Ground Water Monitoring&Remediation

Pharmaceuticals and Other Organic Waste Water Contaminants Within a Leachate Plume Downgradient of a Municipal Landfill

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Abstract

Ground water samples collected from the Norman Landfill research site in central Oklahoma were analyzed as part of the U.S. Geological Survey (USGS) Toxic Substances Hydrology Program's national reconnaissance of pharmaceuticals and other organic waste water contaminants (OWCs) in ground water. Five sites, four of which are located downgradient of the landfill, were sampled in 2000 and analyzed for 76 OWCs using four research methods developed by the USGS. OWCs were detected in water samples from all of the sites sampled, with 22 of the 76 OWCs being detected at least once. Cholesterol (a plant and animal steroid), was detected at all five sites and was the only compound detected in a well upgradient of the landfill. N,N-diethyltoluamide (DEET used in insect repellent) and tri(2-chloroethyl) phosphate (fire-retardant) were detected in water samples from all four sites located within the landfill-derived leachate plume. The sites closest to the landfill had more detections and greater concentrations of each of the detected compounds than sites located farther away. Detection of multiple OWCs occurred in the four sites located within the leachate plume, with a minimum of four and a maximum of 17 OWCs detected. Because the landfill was established in the 1920s and closed in 1985, many compounds detected in the leachate plume were likely disposed of decades ago. These results indicate the potential for long-term persistence and transport of some OWCs in ground water.

Introduction

As demand for fresh water continues to increase, the quality of this limited natural resource is of increasing concern. In the United States, ground water provides ~40% of the nation's public water supply and is an important source of water used for irrigation (Alley et al. 1999). Ground water is also a major contributor to flow in many streams and rivers, and has a substantial influence on river and wetland habitats for plants and animals. Technological advancements in industry, agriculture, medical treatment, and common household conveniences have improved productivity and the quality of life, but have also created public concern for potential adverse human and ecological effects from man-made chemicals present in the environment (Daughton and Ternes 1999). Recent research has indicated that organic waste water contaminants (OWCs) are detectable in soil, ground water, surface water, and drinking water around the world (Buser et al. 1998; Ternes 1998; Stumpf et al. 1999; Golet et al. 2002; Kolpin et al. 2002; Metcalf et al. 2003). Sources of OWCs in the environment are derived from a variety of pathways (Heberer 2002). Landfills are one potential source of OWCs in the environment (Albaiges et al. 1986; Eckel et al. 1993; Holm et al. 1995; Seiler et al. 1999).

Although there has been a decrease in the number of active municipal solid waste landfills in the United States from 7924 in 1988 to 1858 in 2001, the total number of active and closed landfills is greater than 100,000 (Suflita et al. 1992; U.S. Environmental Protection Agency 2003). Landfills built in the last 60 years may contain complex mixtures of contaminants due to the disposal of increased numbers of chemicals manufactured and sold since the 1940s. Many landfills are typically located next to wetlands (Lambou et al. 1990), facilitating exposure of aquatic organisms to landfill leachate and flow to streams and rivers.

In 1994, the U.S. Geological Survey (USGS) as part of the Toxic Substances Hydrology Program (*www.toxics.usgs.gov*), in collaboration with scientists at the University of Oklahoma, Oklahoma State University, and the U.S. Environmental Protection Agency (U.S. EPA), began an investigation

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of a municipal landfill near Norman, Oklahoma (Christenson et al. 1999). This unlined landfill originally was an open dump established in the 1920s on an alluvial plain adjacent to the Canadian River, the channel of which has shifted through time (Figure 1). Wastes in this landfill consist primarily of residential and commercial solid waste, although hazardous wastes have been disposed of in this landfill (Dixon 1992). The landfill was closed in 1985, covered with a clay cap, and vegetated. The vertical and horizontal extent of the leachate plume was determined by geophysical electromagnetic induction surveys performed on the alluvial plain surrounding the Norman Landfill in January and February 1995, and by sampling of hundreds of monitoring wells at the site (Bisdorf and Lucius 1999). Measurements of specific conductance of water samples collected from October 1995 to November 1997 were used to generate a horizontal and vertical distribution of the leachate plume (Figures 1 and 2) (Becker 2001). Additional geochemical and hydrologic data indicate the plume is moving in the direction of ground water flow and has migrated beyond a wetland that is present ~394 ft (120 m) south of the landfill (Cozzarelli et al. 1996). In 2000, a set of four water samples was collected from a transect of wells, A-A′, located along a presumed flowpath in the leachate plume (Figures 1 and 2). In addition, one sample was collected from a well located upgradient of the landfill (Figure 1). These water samples were analyzed for 76 OWCs using research methods developed by the USGS. The purpose of this article is to summarize the OWC results from this study.

Site Selection and Sampling

Ground water samples were collected from five multilevel monitoring wells proximal to the Norman Landfill in central Oklahoma (Figure 1). Four of the wells are located within a transect along a presumed flowpath in the center of the leachate plume. The locations of these wells range from ~3 ft (1 m) to 574 ft (175 m) from the landfill (Table 1).

A fifth well (well NPD) is located upgradient of the landfill in a large grassy field at the Norman Police Department pistol range and is not in the leachate plume (Figure 1). The field has limited use and is situated near the City of Norman's waste water treatment plant, waste transfer station, and animal shelter. Well depths ranged from 10.69 ft (3.26 m) to 20.65 ft (6.29 m) (Table 1).

All samples were collected on September 6, 2000, by USGS personnel using protocols and procedures designed to obtain representative ground water samples (Koterba et al. 1995). Following collection, samples were immediately chilled and sent to the laboratory for analysis. To minimize contamination of samples, personal care products (i.e., insect repellents, colognes, etc.), caffeinated products, and tobacco were not used during sample collection and processing.

Analytical Methods

Four analytical methods were used to determine the environmental extent of 76 OWCs in these ground water samples. The analyzed compounds can be divided into groups based on their association with human, industrial, and agricultural waste waters, and include antibiotics, prescription and nonprescription drugs, steroids, personal care products, products of oil use and combustion, and other extensively used chemicals (Table 2). Twenty-one antibiotic compounds were extracted and analyzed by tandem solid-phase extraction (SPE) and single quadrapole, liquid chromatography/mass spectrometry with electrospray ionization set in positive mode and selected ion monitoring (SIM) (Kolpin et al. 2002; Meyer et al. 2000). Eighteen human prescription and nonprescription drugs, and selected metabolites, were extracted by SPE and measured by high performance liquid chromatography/mass spectrometry (HPLC/MS) using a polar reverse-phase octylsilane (C8) HPLC column (Cahill et al., in review). Forty-three OWCs were extracted using continuous liquid-liquid extraction and measured by capillary-column gas chromatography/mass spectrometry (GC/MS) (Brown et al. 1999). Two steroid compounds were analyzed by GC/MS following a derivatization process (Barber et al. 2000). Eight compounds were analyzed by more than one method (Table 2).

Results and Discussion

Landfills as a source of OWCs may be highly variable, depending on a variety of factors such as length of operation, whether the landfill is currently active, and types of waste being stored. For example, if biosolids from waste water

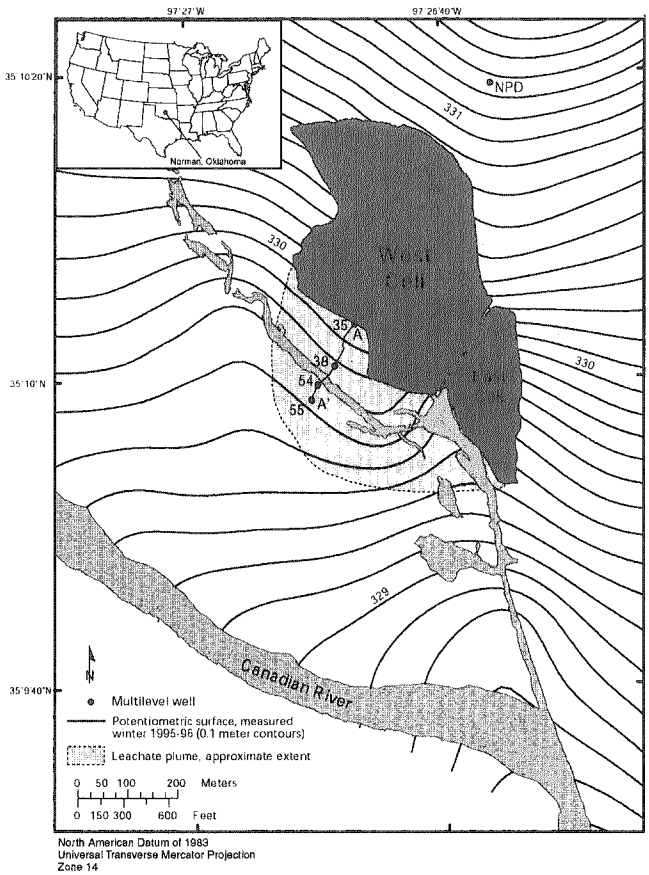

Figure 1. Location of east and west cells of Norman Landfill, location of well sampled along transect A-A′, and approximate extent of the leachate plume (modified from Christenson et al. [1999]).

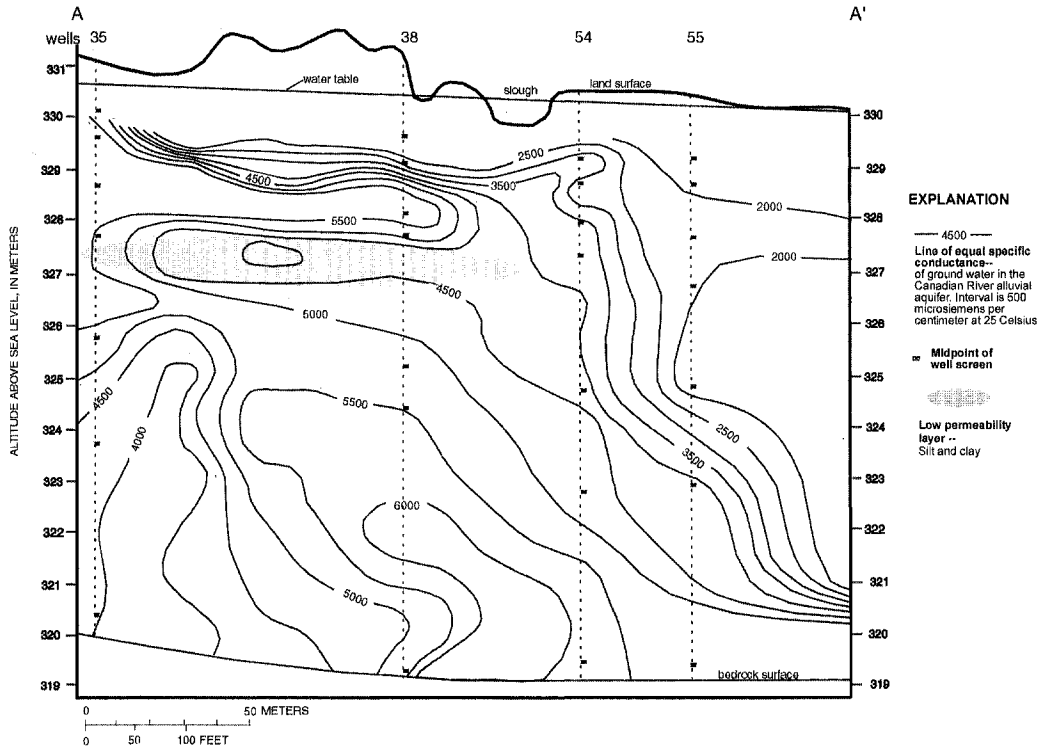

Figure 2. Distribution of specific conditions in the leachate plume along transect A-A′ of multilevel wells at Norman Landfill. Specific conductance measurements were collected from October 1995 to November 1997. Altitude of water table was measured January 29, 1998 (modified from Becker [2001]).

treatment plants are stored in a landfill, the likelihood of detections of pharmaceuticals in the leachate plume would be increased. During this study, 22 of the 76 OWCs were detected in at least one of the five ground water samples collected (Table 3).

Those detected compounds represent a wide range of uses and origins (five detergent metabolites, three steroids, three antioxidants, three polycyclic aromatic hydrocarbons, two disinfectants, two plasticizers, one antibiotic, one flame-retardant, one insect repellent, and a metabolite of a nonprescription drug). The most frequently detected compounds were cholesterol (100%), DEET (80%), tri(2-chloroethyl) phosphate (80%), and cotinine (60%). Of the 43 detections (Table 3), 12 exceeded 1 μg/L. Of the two detected compounds with federal drinking water regulations, only bis(2-ethylhexyl) phthalate exceeded its maximum contaminant level of 6 μg/L (U.S. Environmental Protection Agency 2002).

The samples collected for this investigation are part of a larger, nationwide reconnaissance of the occurrence of OWCs in ground water (Barnes et al. 2003). To determine the effect, if any, of field equipment and procedures on the concentrations of OWCs in water samples, field blanks, made from laboratory-grade organic-free water, were submitted for ~6% of the sites sampled as part of this larger study. These quality assurance/quality control samples were analyzed for all of the 76 OWCs. One field blank had detectable concentrations of 1,4-dichlorobenzene and naphthalene, with both detections being near their respective reporting levels.

Table 1
Well Depths, Distance from the Norman Landfill, and Field Parameters at Time of Sample Collection (September 6, 2000)

Well	Well Depth (ft/m)	Distance from Landfill[a] (ft/m)	Specific Conductance (μS/cm)	Water Temperature (°C)	pH
35	18.63/5.68	3.28/1	4720	21.5	6.72
38	20.65/6.29	305/93	6160	17.1	7.04
54	10.69/3.26	469/143	4130	19.8	6.16
55	12.68/3.86	574/175	3920	20.3	6.42
NPD	20.43/6.23	–534[b]/–163[b]	1530	17.8	6.30

[a]Distance is to edge of landfill along the presumed flowpath, which is not necessarily the absolute shortest distance to the landfill.
[b]Negative value represents a distance upgradient of the landfill.

Table 2
76 Compounds Analyzed in the Five Water Samples Collected Near the Norman Landfill, 2000 (reporting level is in μg/L)

Compound Name	CASRN	Use	Reporting Limit
1,4-dichlorobenzene[c]	106-46-7	deodorizer	0.03
2,6-di-*tert*-butylphenol[c]	128-39-2	antioxidant	0.08
2,6,di-*tert*-butyl-1,4-benzoquinone[c]	719-22-2	antioxidant	0.5
5-methyl-1H-benzotriazole[c]	136-85-6	antiocorrosive	0.10
acetaminophen[b]	103-90-2	antipyretic	0.009
acetophenone[c]	98-86-2	fragrance	0.15
anthracene[c]	120-12-7	PAH	0.05
benzo[a]pyrene[c]	50-32-8	PAH	0.07
3-*tert*-butyl-4-hydroxy anisole[c]	25013-16-5	antioxidant	0.12
butylated hydroxy toluene[c]	128-37-0	antioxidant	0.08
bis(2-ethylhexyl) phthalate[c]	117-81-7	plasticizer	2.5
bisphenol A[c]	80-05-7	plasticizer	0.09
caffeine[b,c]	58-08-2	stimulant	0.014, 0.08
carbaryl[c]	63-25-2	insecticide	0.06
carbodox[a]	6804-07-5	antibiotic	0.10
chlorpyrifos[c]	2921-88-2	insecticide	0.02
chlortetracycline[a]	57-62-5	antibiotic	0.05
cholesterol[c,d]	57-88-5	plant/animal steroid	0.005, 1.5
cimetidine[b]	51481-61-9	antacid	0.007
ciprofloxacin[a]	85721-33-1	antibiotic	0.02
cis-chlordane[c]	5103-71-9	insecticide	0.04
codeine[b,c]	76-57-3	analgesic	0.24, 0.1
coprostanol[c,d]	360-68-9	fecal steroid	0.005, 0.6
dehydronifedipine[b]	67035-22-7	antianginal	0.01
cotinine[b,c]	486-56-6	nicotine metabolite	0.023, 0.08
diazinon[c]	333-41-5	insecticide	0.03
dieldrin[c]	60-57-1	insecticide	0.08
diethylphthalate[c]	84-66-2	plasticizer	0.25
digoxigenin[b]	1672-46-4	digoxin metabolite	0.008
diltiazem[b]	42399-41-7	antihypertensive	0.012
1,7-dimethylxanthine[b]	611-59-6	caffeine metabolite	0.018
doxycycline[a]	564-25-0	antibiotic	0.1
enrofloxacin[a]	93106-60-6	antibiotic	0.02
erythromycin-H_2O[a]	114-07-8	erythromycin metabolite	0.05
ethanol,2-butoxy-phosphate[c]	78-51-3	plasticizer	0.2
fluoranthene[c]	206-44-0	PAH	0.03
fluoxetine[b]	54910-89-3	antidepressant	0.018
gemfibrozil[b]	25812-30-0	antihyperlipidemic	0.015
ibuprofen[b]	15687-27-1	antiinflammatory	0.018
lincomycin[a]	154-21-2	antibiotic	0.05
lindane[c]	58-89-9	insecticide	0.05
methyl parathion[c]	298-00-0	insecticide	0.06
4-methyl phenol[c]	106-44-5	disinfectant	0.04
naphthalene[c]	91-20-3	PAH	0.02
N,N-diethyltoluamide[c]	134-62-3	insect repellent	0.04
4-nonylphenol[c]	84852-15-3	nonionic detergent metabolite	0.5
4-nonylphenol monoethoxylate[c]	n/a	nonionic detergent metabolite	1.0
4-nonylphenol diethoxylate[c]	n/a	nonionic detergent metabolite	1.1
4-octylphenol monoethoxylate[c]	n/a	nonionic detergent metabolite	0.1
4-octylphenol diethoxylate[c]	n/a	nonionic detergent metabolite	0.2
norfloxacin[a]	70458-96-7	antibiotic	0.02
oxytetracycline[a]	79-57-2	antibiotic	0.1
phenanthrene[c]	85-01-8	PAH	0.06
phenol[c]	108-95-2	disinfectant	0.25
pyrene[c]	129-00-0	PAH	0.03
ranitidine[b]	66357-35-5	antacid	0.01
roxithromycin[a]	80214-83-1	antibiotic	0.03

Table 2 (continued)

Compound Name	CASRN	Use	Reporting Limit
salbutamol[b]	18559-94-9	antiasthmatic	0.029
sarafloxacin[a]	98105-99-8	antibiotic	0.02
stigmastanol[c]	19466-47-8	plant steroid	2.0
sulfadimethoxine[a]	122-11-2	antibiotic	0.05
sulfamerazine[a]	127-79-7	antibiotic	0.05
sulfamethazine[a]	57-68-1	antibiotic	0.05
sulfamethizole[a]	144-82-1	antibiotic	0.05
sulfamethoxazole[a,b]	723-46-6	antibiotic	0.05, 0.023
sulfathiazole[a]	72-14-0	antibiotic	0.10
tetrachloroethylene[c]	127-18-4	solvent, degreaser	0.03
tetracycline[a]	60-54-8	antibiotic	0.10, 0.05
triclosan[c]	3380-34-5	antimicrobial disinfectant	0.05
tri(2-chloroethyl) phosphate[c]	115-96-8	fire retardant	0.04
tri(dichlorisopropyl) phosphate[c]	13674-87-8	fire retardant	0.1
trimethoprim[a,b]	738-70-5	antibiotic	0.03, 0.014
triphenyl phosphate[c]	115-86-6	plasticizer	0.1
tylosin[a]	1401-69-0	antibiotic	0.05
virginiamycin[a]	21411-53-0	antibiotic	0.10
warfarin[b]	81-81-2	anticoagulant	0.001

[a]Method 1: LCMS
[b]Method 2: SPE HPLC/MS
[c]Method 3: CCLE GC/MS
[d]Method 4: CCLE, derivitization, GC/MS
Compounds suspected of being hormonally active are in bold (National Research Council 1999; Foran et al. 2000).
CASRN—Chemical Abstracts Service registry number
PAH—polycyclic aromatic hydrocarbon
n/a—not available

Only cholesterol (a naturally occurring steroid) was detected in the well upgradient of the landfill (Table 3). This relative lack of detection of OWCs in upgradient ground water, along with the fact that no other known ground water sources of OWCs exist in this area, indicate the landfill is the source of most of the detected OWCs in the leachate plume. The sites closest to the landfill (wells 35 and 38) had much greater numbers of detections and concentrations of OWCs than sites more distant to the landfill (wells 54 and 55) (Figures 1 and 3). Although fate and transport analyses are beyond the scope of this investigation, factors such as sorption, degradation, time of travel, proximity to source, and dilution probably affect OWC concentrations as water flows away from the landfill.

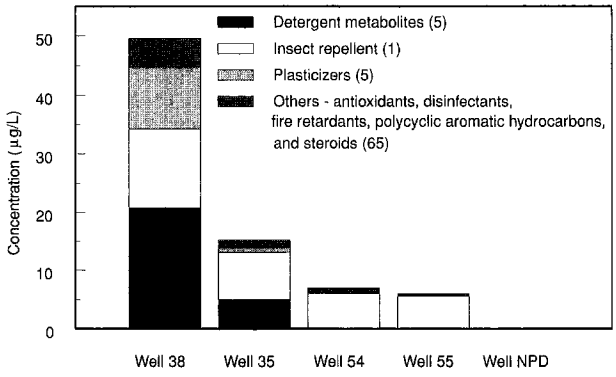

Figure 3. Total measured concentrations of organic waste water contaminant general use groups, by site. Number of compounds in each group shown in legend.

Closure of the landfill in 1985 defines the end to potential input of source materials. Therefore, detections of OWCs in the landfill leachate in 2000 indicate that some have persisted in ground water for several decades. Although well 35 is the closest to the edge of the landfill, well 38 had the most detections and the greatest concentrations of OWCs. Water from well 38 also had much greater specific conductance than the others at the time of sample collection and is consistent with Becker's (2001) conclusion that well 38 is completed in the most concentrated part of the plume. Other reports have indicated that shortly after a landfill closes, the leachate concentration peaks, then slowly declines with time (McBean et al. 1995). The sample from well 38 may represent water leaving the landfill close to the peak of leachate seepage; therefore, concentrations of OWCs were greatest at that location at the time of sample collection.

To increase understanding of the results for this study, the 22 compounds detected were divided into four groups based on their general use category (Tables 2 and 3, and Figure 3). Three groups (detergent metabolites, plasticizers, and others) showed substantially decreased concentrations and numbers of detections as distance from the landfill increased. In contrast, DEET is much more persistent and is the only compound with a concentration greater than 1 µg/L in samples farthest from the landfill (wells 54 and 55). Other research has shown that DEET can be transported substantial distances from potential sources (Hendriks et al. 1994; Wiegel et al. 2002). Therefore, the greatest OWC contamination from landfills appears to be nearest to landfills; however, some OWCs have the potential to be transported

Table 3
Analytical Results of Ground Water Sites Sampled for 76 Organic Waste Water Contaminants (concentrations in μg/L)

Compound Name	Well 35	Well 38	Well 54	Well 55	Well NPD
cholesterol	0.042	0.044	0.022	0.039	0.022
coprostanol	0.074	0.057	< 0.005	< 0.005	< 0.005
cotinine	0.13	< 0.05	0.12	0.10	< 0.05
2,6-di-*tert*-butylphenol	< 0.15	0.23	< 0.08	< 0.08	< 0.15
2,6-di-*tert*-butyl-1,4-benzoquinone	0.4[a]	0.9	< 0.5	< 0.5	< 0.6
anthracene	< 0.06	< 0.06	0.02[d]	< 0.05	< 0.05
3-*tert*-butyl-4-hydroxy anisole	0.2[a]	< 0.12	< 0.12	< 0.12	< 0.12
bis(2-ethylhexyl) phthalate	< 2.5	250[b]	< 2.5	< 2.5	< 2.5
bisphenol A	0.84	0.50	< 0.09	< 0.09	< 0.09
fluoranthene	< 0.03	< 0.03	0.01[d]	< 0.03	< 0.03
lincomycin	0.10	< 0.05	< 0.05	< 0.05	< 0.05
4-methyl phenol	0.19	0.49	< 0.04	< 0.04	< 0.06
naphthalene	0.063	0.09	< 0.02	< 0.02	< 0.025
N,N-diethyltoluamide	8.1	13	6.1	5.5	< 0.08
4-nonylphenol	1[c]	3[c]	< 0.5	< 0.5	< 0.70
4-nonylphenol monoethoxylate	3[c]	7[c]	< 1.00	< 1.00	< 1.00
4-nonylphenol diethoxylate	< 1.10	10[c]	< 1.10	< 1.10	< 1.10
4-octylphenol monoethoxylate	0.4[c]	1[c]	< 0.10	< 0.10	< 0.12
4-octylphenol diethoxylate	0.2[c]	0.3[c]	< 0.20	< 0.20	< 0.20
stigmastanol	< 2.0	2[a]	< 2.0	< 2.0	< 2.0
triclosan	< 0.05	0.21	< 0.04	< 0.04	< 0.05
tri(2-chloroethyl) phosphate	0.36	0.74	0.25	0.22	< 0.04

[a]Concentration estimated—average recovery < 60%
[b]Concentration estimated and changed to 10 μg/L (five times the reporting level) for summary statistics; value greater than highest point on calibration curve
[c]Concentration estimated—reference standard prepared from a technical mixture
[d]Concentration estimated—value less than reporting level
Compounds suspected of being hormonally active are in bold (National Research Council 1999; Foran et al. 2000).

substantial distances away from the sources. Similar results have been reported previously (Eckel et al. 1993; Holm et al. 1995).

Conclusions

The results of this study confirm previous studies documenting landfills as a source of OWCs to ground water (Albaiges et al. 1986; Eckel et al. 1993; Holm et al. 1995). Only cholesterol (a naturally occurring compound) was detected in the well upgradient of the landfill. The numbers and concentrations of OWCs generally decreased with distance from the landfill. Select compounds, i.e., cotinine, DEET, tri(2-chloroethyl) phosphate, however, were detected through the entire length of the leachate plume being investigated. This study has shown that persistence, transformation, and transport of some OWCs occurs in this ground water flow system and the landfill has been the source of OWCs to ground water for many years.

Acknowledgments

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