

Caffeine and Pharmaceuticals as Indicators of Waste Water Contamination in Wells

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Abstract

The presence of caffeine or human pharmaceuticals in ground water with elevated nitrate concentrations can provide a clear, unambiguous indication that domestic waste water is a source of some of the nitrate. Water from domestic, public supply, and monitoring wells in three communities near Reno, Nevada, was sampled to test if caffeine or pharmaceuticals are common, persistent, and mobile enough in the environment that they can be detected in nitrate-contaminated ground water and, thus, can be useful indicators of recharge from domestic waste water. Results of this study indicate that these compounds can be used as indicators of recharge from domestic waste water, although their usefulness is limited because caffeine is apparently nonconservative and the presence of prescription pharmaceuticals is unpredictable. The absence of caffeine or pharmaceuticals in ground water with elevated nitrate concentrations does not demonstrate that the aquifer is free of waste water contamination. Caffeine was detected in ground water samples at concentrations up to 0.23 $\mu\text{g/L}$. The human pharmaceuticals chlorpropamide, phensuximide, and carbamazepine also were detected in some samples.

Introduction

Ground water contaminated by nitrate from septic systems is a potential health hazard in many rural and suburban areas in the United States. Determination of specific chemicals that identify the source(s) of nitrate contamination in a way that is conceptually easy for the public to understand is important because water managers and health officials must persuade the public of the need for costly sewage disposal to protect aquifers used as sources of drinking water. Recently the U.S. Geological Survey (USGS) and the Center for Environmental Science and Engineering (CESE) at the University of Nevada–Reno investigated the potential for using caffeine and human pharmaceuticals as indicators of domestic waste water contamination to ground water.

Ground water contaminated by domestic waste water is likely to contain minute amounts of chemicals that are consumed exclusively by humans, such as caffeine and human pharmaceuticals. The ability of modern analytical instruments to detect nanogram per liter concentrations of organic compounds allows for the detection of low concentrations of caffeine and human pharmaceuticals. Because pharmaceutical compounds are designed to persist in the body long enough to have the desired therapeutic effect, they may also persist in the environment. Well water from areas where septic systems are used for waste water disposal was sampled to test whether caffeine or human pharmaceuticals are common, persistent, and mobile enough in the environment that they can be detected in nitrate-contaminated ground water and, thus, be useful indicators of recharge from domestic waste water.

Caffeine is a potential indicator of domestic waste water because it is clearly of anthropogenic origin and often has been detected in waste water and surface water. Early reports of caffeine in waste water (Sievers et al. 1977) and surface water that had been contaminated by waste water (Sheldon and Hites 1978; Gould and Richards 1984) noted the presence of caffeine but did not determine its concentrations. Paxéus and Schröder (1996) reported 37 $\mu\text{g/L}$ of caffeine in influent to a Swedish sewage treatment plant (STP) and 4 $\mu\text{g/L}$ in its effluent. Rogers et al. (1986) reported caffeine concentrations in municipal waste water ranging from 20 to 300 $\mu\text{g/L}$. Buszka et al. (1994) reported the presence of caffeine (1.3 to 2.4 $\mu\text{g/L}$) in surface water as far as 13.5 km downstream from a municipal waste water discharge point on Rowlett Creek, near Dallas, Texas. Concentrations of caffeine in water along the main stem of the Mississippi River, from Minneapolis, Minnesota, to New Orleans, Louisiana, ranged from 0.01 to 0.07 $\mu\text{g/L}$ (Barber et al. 1995); the highest concentrations were associated with population centers along the river and its tributaries.

Because caffeine is present in such large amounts in coffee, it seemed possible that caffeine concentrations in ground water contaminated by domestic waste water would be high enough to be readily detected. Gilbert et al. (1976) reported that the median caffeine content of 46 coffee samples as consumed (e.g., diluted with cream) was 360 mg/L (range 102 to 1170 mg/L). The average caffeine concentration in 33 samples of home-brewed coffee was 346 mg/L, ranging from 83 to 741 mg/L (Stavric et al. 1988). Although caffeine is present in elevated concentrations in tea and some soft drinks, caffeine concentrations are considerably higher in coffee (Bunker and McWilliams 1979).

Because a pot of coffee generally contains about 1500 to 1800 mL (10 to 12 five-ounce cups), a household that consumes coffee can generate hundreds to thousands of mg of caffeine daily. A large proportion of this caffeine may be discharged unmetabolized as waste to a household septic system. Caffeine is extensively metabolized; only about 3% of ingested caffeine is excreted unmetabolized in the urine (Tang-Liu et al. 1983). Thus, a household could discharge small amounts of caffeine to domestic waste

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water on a daily basis. A far larger source of caffeine to domestic waste water is likely to be disposal of unconsumed coffee, tea, or soft drinks down the sink, and rinsing of coffee pots and cups. Disposal of even a few cups of coffee into the sink could contribute hundreds of mg of caffeine to domestic waste water. For a family of four, daily discharge of liquid waste to a septic system averages 680 L per day (U.S. Environmental Protection Agency 1980). Assuming that caffeine acts conservatively, septic tank effluent could contain tens to hundreds of $\mu\text{g/L}$ of caffeine. Caffeine concentrations of 100 and 120 $\mu\text{g/L}$ in two waste water samples taken from septic tanks were reported by Umari et al. (1995). With these concentrations, it seemed reasonable that caffeine could be present in ground water contaminated by waste water, even if most of the caffeine was sorbed onto sediment, catabolized by bacteria, or diluted by mixing.

Human pharmaceuticals are also potential waste water indicators that have been detected in ground water and leachate from landfills. The analgesic phenacetin, in addition to caffeine, was among the more than 50 organic compounds detected in leachate from a sanitary landfill in Barcelona, Spain (Albaiges et al. 1986). Pharmaceuticals, including pentobarbital, meprobamate, phen-suximide, and four sulfonamide derivatives were detected in ground water 300 m downgradient from a landfill in Florida that may have received waste from a large hospital (Eckel et al. 1993). Sulfonamides and barbiturates originating as waste from a pharmaceutical company were shown to migrate in shallow ground water from their disposal site in a landfill in Denmark to wells more than 50 m downgradient (Holm et al. 1995).

Ground water samples were collected in three northern Nevada communities where on-site septic systems are the principal method for disposal and treatment of domestic waste water. Samples of effluent from a municipal STP and ground water beneath an alfalfa field irrigated with the effluent also were collected. The main thrust of the investigation was to determine if a unique chemical signature for ground water contaminated by domestic waste water could be identified. Samples were analyzed for caffeine and pharmaceuticals because the presence of these compounds would positively identify domestic waste water as a source of contamination.

Study Areas

A brief description of the three communities where samples were collected is given in Table 1. The Rancho subdivision and the community of Golden Valley are part of a small topographically closed basin known as Lemmon Valley, about 8 to 11 km north of Reno-Sparks, Nevada (Figure 1; Table 1). The Indian Hills subdivision in Carson Valley is approximately 50 km south of Reno-Sparks. In each of the communities, domestic sewage is disposed of in septic systems.

Lemmon and Carson Valleys are in the rainshadow of the Sierra Nevada mountain range. Annual precipitation on the valley floors is generally less than 20 to 25 cm and annual evaporation is greater than 100 cm (Harrill 1973; Maurer 1986). Vegetation in undeveloped areas is dominated by sagebrush (*Artemisia tridentata*) on the valley floors and pinyon-juniper forest in higher altitudes of the surrounding mountains.

Lemmon and Carson Valleys lie on the extreme western edge of the basin and range physiographic province of the western United States. They are typical basin and range valleys; their long axis is oriented north-south with a down-dropped structural block below the valley and up-thrown mountain blocks to the east and

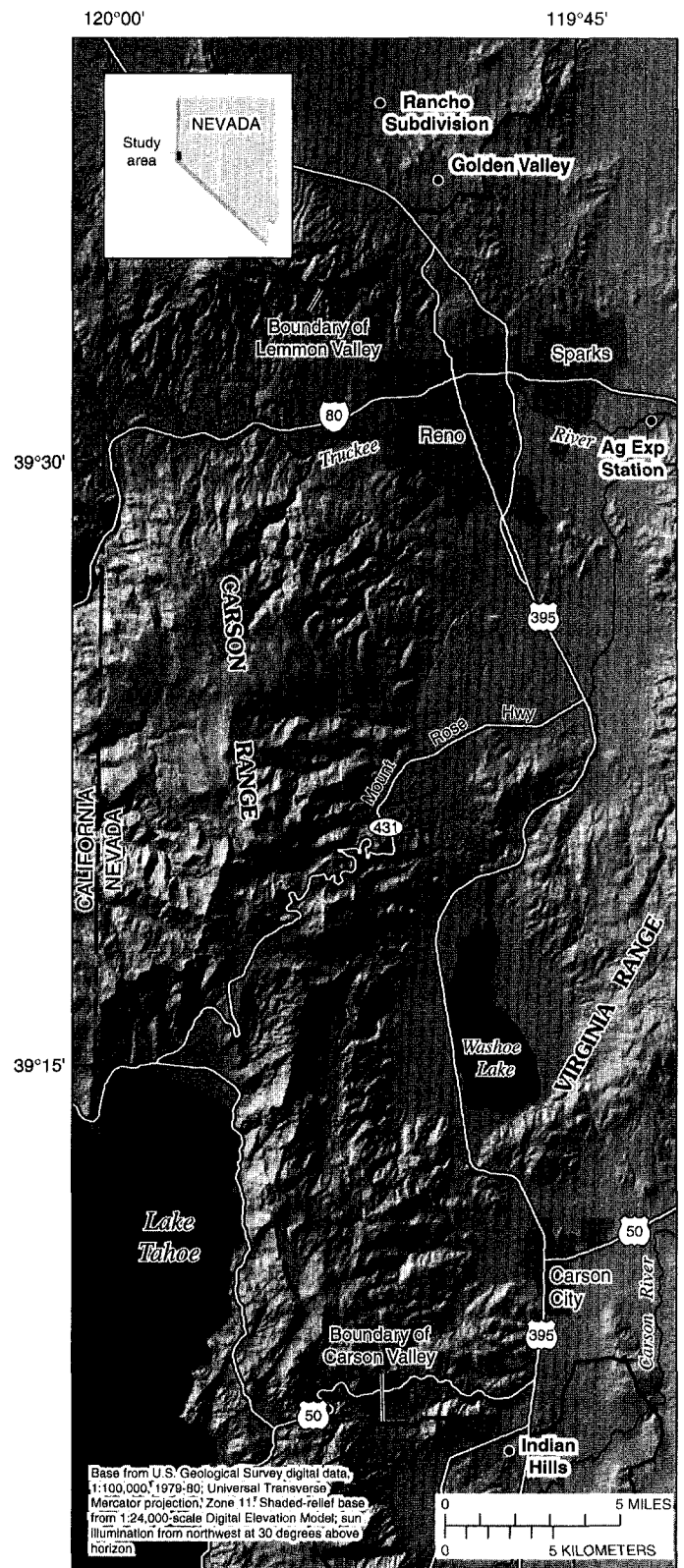


Figure 1. Communities in northern Nevada where ground water and sewage treatment plant effluent samples were collected for analysis of caffeine and pharmaceuticals.

west. Structural depressions occupied by the valleys are partly filled with interbedded deposits of sand, gravel, clay, and silt derived primarily from adjacent mountains. These deposits are not laterally extensive and the unconsolidated deposits are highly porous and commonly transmit water rapidly. Consolidated rocks, which compose the mountains and underlie the valley fill, commonly

Table 1
Summary of Four Locations Studied

Area	Site Description	Aquifer Characteristics	Source of Household Water
Rancho subdivision, Lemmon Valley	0.125–0.25 acre lots, altitude 1500–1515 m	2–7.5 m to water, valley fill	Off-site public supply wells
Golden Valley area, Lemmon Valley	1–3 acre lots, altitude 1550–1620 m	20–60 m to water, valley fill and fractured/decomposed granite along mountain front	On-site domestic wells
Indian Hills subdivision, Carson Valley	0.5–5 acre lots, altitude 1465–1525 m	15–30 m to water, valley fill	On-site public supply wells
Agricultural Experiment Station	Alfalfa fields irrigated with STP effluent, altitude 1335–1340 m	0.5–4 m to water, valley fill, flood plain of Truckee River	Not applicable

have low porosity and do not transmit water rapidly except where highly fractured.

In Lemmon Valley, ground water flows from recharge areas in the surrounding mountains to playa areas near the center of the valley where it discharges by evapotranspiration (Harrill 1973). Recharge in Lemmon Valley is from infiltration of precipitation in the mountains, effluent from septic systems, and, recently, deep injection of imported water. In the Golden Valley area of Lemmon Valley, effluent from septic systems is estimated to provide about 50% of annual ground water recharge (Widmer and McKay 1994). Houses in the Golden Valley area are located on the mountain front, alluvial apron, and higher altitudes of the valley floor. The Rancho subdivision is in a low-lying area of the valley floor, within 1 to 2 km of a playa.

In Carson Valley, ground water flows from recharge areas in the surrounding mountains to the valley floor where it discharges to the Carson River (Maurer 1986). The Indian Hills subdivision is located on hills along the flank of the Sierra Nevada and depth to water is 15 to 30 m. The subdivision lies between the recharge area and the Carson River.

The altitude of the water table measured in the monitoring wells at the time of sample collection is provided in Table 2. The water table elevations in the domestic and public supply wells are not helpful in evaluating ground water flow over short distances because the wells have not been surveyed and the water levels, taken from the well drillers' logs, were made in different years and seasons.

In this study, comparisons were not made between upgradient and downgradient sites because samples were not collected along defined flowpaths above and below known sources of waste water contamination. Instead, samples were collected primarily from wells that were known to have elevated nitrate concentrations and were spread through the communities. The monitoring wells in the Rancho subdivision were drilled on public right-of-ways and their nearness to a source of waste water was a matter of chance. In Golden Valley and in the Indian Hills subdivision, the domestic and public supply wells are required by law to be at least 100 feet (30.48 m) from the nearest septic system.

Methods

Sampling Sites

Ground water samples in the Rancho subdivision were collected from shallow, 2 inch diameter PVC monitoring wells. In Golden Valley, samples were collected from domestic wells and in Carson Valley samples were collected from domestic and public supply

wells. For comparison with ground water samples, two samples of treated sewage effluent were collected at the Truckee Meadows Water Reclamation Facility, an STP that treats waste water for the cities of Reno and Sparks. At the University of Nevada–Reno agricultural experiment station in Sparks, two ground water samples were collected from a shallow, 2 inch diameter PVC monitoring well at the edge of an alfalfa field where effluent from the STP is used for irrigation.

Sampling Procedure

Ground water samples were collected using three methods. Public supply and domestic well samples were collected from faucets before any storage or treatment tanks. Shallow monitoring wells were developed and samples were collected using either disposable Teflon™ bailers or a Bennett™ submersible pump discharging through a copper tube. Samples from pumping wells were collected after more than three casing volumes of water were pumped from the well and after on-site measurements of pH, specific conductance, temperature, and dissolved oxygen had stabilized. Samples of treated sewage effluent were collected at the STP from a faucet on a pipe carrying treated effluent to the University of Nevada–Reno agricultural experiment station.

Unfiltered samples for caffeine and pharmaceutical chemical analysis were stored in 1 L, amber, baked-glass bottles. Samples were chilled on ice after collection and stored in refrigerators at 4°C until analyzed.

A sample blank was collected in the field by pouring volatile organic and pesticide-free blank water into a disposable Teflon bailer and then emptying it into a sample bottle. The sample blank was then shipped to the USGS laboratory in Arvada, Colorado, and analyzed with other samples collected that day or a few days earlier. No caffeine or pharmaceutical compounds were detected in the blank and the pharmaceutical compounds identified in this paper are not used by the personnel who collected the samples. Because of concerns about sample contamination, coffee was not consumed in the field laboratory truck during sample collection.

Chemical Analysis.

Caffeine samples were analyzed using two methods. A simple method for the analysis of caffeine in ground water that achieved a detection limit of 5 µg/L was developed at the CESE at the University of Nevada–Reno that uses high performance liquid chromatography (HPLC) and requires no extraction. The method was modified from an existing procedure that results in a baseline

Table 2
Data-Collection Site Information, Concentrations of Nitrate, Ammonia, and Caffeine in Ground Water
and STP Effluent in Northern Nevada Valleys

Well depth and potentiometric surface are in meters (m). Concentrations are in milligrams (mg) or micrograms (μg) per liter (L).
 Well types are M, monitoring; D, domestic; and P, public supply; NA, Not Analyzed.

Site	Well Depth (m)	Potentiometric Surface	Well Type	Sample Date	Nitrogen (mg/L - N)		Caffeine
					NO ₃	NH ₃	($\mu\text{g/L}$)
Rancho Subdivision (Lemmon Valley) Nevada							
LV2	7.5	1502	M	13-Jun-96	7.3	0.011	<5
				17-July-97	8.6	<0.015	<0.04
LV3	7.5	1500	M	14-Aug-96	3.5	0.021	<5
LV4	9.0	1499	M	05-Sep-96	5.1	0.005	<5
LV5	9.0	1505	M	05-Sep-96	17	<0.002	<5
				19-Jun-97	30	<0.015	0.23
LV6	6.0	1502	M	12-Aug-96	5.4	<0.002	<5
			M	17-July-97	7.8	<0.015	0.001 ^a
LV8	4.5	1510	M	14-Aug-96	0.29	<0.002	<5
			M	19-Jun-97	<0.05	<0.015	0.03 ^a
LV9	6.5	1508	M	12-Aug-96	5.4	<0.002	<5
			M	17-Jul-97	5.4	<0.015	<0.04
LV10	10.5	1497	M	14-Aug-96	0.78	0.184	<5
				17-Jul-97	0.73	0.02	<0.04
Golden Valley area (Lemmon Valley) Nevada							
GV1	61	—	D	03-Jun-96	9	0.005	<5
				19-Jun-97	12	<0.015	<0.04
GV2	61	—	D	21-Jun-96	8.8	0.002	<5
				19-Jun-97	12	<0.015	<0.04
GV3	30	—	D	21-Jun-96	1.5	0.004	<5
				19-Jun-97	1.3	<0.015	<0.04
GV4	76	—	D	07-Jun-96	4.2	0.004	<5
Indian Hills Subdivision (Carson Valley) Nevada							
CV1	>15	—	D	10-Jun-96	1.5	<0.015	<0.04
CV2	77	—	D	11-Jun-96	2.1	<0.015	<0.04
RV5	72	—	P	04-Jun-96	1.8	0.002	<0.04
RV4	72	—	P	03-May-96	5.5	0.002	<0.04
				04-Jun-96	3.1	0.002	<0.04
Agricultural Experiment Station (Sparks, Nevada)							
AG-5	5.5	1335	M	16-Jul-97	12	<0.015	0.04
				09-Sep-97	—	—	0.03 ^a
Truckee Meadows Water Reclamation Facility (Sparks, Nevada)							
STP	—	—	—	16-Jul-97	<0.05	0.033	0.06
				05-Sep-97	—	—	0.08

^a Concentrations for caffeine less than the method reporting level (0.04 $\mu\text{g/L}$) are estimated; the analyte has passed all qualitative criteria and only the concentration is estimated, not the presence.

separation of cotinine from caffeine in plasma (Perkins et al. 1991). Given the potential for high concentrations of caffeine in ground water contaminated by domestic waste water, it seemed possible that caffeine could be detected in environmental samples without prior extraction steps. However, analysis indicated the need for much lower detection levels.

Because of the need for caffeine analysis in the sub part-per-billion range, the USGS National Water Quality Laboratory (NWQL) in Arvada, Colorado, developed a method requiring sample extraction that achieves a detection limit of 0.04 $\mu\text{g/L}$. One-liter water samples were extracted with methylene chloride using con-

tinuous liquid-liquid extraction. The extracts were analyzed using gas chromatography mass spectral (GCMS) analysis in the selected ion monitoring mode.

Pharmaceutical compounds were tentatively identified by mass spectral library search routines using the National Institute of Standards and Technology (NIST) mass spectral library. A compound was considered tentatively identified when its mass spectrum was judged to match a spectrum in the library, and the retention index matched that of the library spectrum. Standards for pharmaceuticals were not subjected to GCMS.

Results

Caffeine

The highest concentration of caffeine measured during this study was 0.23 µg/L. This was found in water from a shallow monitoring well in the Rancho subdivision that contained 30 mg/L nitrate. Much lower concentrations of caffeine were measured in samples from two other shallow monitoring wells in the subdivision (Table 2). Caffeine was not detected in any of the samples from deep wells (greater than 10 m) even though nitrate concentrations in two samples exceeded 10 mg/L (Table 2).

Caffeine concentrations in STP effluent were 0.06 and 0.08 µg/L (Table 2). In well AG5, a shallow monitoring well in a field irrigated with sewage effluent, the caffeine concentrations were 0.03 and 0.04 µg/L (Table 2). This concentration is one-half that of the STP effluent concentration. No metabolites of caffeine (e.g., 3-methylxanthine) were identified in ground water contaminated by septic systems nor were chlorinated derivatives of caffeine (e.g., 8-chlorocaffeine) detected in STP effluent. Dimethyl-imidazolidinetrione was detected in one sample of the STP effluent. This chemical is the major product (61%) of caffeine chlorination (Gould and Hay 1982).

Pharmaceuticals

Three pharmaceutical compounds were tentatively identified in two nitrate-contaminated wells (one monitoring, one domestic). The authors did not inquire whether these pharmaceuticals were used in households near the contaminated wells. The identities of the wells where specific pharmaceuticals were found are not reported to protect the privacy of people living near the wells. Chlorpropamide was detected in a shallow monitoring well in the Rancho subdivision and detection was confirmed in a repeat analysis a year later. Chlorpropamide is used in the treatment of diabetes. The average maintenance dose is 250 mg daily and approximately 20 to 30% is excreted unchanged in the urine (Katzung 1992). That such a large percentage of the dose is excreted unmetabolized by the body suggests that chlorpropamide may be persistent in the environment.

Phensuximide and carbamazepine were detected in a domestic well in Golden Valley. Both these drugs are used in the treatment of seizures (Goodman and Gilman 1975; Katzung 1992). Also detected in the well was iminostilbene, a metabolite of carbamazepine (Csetenyi et al. 1973). Subsequent sampling for pharmaceuticals in these domestic wells has not been possible, so detections were not confirmed by repeat analysis. However, detecting two distinct pharmaceuticals used for treatment of the same condition suggests that the findings are real. Concentrations of unmetabolized carbamazepine in urine are 2.4 to 3.8 mg/L for patients receiving long-term drug treatment (Maggs et al. 1997); carbamazepine doses of 1.0 to 2.0 g/day are tolerated (Katzung 1992). The usual adult daily dose for phensuximide is 2 to 4 g/day; unmetabolized phensuximide is excreted in the urine, although concentrations were considered negligible by Goodman and Gilman (1975). In dogs, trace amounts of the unmetabolized drug are excreted in the urine (Dudley et al. 1972). Another possible source of unmetabolized pharmaceuticals would be disposal of unused medicines in the toilet; physicians commonly suggest this method of disposal because they cannot be returned (Pintar 1997).

The domestic well is thought to be contaminated with waste water from a septic system for several reasons. Other organic chemicals were detected in the sample (e.g., the insect repellent DEET) and the well water has such a foul taste that bottled water

is used in the house. However, finding pharmaceuticals in the ground water was unexpected because the well has a 15 m sanitary seal, and the screened interval is more than 50 m below land surface, and depth to water is more than 30 m. The driller's log indicates the depth to weathered and fractured granitic rock is less than 10 m. Hydraulic conductivity for weathered and fractured granite from a similar setting in Carson City, Nevada, (Figure 1) was estimated to range from 3 to 9 m/day (Maurer and Berger 1997). Thus, effluent from the household septic system, or those of nearby houses, likely moves rapidly through the fractured rock to depth in the aquifer.

Conclusions

The presence of even low levels of caffeine and human pharmaceuticals in ground water with elevated nitrate concentrations is clear, unambiguous evidence that domestic waste water is a source of contamination except under unusual circumstances. These compounds might also be found in ground water in areas where hospital or pharmaceutical manufacturing wastes have been disposed in landfills.

Because no nitrate was detected in the STP effluent and well LV8, both of which contain caffeine, the presence of caffeine can indicate recharge from domestic waste water even when nitrate is not present in the water. The second highest caffeine concentration was from the sewage treatment plant, which denitrifies effluent before discharging it to the river. In well LV8, unpublished ¹¹B data indicate that this well is contaminated with domestic waste water even though it had very low nitrate concentrations.

Caffeine concentrations in ground water from a shallow monitoring well adjacent to alfalfa fields are one-half that of the STP effluent used to irrigate the fields. If the caffeine concentrations measured in the effluent are typical of the rest of the irrigation season, it indicates that much of the caffeine in effluent applied to fields can migrate through the unsaturated zone to the ground water. This suggests that sorption of caffeine to soil is not principally responsible for the lack of transport between septic systems and wells sampled for this study. Nearly complete catabolism of caffeine in the bacteria-rich environment of the septic system is a likely reason for caffeine not being detected more frequently in ground water near septic systems. Concentrations of caffeine in septic system effluent may be low even though caffeine concentrations in water inside a septic tank may exceed 100 µg/L (Umari et al. 1995). Nearly complete consumption of caffeine may occur either in the anaerobic septic tank itself or in the aerobic leach field.

The usefulness of caffeine as an indicator of recharge from septic systems is limited because it apparently is not conservative. The usefulness of human pharmaceuticals as indicators also is limited because the presence of pharmaceuticals is unpredictable. If caffeine and human pharmaceuticals are not detected, it does not demonstrate that a well has not been affected by domestic waste water. Other chemicals might be better indicators of waste water contamination if they do not undergo significant biodegradation and their occurrence is widespread and predictable. Barber et al. (1997) showed other chemicals originating in waste water, such as EDTA and detergent compounds, were persistent in ground water following aquifer injection of reclaimed water derived from secondary and tertiary-treated sewage effluent. Although EDTA and other chemicals may be better scientific indicators of waste water contamination, analysis for caffeine and human pharmaceuticals may be useful under certain circumstances when it is important to demonstrate the

source of nitrate contamination in a way that is conceptually easy for the public to understand.

The low concentrations of caffeine and pharmaceuticals found in wells with high nitrate concentrations during this study indicate that processes of catabolism, dilution with native ground water, and sorption to soils substantially reduce the amount of these chemicals in household waste water that make it through a septic system and unsaturated zone to the water table. Given this, caffeine and pharmaceuticals can be expected to be found in ground water under limited circumstances, such as when there is a septic system failure or where the flowpath between the waste water source and the well is short and there is minimal dilution and exposure to fine grained or organic-rich sediment. These compounds could be detected in deep wells if there is preferential flow through fractured rocks or through an inadequately sealed well annulus.

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Disclaimer

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Registry No. Caffeine, 00058-08-2; Carbamazepine, 298-46-4; Chlorpropamide; 94-20-2; DEET, 134-62-3; EDTA, 60-00-4; Iminostilbene, 256-96-2; Dimethyl-imidazolidinetrione, 5176-82-9; 3-methylxanthine, 1076-22-8; Phensuximide, 86-34-0

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