williams, C.F. 2017a. "Micropollutants in groundwater from septic systems: Transofrmations, transport mechanism, and human health risk assessment." *Water Research* 123: 258-267.

Appendix A. Quality Assurance/ Quality Control

Background and Purpose

Before sampling, DEQ developed separate Quality Assurance Project Plans (QAPPs) for surface water monitoring (DEQ 2018), ground water nitrate monitoring (DEQ 2018a) and septic indicator sampling DEQ (2018b). The QAPPs specified data quality objectives and criteria were specified for data accuracy, precision, measurement range, representativeness, comparability, and completeness. Quality assurance data associated with surface water nitrate data is described in the *Lindsay Creek Surface Water Quality Monitoring Report: 2018* (DEQ 2018c). This appendix reviews quality assurance data associated with septic indicator sampling (DEQ 2018b).

Project Parameters

The project QAPP (DEQ 2018b) stated that project ground water, surface water, and septic liquid would be analyzed for acesulfame, sucralose, and NO_3+NO_2-N. Several additional parameters were added during the course of the project. When samples were submitted to the lab, Anatek offered to analyze samples for caffeine in all samples in addition to acesulfame and sucralose; sample holding times, preservation requirements, and analytical methods were identical for caffeine, which Anatek could easily report from mass spectrometer results without additional work. In addition, DEQ requested Anatek analyze septic liquid samples for NH_3-N (APHA 4500NH3G) and total Kjeldahl nitrogen (TKN) (APHA 4500NORGC) after initial NO_3+NO_2-N results were below detection. The QAPP did not specify quality assurance requirements for caffeine, TKN, and NH_3, but quality assurance procedures used are described below.

Precision

Precision is a measure of agreement between two measurements of the same parameter under prescribed conditions. The relative percent difference (RPD) between original and field duplicate samples can be used as a measure of precision. Field duplicates are two samples collected from the same location, representing the same sampling event, and carried through all assessment and analytical procedures in an identical manner.

The project QAPP (DEW 2018c) required that DEQ collect one surface water field duplicate and one septic liquid field duplicate for analysis of septic indicator parameters. This requirement was met (Table A1). The QAPP also defined a RPD goal of +/- 50% for results < 5 times the practical quantitation limit (pql), and +/- 25% for results > 5x the pql. These requirements were also met (Table A1).

Table A1. Field duplicate results.

Sample Date	Media	Parameter	Original Result	Duplicate Result	RPD (%)	PQL	Lab Report
9-10-18	Ground water	Acesulfame (µg/L)	0.506	0.503	0.6	0.04	180911049_REG2
9-10-18	Ground water	Sucralose (µg/L)	ND	ND	0	1	180911049_REG2
9-10-18	Ground water	Caffeine (µg/L)	ND	ND	0	0.01	180911049_REG2
10-30-2018	Septic liquid	NO_3+NO_2-N (mg N/L)	ND	ND	0	0.1	180131009_REG2_ADDON

10-30-2018	Septic liquid	NH_3-N (mg N/L)	72.6	72.3	0.4	0.5	180131009_REG2_ADDON
10-30-2018	Septic liquid	TKN (mg N/L)	73.1	82.6	-12.2	5	180131009_REG2_ADDON
10-30-2018	Septic liquid	Acesulfame (µg/L)	51.3	60.3	-16.1	5	180131009_REG2_ADDON
10-30-2018	Septic liquid	Sucralose (µg/L)	85.9	80.0	7.1	25	180131009_REG2_ADDON
10-30-2018	Septic liquid	Caffeine (µg/L)	119	117	1.7	10	180131009_REG2_ADDON

Accuracy

Accuracy is a measure of agreement between a "true" or reference value and the associated measured value. The QAPP required collection and analysis of one surface water field blank and one septic liquid field blank to evaluate accuracy. Field blanks are samples of a blank matrix, typically distilled water, prepared in the field under the same conditions, processed the same, and included for analysis as a regular sample. Results of all field blanks were below detection, and met QAPP requirements (Table A2).

Table A2. Field blank results.

Sample Date	Media	Parameter	Field Blank Result	PQL	Lab Report
9-10-18	Surface water	NO_3+NO_2-N (mg N/L)	ND	0.1	180911049_REG2
9-10-18	Surface water	Acesulfame (µg/L)	ND	0.04	180911049_REG2
9-10-18	Surface water	Sucralose (µg/L)	ND	1	180911049_REG2
9-10-18	Surface water	Caffeine (µg/L)	ND	0.01	180911049_REG2
10-30-18	Septic liquid	NO_3+NO_2-N (mg n/L)	ND	0.1	180131009_REG2_ADDON
10-30-18	Septic liquid	NH_3-N (mg N/L)	ND	0.05	180131009_REG2_ADDON
10-30-18	Septic liquid	TKN (mg N/L)	ND	0.5	180131009_REG2_ADDON
10-30-18	Septic liquid	Acesulfame (µg/L)	ND	5	180131009_REG2_ADDON
10-30-18	Septic liquid	Sucralose (µg/L)	ND	25	180131009_REG2_ADDON
10-30-18	Septic liquid	Caffeine (µg/L)	ND	10	180131009_REG2_ADDON

Sample Holding and Preservation Requirements

Sample holding times and preservation requirements were met for all ground water and surface water samples. Samples analyzed for acesulfame, sucralose, and caffeine were extracted within 7 days and analyzed within 14 days. The QAPP specified a 7 day holding time prior to extraction. Anatek did not flag any samples as having sample holding or preservation issues.

Sample holding and preservation requirements were also met for septic liquid samples analyzed for acesulfame, sucralose, and caffeine. DEQ requested Antaek re-analyze sucralose in septic liquid samples (including the field blank and field duplicate) after Antatek initially reported sucralose was below detection with a pql of 100 µg/L. Anatek re-analyzed an acidified aliquot of each sample on 12/5/18, 36 days after the sample was collected. Anatek staff reported they

expected the acidified aliquot to be stable (Mark Ritari, personal communication, 11/27/2018), and did not assign any qualifier to revised results.

Sample holding times were not met for septic liquid TKN and NH_3-N analyses. Anatek reported septic liquid results to DEQ on 11-26-2018. Because NO_3+NO_2-N results were below detection, DEQ requested Anatek also analyze TKN and NH_3-N using remaining septic liquid sample. Anatek analyzed NH_3-N on 11-28-2018, and TKN on 12-3-2018; both analyses exceeded the 28-day holding time. Samples were acidified in the field with H_2SO_4 prior to analysis. Because holding times were not met, NH_3 and TKN results may not accurately reflect nitrogen speciation in septic liquid samples. In this report, TKN results were only used to calculate total nitrogen in septic liquid by summing TKN and NO_3+NO_2-N results.

Data Representativeness

Data representativeness is the degree to which the sample data accurately and precisely represent site conditions. The project QAPP did not provide specific representativeness criteria. Field sampling and laboratory analysis followed standard procedures, samples were collected at representative locations, accuracy and precision requirements were met. DEQ does not believe sucralose, TKN and NH_3-N holding time issues described above significantly affects data representativeness. All project data therefore are considered adequately representative for purposes of this project.

Data Comparability

Comparability is the confidence with which one data set can be compared to another data set. The project QAPP did not specify comparability criteria. Because standard sampling and laboratory procedures were followed, procedures were consistent with those used in other similar studies, and no issues were identified during project data verification and validation, all data are considered adequately comparable.

Data Completeness

Data completeness is the percentage of valid data relative to the total possible data points. Because no sample results were rejected, project data completeness is 100%.

Conclusion

DEQ requires several internal quality assurance procedures. These include consultation with the DEQ quality assurance manager, registration of the project in a tracking spreadsheet, completion of three standardized quality assurance checklists, and review of all quality assurance data points. Project quality assurance goals were met. DEQ therefore considers all project data adequate for use in this report.