

Pharmaceuticals, Personal Care Products, and Endocrine Active Chemical Monitoring in Lakes and Rivers: 2013

Contaminants that were detected in a 2008 sampling of select Minnesota lakes are compared to a follow-up 2013 study of the same locations with an expanded list of pharmaceuticals and other chemicals that behave like hormones.



Authors

Mark Ferrey

Contributors/acknowledgements

This report contains the results of a study that characterizes the presence of unregulated contaminants in Minnesota's lakes and rivers. The study was made possible through funding by the Minnesota Clean Water Fund.

The Minnesota Pollution Control Agency (MPCA) thanks the following for assistance and advice in designing and carrying out this study: Harold Wiegner for invaluable help in collecting samples; Kirsten Anderson and Richard Grace of AXYS Analytical Services for their expert help in developing the list of analytes for this study and logistics to make it a success.

The MPCA is reducing printing and mailing costs by using the Internet to distribute reports and information to wider audience. Visit our website for more information.

MPCA reports are printed on 100% post-consumer recycled content paper manufactured without chlorine or chlorine derivatives.



Minnesota Pollution Control Agency

520 Lafayette Road North | Saint Paul, MN 55155-4194 | www.pca.state.mn.us | 651-296-6300

Toll free 800-657-3864 | TTY 651-282-5332

This report is available in alternative formats upon request, and online at www.pca.state.mn.us.

Document number: tdr-g1-18

Contents

Summary	1
Introduction	1
Sampling locations and procedures.....	1
Results and discussion	2
Lakes.....	2
Streams.	2
Comparisons with previous studies.	2
References	5
Appendix A: Lake and wastewater treatment plant descriptions and location information	23
Appendix B: Sample collection	18
Appendix C: Laboratory analytical methods and quality assurance	27
Appendix D: Analytical	30

Glossary of terms

mg/L	milligrams per liter, or parts per million (ppm)
µg/L	micrograms per liter, or parts per billion (ppb)
ng/L	nanograms per liter, or parts per trillion (ppt)
EAC	Endocrine active chemical
PPCPs	Pharmaceuticals and personal care products
WWTP	Wastewater treatment plant

Summary

Surface water from 11 lakes and 4 streams was analyzed for pharmaceuticals, personal care products (PPCPs), and other micropollutants. The stream samples were taken from above and below wastewater treatment plant effluent outfall locations. Several pharmaceuticals were frequently detected, including the anti-diabetic medicine metformin, numerous antibiotics, antidepressants, blood pressure medications, illicit drugs, lipid regulators, and the x-ray contrasting pharmaceutical iopamidol. Many of the chemicals that were detected, such as bisphenol A (BPA), are either known or suspected endocrine active chemicals (EACs). The results of this study, while largely consistent with a 2008 study of these same locations, expand our understanding of the varieties of contaminants present in our aquatic environments.

Introduction

Concern over the presence of PPCPs and EACs is reflected in the numerous studies of these chemicals in the aquatic environment. While initial sampling efforts in Minnesota have focused primarily on locations where there was an obvious source of these contaminants, such as wastewater treatment plants (1, 2), more recent studies by the Minnesota Pollution Control Agency (MPCA) have expanded this inquiry to include a large number of randomly selected lakes (3) and rivers (4) many of which have no direct sources of these contaminants. These studies show that pharmaceuticals and other micropollutants are more ubiquitous in surface water than was previously suspected.

In 2008, the MPCA, the U.S. Geological Survey, and St. Cloud State University sampled water from selected lakes representing various trophic levels and land use settings. That study revealed that lakes contained many of the same pharmaceutical contaminants found downstream of wastewater treatment plants (WWTPs), including lakes without lakeshore development. Fish exhibited the effects of endocrine disruption in all of the lakes (5).

In 2013, these same 11 lakes were sampled again, in addition to water upstream and downstream of four of the WWTPs that were part of the 2009 WWTP study (6). Some of the contaminants discovered in lake water in 2008 were again detected at similar concentrations in 2013, and several pharmaceuticals that were not analyzed in 2008 were detected in 2013.

Sampling locations and procedures

Locations that were included in this study are shown in Figure 1. The lake locations are described in greater detail in the 2008 report to the Minnesota Legislature (7) and in Appendix A. Three of these lakes reflect urban, sewer residential land use (Owasso, Budd, Cedar); four have unsewered residential development (Sullivan, White Sand, Red Sand, and Kabetogama); and four are in areas with very limited (Shingobee and Stewart) or no lakeshore development (Elk and Northern Light).

Surface water upstream and downstream of the outfall of four WWTPs (Hinckley, Marshall, Sauk Center, and the Twin Cities Metro Plant) was sampled. These locations were included in the 2009 study of 25 WWTPs (6) and details on these sampling locations can be found in [Appendix A](#).

Surface water was collected for analysis once from each location during the summer of 2013. Grab samples of water were collected by immersing sample bottles approximately six inches beneath the surface. Powder-free nitrile gloves were worn while sampling and handling sample bottles. Sample bottles were rinsed three times with the surface water prior to filling completely with sample.

After filling, bottles were put in coolers and chilled on ice. Samples were shipped overnight to AXYS Analytical Services in Vancouver, British Columbia, with a maximum holding time of seven days, and analyzed for the chemicals listed in Table 1. A detailed description of sampling procedures is included in [Appendix B](#). The analytical methods and quality assurance detail is included in [Appendix C](#).

Results and discussion

Lakes

[Figure 2](#) shows the frequency at which contaminants were detected in lakes in 2013. A total of 27 pharmaceuticals, disinfectants, or other contaminants were found. As in other studies, DEET was the most frequently detected chemical, appearing in 91% of the water samples at a maximum concentration of 103 parts per trillion (ppt) ([Table 2](#)). Cotinine (a metabolite of nicotine) and iopamidol (an x-ray contrasting agent) were both detected in 73% of the lakes at maximum concentrations of 42 and 510 ppt, respectively. Bisphenol A and metformin were both found at 36% of the locations at maximum concentrations of 36 ppt and 18 ppt, respectively, with the steroidal hormone androstenedione detected in 27% of the lakes up to 5 ppt. Several other pharmaceuticals were detected less frequently, including the disinfectants triclosan and triclocarban as well as the antibiotics erythromycin, ciprofloxacin, sulfachloropyridazine, and carbadox.

Streams

A total of 56 PPCPs and other chemicals were detected in water samples collected downstream of the four WWTPs included in this study. Sixteen of these chemicals were found in every surface water sample that was collected downstream of the facilities ([Figure 3](#)), including antidepressants, antibiotics, and several pharmaceuticals used to treat hypertension. In upstream water, 33 were detected ([Figure 4](#)), with metformin, BPA, and cotinine found at all four upstream locations.

Six chemicals out of the total number of 56 chemicals detected - BPA, carbadox, virginiamycin, DEET, methylprednisolone, and triclosan – were detected more frequently in upstream water than downstream water. Eight chemicals – BPA (at 237 ppt), carbadox (at 3 ppt), fluoxetine (at 12 ppt), sulfamethazine (at 31 ppt), virginiamycin (at 68 ppt), methylprednisolone (at 6 ppt), moxifloxacin (at 16 ppt), and triclosan (at 10 ppt) – were present at higher concentrations at upstream locations than at downstream locations from at least one WWTP location ([Table 3](#)).

Complete analytical results are found in [Appendix D](#).

Comparisons with previous studies

The 11 lakes in this study were initially sampled in 2008 (5); Tables 4 through 14 show data for these lakes from 2008 and 2013. Expanded laboratory methods made it possible to look for a greater number of analytes in 2013 than in 2008, particularly for pharmaceuticals, though there were some chemicals analyzed in 2008 that were not in 2013.

In 2008, DEET was detected in every lake, up to a concentration of 579 ppt, whereas in 2013 it was detected in 10 of the 11 lakes and at a maximum concentration of 103 ppt. Caffeine and BPA were also found in these lakes in both 2013 and 2008.

Figure 5 compares the results of this study with the 2008 study and the 2012 National Lake Assessment (NLA) study of 50 randomly selected Minnesota lakes (3). Together, these studies show that DEET, BPA, androstenedione, amitriptyline, and caffeine are consistently the most frequently detected in lake water. The greater number of detections for estrone and androstenedione in 2008 is likely due to the lower analytical detection limits for steroidal hormones for that particular study.

More chemicals were detected in lakes in the 2012 NLA study than in 2013. This is probably due to the greater number of lakes included in the 2012 study and thus the greater chance of detecting a particular analyte. However, cocaine, carbadox, and amitriptyline, which were detected in roughly a third of the lakes in 2012, were not detected as frequently in 2013.

The results from 2013 demonstrate again that even remote lakes in Minnesota contain a surprising variety of pharmaceuticals that is similar to the profile of chemicals in lakes with shoreline residential development. Elk Lake, in Itasca State Park, contained DEET, cotinine, metformin, colchicine, and BPA. Northern Light Lake, north of Grand Marais and just outside the Boundary Waters Canoe Area Wilderness (BWCAW), contained DEET, BPA, diltiazem, the antibiotics ciprofloxacin, erythromycin, trimethoprim, and clinafloxacin. Results from the 2008 study, the 2012 NLA study, and a 2009 study of four remote lakes in Itasca State Park and Voyageur's National Park consistently indicate that relatively isolated lakes often contain contaminants common to those waters more influenced by human activity.

This is the first study in Minnesota that included iopamidol, an x-ray contrasting agent, in the analysis of Minnesota surface water samples. It was detected in 73% of the lakes ([Figure 2](#)), both in urban settings as well as in lakes with minimal development. The highest concentration was detected in Lake Kabetogama, near the U.S. - Canada border, at 510 ppt. It was also found in 50% of the samples collected from downstream of WWTPs up to 1,230 ppt and at one upstream location at 356 ppt ([Table 3](#)). Diatrizoic acid, another x-ray contrasting agent, was found in one water sample downstream of a WWTP at 91 ppt.

Three of the pharmaceuticals that were detected upstream of WWTPs- the antibiotics virginiamycin, sulfamethazine, and carbadox -are used in raising livestock, possibly indicating upstream agricultural sources of these chemicals.

Pharmaceuticals that were detected in this study were grouped according to their physiologic endpoint ([Tables 15, 16](#)). The Anatomical Therapeutic Classification (ATC) system, developed by the World Health Organization (8), provides a means for grouping the variety of pharmaceuticals that were found in surface water according to their intended physiological mode of action.

In the lakes, there were 4 anti-infectives (in addition to the veterinary antibiotic carbadox, which does not have an ATC designation). Two drugs that were detected affect the musculo-skeletal system. Three medications affect the nervous system, and 2 affect the cardiovascular system. Two drugs are genito-urinary specific drugs, 2 are dermatologicals, and 1 is an antiparasitic. Nine of the 27 chemicals found in the 11 lakes are known or suspected EACs.

For the WWTPs, nine cardiovascular drugs were detected. Ten drugs were found that are systemic anti-infectives, 10 that affect the nervous system, 6 respiratory medications, and 4 that are typically prescribed for sensory organs such as eye medications. Two x-ray contrasting agents, 3 alimentary or metabolic drugs, and one musculoskeletal drug were also found. Several others without ATC designations, including the antibiotic carbadox; amitriptyline, an antidepressant; and the illicit drug methamphetamine as well as the breakdown product of cocaine, benzoylecgonine, were also found.

The presence of these contaminants in lake water is often explained by or correlated with lakeshore development and accompanying septic drain fields, agricultural row cropping, stormwater runoff, or livestock operations. However, it is not clear how these chemicals can be reaching lakes that lack shoreline development or other obvious sources of contamination. The 2012 NLA study of 50 lakes

revealed that roughly a third of them, some without development or public access, contained cocaine, amitriptyline, and carbadox. The atmospheric transport of fine particulate matter, to which drugs or other chemicals can be attached, and its subsequent deposition in surface water, is a possible explanation for the widespread presence of some of these chemicals in lakes. Indeed, the routine detection of cocaine in air samples (9) demonstrates that atmospheric transport is a plausible route for chemicals such as the ones detected in this study to enter surface water.

The effects that these contaminants are having on aquatic ecosystems are not entirely clear, though several studies have demonstrated genetic, cellular, physiological, and population level effects with even very small amounts of the chemicals reported in this study. Antidepressants, for example, have a dramatic effect on the reproductive cycles of freshwater mussels (10), can accumulate in fish tissue (11), and cause behavioral changes in fathead minnows (12). Mixtures of pharmaceuticals at concentrations commonly detected in surface water can inhibit cell proliferation (13), and water samples collected from downstream of wastewater treatment plants alter the expression of hundreds of different genes found in fish (14). Commonly used analgesics are known to be acutely toxic in particular bird species (15) and to bivalves (16). Several of these chemicals, such as nonylphenol or triclosan, have hormone-like properties and can exert effect at extremely low concentrations (17). Thus, it is possible that these contaminants are adversely affecting fish and wildlife in ways we are only beginning to understand.

There is little information about human health effects from exposures to the very low concentrations of many of the chemicals reported here. The Minnesota Department of Health (MDH) Drinking Water Contaminants of Emerging Concern (CEC) Program has developed human health-based water guidance values for some of the detected chemicals such as BPA, DEET, sulfamethoxazole, and triclosan (<http://www.health.state.mn.us/divs/eh/risk/guidance/gw/table.html>). The MDH is also concerned about low levels of exposure to active pharmaceutical ingredients (APIs) due to their very potent actions on very specific biological targets and the lack of testing for all possible effects over a lifetime of exposure to low concentrations. The MDH CEC Program Pharmaceutical Screening Project will provide a method for deriving screening water concentrations based on the lowest therapeutic dose, toxicological information contained on the drug label, and assumptions about drinking water consumption for the most-prescribed APIs in the United States, including many that MPCA is also researching. This will provide human-health risk context for occurrence data and assist in prioritizing research needs. The MDH anticipates releasing the Pharmaceutical Screening Project report by June 2015.

This and previous monitoring studies have shown that some of the chemicals we monitor are found in surface water more frequently than others. This observation can focus the direction of future studies, such as measuring or assessing the impacts of particular contaminants from wastewater sources. Combined with the recent advances in understanding the genetic effects that particular chemicals have on fish and wildlife, this may lead to methods that identify surface water locations of greatest risk and concern, or it might allow a better correlation of effect with the presence of particular contaminants in surface water. Finally, our increased understanding of which chemicals are of most concern may lead to simplified prevention strategies or wastewater treatment options that are most cost-effective in protecting aquatic ecosystems.

References

1. Lee K, Schoenfuss HL, Barber LB, Blazer VS, Kiesling RL, Ferrey M. 2010. Endocrine active chemicals and endocrine disruption in Minnesota streams and lakes - implications for aquatic resources, 1994-2008. U.S. Geological Survey Scientific Investigations Report 2010-5107, 47 p. with Appendixes.
2. Lee KE, Schoenfuss HL, Jahns ND, Brown GK, Barber LB. 2008. Alkylphenols, other endocrine-active chemicals, and fish responses in three streams in Minnesota-Study design and data, February-September 2007. U.S. Geological Survey Data Series 405, 44 p. with Appendixes.
3. Ferrey ML. 2012. Pharmaceuticals and Endocrine Active Chemicals in Minnesota Lakes. Minnesota Pollution Control Agency, Document no. tdr-g1-16. 47 p.
4. Ferrey M, Streets S, Lueck A. 2013. Pharmaceuticals and Personal Care Products in Minnesota's Rivers and Streams: 2010. Minnesota Pollution Control Agency, Document no. tdr-g1-17, 36 p.
5. Writer JH, Barber LB, Brown GK, Taylor HE, Kiesling RL, Ferrey ML, Jahns ND, Bartell SE, Schoenfuss HL. 2010. Anthropogenic tracers, endocrine disrupting chemicals, and endocrine disruption in Minnesota lakes. *Sci Total Environ* 409:100-11.
6. Lee KE, Langer SK, Barber LB, Writer JH, Ferrey M, Schoenfuss HL, Gray JL, Revello RC, Martinovic D, Woodruff OP, Keefe SH, Brown GK, Taylor HE, Ferrer I, Thurman EM. 2011. Endocrine active chemicals, pharmaceuticals, and other chemicals of concern in surface water, wastewater effluent, and bottom sediment in Minnesota - site description, methods, and data. U.S. Geological Survey Data Series 575, 54 p with Appendixes.
7. Ferrey M, Preimesberger A, Schoenfuss HL, Kiesling RL, Barber LB, Writer JH. 2008. Statewide Endocrine Disrupting Compound Monitoring Study, 2007-2008. Minnesota Pollution Control Agency, Document no. tdr-g1-08, 87 p.
8. World Health Organization Collaborating Centre for Drug Statistics Methodology 2015. ATC/DDD Index 2015. [cited December 29, 2014] Available from: http://www.whocc.no/atc_ddd_index/.
9. Cecinato A, Balducci C, Nervegna G. 2009. Occurrence of cocaine in the air of the world's cities: An emerging problem? A new tool to investigate the social incidence of drugs? *Sci Total Environ* 407:1683-90.
10. Fong PP. 1998. Zebra mussel spawning is induced in low concentrations of putative serotonin reuptake inhibitors. *Biol Bull* 194:143-9.
11. Ramirez AJ, Brain RA, Usenko S, Mottaleb MA, O'donnell JG, Stahl LL, Wathen JB, Snyder BD, Pitt JL, Perez-Hurtado P, Dobbins LL, Brooks BW, Chambliss CK. 2009. Occurrence of pharmaceuticals and personal care products in fish: results of a national pilot study in the United States. *Environ Toxicol Chem* 28:2587-97.
12. Painter MM, Buerkley MA, Julius ML, Vajda AM, Norris DO, Barber LB, Furlong ET, Schultz MM, Schoenfuss HL. 2009. Antidepressants at environmentally relevant concentrations affect predator avoidance behavior of larval fathead minnows (*Pimephales promelas*). *Environ Toxicol Chem* 28:2677-84.
13. Pomati F, Cotsapas CJ, Castiglioni S, Zuccato E, Calamari D. 2007. Gene expression profiles in zebrafish (*Danio rerio*) liver cells exposed to a mixture of pharmaceuticals at environmentally relevant concentrations. *Chemosphere* 70:65-73.

14. Martinović-Weigelt D, Mehinto AC, Ankley GT, Denslow ND, Barber LB, Lee KE, King RJ, Schoenfuss HL, Schroeder AL, Villeneuve DL. 2014. Transcriptomic effects-based monitoring for endocrine active chemicals: Assessing relative contribution of treated wastewater to downstream pollution. *Environ Sci Technol* 48:2385-94.
15. Oaks JL, Gilbert M, Virani MZ, Watson RT, Meteyer CU, Rideout BA, Shivaprasad HL, Ahmed S, Chaudhry MJ, Arshad M, Mahmood S, Ali A, Khan AA. 2004. Diclofenac residues as the cause of vulture population decline in Pakistan. *Nature* 427:630-3.
16. Parolini M, Binelli A. 2012. Sub-lethal effects induced by a mixture of three non-steroidal anti-inflammatory drugs (NSAIDs) on the freshwater bivalve *Dreissena polymorpha*. *Ecotoxicology* 21:379-92.
17. Streets S, Ferrey M, Solem L, Preimesberger A, Hoff P. 2008. Endocrine Disrupting Compounds: A Report to the Minnesota Legislature. Minnesota Pollution Control Agency, Document no. Irp-ei-1syo8.

Table 1. Chemicals analyzed in the 2013 surface water study

1,7-Dimethylxanthine	Dehydronifedipine	Ormetoprim
10-hydroxy-amitriptyline	Demeclocycline	Oxacillin
17 alpha-Dihydroequilin	Desmethyldiltiazem	Oxazepam
17 alpha-Estradiol	Desogestrel	Oxolinic Acid
17 alpha-Ethinyl-Estradiol	Diatrizoic acid	Oxycodone
17 beta-Estradiol	Diazepam	Oxytetracycline [OTC]
2-Hydroxy-ibuprofen	Digoxigenin	Paroxetine
4-Epianhydrochlortetracycline [EACTC]	Digoxin	Penicillin G
4-Epianhydrotetracycline [EATC]	Diltiazem	Penicillin V
4-Epichlortetracycline [ECTC]	Diphenhydramine	Prednisolone
4-Epioxytetracycline [EOTC]	Doxorubicin	Prednisone
4-Epitetracycline [ETC]	Doxycycline	Progesterone
4-Nonylphenol diethoxylates	Drospirenone	Promethazine
4-Nonylphenol monoethoxylates	Enalapril	Propoxyphene
4-Nonylphenols	Enrofloxacin	Propranolol
Acetaminophen	Equilenin	Ranitidine
Albuterol	Equilin	Rosuvastatin
Allyl Trenbolone	Erythromycin-H2O	Roxithromycin
Alprazolam	Estriol	Sarafloxacin
Amitriptyline	Estrone	Sertraline
Amlodipine	Etoposide	Simvastatin
Amphetamine	Flumequine	Sulfachloropyridazine
Amsacrine	Fluocinonide	Sulfadiazine
Androstenedione	Fluoxetine	Sulfadimethoxine
Androsterone	Fluticasone propionate	Sulfamerazine
Anhydrochlortetracycline [ACTC]	Furosemide	Sulfamethazine
Anhydrotetracycline [ATC]	Gemfibrozil	Sulfamethizole
Atenolol	Glipizide	Sulfamethoxazole
Atorvastatin	Glyburide	Sulfanilamide
Azathioprine	Hydrochlorothiazide	Sulfathiazole
Azithromycin	Hydrocodone	Tamoxifen
Benzoylcegonine	Hydrocortisone	Teniposide
Benztropine	Ibuprofen	Testosterone
Betamethasone	Iopamidol	Tetracycline [TC]
Bisphenol A	Isochlortetracycline [ICTC]	Theophylline
Busulfan	Lincomycin	Thiabendazole
Caffeine	Lomefloxacin	Trenbolone
Carbadox	Lomustine	Trenbolone acetate
Carbamazepine	Medroxyprogesterone Acetate	Triamterene
Carmustine	Melphalan	Triclocarban
Cefotaxime	Meprobamate	Triclosan
Chloramphenicol	Mestranol	Trimethoprim
Chlortetracycline [CTC]	Metformin	Tylosin
Cimetidine	Methylprednisolone	Valsartan
Ciprofloxacin	Metoprolol	Venlafaxine

Citalopram	Metronidazole	Verapamil
Clarithromycin	Miconazole	Virginiamycin M1
Clinafloxacin	Minocycline	Warfarin
Clonidine	Moxifloxacin	Zidovudine
Clotrimazole	Naproxen	3,4-methylenedioxy-N-methylamphetamine (MDMA)
Cloxacillin	Norethindrone	Amphetamine
Cocaine	Norfloxacin	Benzoylcegonine
Codeine	Norfluoxetine	Cocaine (MDH)
Colchicine	Norgestimate	Methamphetamine
Cotinine	Norgestrel	Morphine
Cyclophosphamide	Norverapamil	Pseudoephedrine/Ephedrine
Daunorubicin	Octylphenol	
DEET	Ofloxacin	

Table 2. Maximum concentrations and reporting limits of chemicals detected in lakes in 2013

Chemical	Maximum concentration (ppt)	Range of reporting limits (ppt)
Amitriptyline	0.503	0.301-1.23
Amphetamine	2.91	1.48-1.53
Androstenedione	5	2.00-2.81
Androsterone	128	19.8-107
Bisphenol A	35.8	1.68-2.36
Caffeine	69.7	14.9-16.2
Carbadox	5.45	1.49-29.0
Ciprofloxacin	36.8	16.7-38.7
Clinafloxacin	184	49.3-124
Colchicine	8.52	2.03-6.10
Cotinine	42.1	1.48-1.52
DEET	103	0.591-0.647
Diltiazem	1.19	0.3-1.70
Erythromycin-H2O	3.33	2.30-3.08
Estrone	24.6	3.96-4.23
Gemfibrozil	2.07	1.49-5.08
Glyburide	2.92	2.11-3.21
Iopamidol	510	79.3-257
Metformin	18.0	2.96-13.6
Naproxen	3.85	2.98-14.7
Norverapamil	0.187	1.49-0.186
Sertraline	12.7	0.397-0.453
Sulfachloropyridazine	3.83	1.52-5.54
Thiabendazole	12.7	1.49-1.60
Triclocarban	3.28	2.98-3.21
Trimethoprim	3.63	1.49-1.75
Triclosan	5.75	4.89-5.78

Table 3. Maximum concentration and reporting limits for chemicals detected in river surface water in 2013

Chemical	Maximum concentration(ppt)		Range of reporting limits (ppt)
	Upstream	Downstream	
10-hydroxy-amitriptyline		1.45	1.49-1.56
4-Nonylphenol diethoxylates		13	2.45-14.6
4-Nonylphenols		30.1	0.722-4.28
Albuterol	0.314	1.9	0.295-0.312
Alprazolam		0.339	0.296-0.339
Amitriptyline	0.471	0.724	0.296-1.39
Amphetamine		1.65	1.49-2.42
Atenolol	1.67	26.3	0.596-0.651
Benzoyllecgonine	0.469	4.14	0.297-0.469
Bisphenol A	237	6.98	1.68-2.36
Caffeine	37	80.1	14.8-15.3
Carbadox	3.23		1.48-7.53
Carbamazepine	7.62	35.7	1.49-1.56
Cimetidine		7.1	0.596-0.625
Ciprofloxacin		22.6	10.2-25.6
Citalopram		8.5	0.389-0.706
Clarithromycin		7.95	1.48-1.56
Cocaine	0.203	0.291	1.48-1.55
Codeine		4.4	0.295-3.12
Cotinine	5.19	8.32	1.48-3.03
DEET	11.2	20.3	0.591-0.647
Dehydronifedipine	0.72	1.08	0.597-0.623
Desmethyldiltiazem	0.27	3.33	1.49-1.56
Diatrizoic acid		91.2	39.4-41.5
Diltiazem	2.26	19.5	0.299-0.991
Diphenhydramine		6.61	0.593-0.623
Erythromycin-H2O	4.96	13.2	2.29-2.39
Fluoxetine	12.1	7.73	1.48-1.56
Furosemide		67	39.4-41.5
Gemfibrozil	2.33	11.1	1.49-4.94
Hydrochlorothiazide		25.4	19.8-208
Hydrocodone		5.2	1.49-1.56
Iopamidol	356	1230	79.3-257
Meprobamate		18.3	3.94-10.8
Metformin	53.9	434	2.96-13.6
Methamphetamine		3	0.75
Methylprednisolone	6.06		4.74-44.3
Metoprolol	7.79	51	6.57-9.68

Moxifloxacin	15.5	7.63	9.50-20.2
Naproxen		9.75	2.97-9.95
Ofloxacin		23.4	1.48-3.50
Oxycodone		8.8	0.596-1.45
Propranolol		8.6	1.97-2.08
Pseudoephedrine/Ephedrine		2.3	0.78-0.84
Ranitidine	0.744	24.2	0.596-2.08
Sertraline	0.482	3.48	0.398-4.15
Sulfadimethoxine	0.8	4.58	0.299-2.10
Sulfamethazine	30.9	25.6	1.02-5.81
Sulfamethoxazole	52.8	186	0.597-0.623
Theophylline		94.6	59.3-199
Thiabendazole		1.81	1.48-1.56
Triamterene	3.19	18.1	0.312-0.364
Triclosan	9.53		4.31-5.02
Trimethoprim	9.37	38.1	1.49-1.56
Tylosin		8.1	5.91-6.23
Valsartan	4.44	13.2	3.94-4.15
Venlafaxine	5.95	50.4	1.19-1.25
Verapamil		0.521	0.148-0.156
Virginiamycin M1	67.6	34	13.3-21.0

For tables 4 through 14:

All concentrations are in parts per trillion (ppt)

na: not analyzed

nd: not detected

qa: laboratory quality control issues prevented quantification

nm: not measured

<: not detected above the stated reporting limit

B: contaminant also detected in laboratory blank

Table 4. Cedar Lake

Chemical	2008	2013
17-β-estradiol	0.38	<4.2
2,6-di-tert-butyl-4-methylphenol	65	na
5-methyl-1H-benzotriazole	38	na
Androstenedione	nd	3.9
Benzoquinone	142	na
BPA	<10	6.4
Caffeine	87	69.7
Cholesterol	7842	na
Coprostanol	28	na
Cotinine	na	42.1

DEET	48	103
Estrone	qa	24.6
Iopamidol	na	212
Methylphenol	19.9	na
Norverapamil	na	0.19
Thiabendazole	na	12.7

Table 5. Budd Lake

Chemical	2008	2013
17- β -estradiol	0.04	<4
2,6-di-tert-butyl-4-methylphenol	68.7	na
2,6-di-tert-butylphenol	180	na
5-methyl-1H-benzotriazole	13.6	na
Androstenedione	na	5
BPA	37.9	<2.0
Caffeine	17.7	<16
Cholesterol	2536	na
Coprostanol	39.1	na
Cotinine	na	12.8
DEET	25.3	18.3
Gemfibrozil	na	2.1
Nonylphenol monoethoxylate	59.1	<2.8
Octylphenol diethoxylate	25.8	na
Triclocarban	na	3.3

Table 6. Lake Owasso

Chemical	2008	2013
17- β -estradiol	0.1	<4.2
2,6-di-tert-butyl-4-methylphenol	65.5	na
4-methylphenol	28.6	na
Androstenedione	qa	2.6
Androsterone	nd	128
BPA	25.6	3.6
Caffeine	129.4	16.5
Cholesterol	8982	na
Coprostanol	nd	na
Cotinine	na	15.6
DEET	90	52
Iopamidol	na	273
Mestranol	0.17	<50.4

Table 7. White Sand Lake

Chemical	2008	2013
17- β -estradiol	0.12	<4.0
2,6-di-tert-butyl-4-methylphenol	15.5	na
Amitriptyline	na	0.49
Androstenedione	1	<2.3
Benzoquinone	87.8	na
Cholesterol	7520	na
Coprostanol	552.5	na
Cotinine	na	6.9
DEET	33.4	69.3
Equilin	0.16	<9.0
Estrone	0.64	<4.0
Iopamidol	na	140
Metformin	na	4.1
Methylphenol	23	na
Naproxen	na	3.9
Testosterone	0.23	<2.6

Table 8. Red Sand Lake

Chemical	2008	2013
17- β -estradiol	0.03	<3.96
2,6-di-tert-butyl-4-methylphenol	17	na
Amitriptyline	na	0.5
Androstenedione	0.22	<2.25
Benzoquinone	38	na
BPA	<10.0	<2.0
Carbadox	na	5.5
Cholesterol	5688	na
Coprostanol	200	na
Cotinine	na	3.6
DEET	11.5	37.1
Diphenhydramine	14	<2.0
Estrone	0.63	<3.96
Iopamidol	na	339
Metformin	na	8.4
Methylphenol	15	na
Triclosan	na	5.8

Table 9. Sullivan Lake

Chemical	2008	2013
17- β -estradiol	0.08	<4.2
Androstenedione	0.72	<2.1
Benzoquinone	82.6	na
Cholesterol	7133	na
Coprostanol	50.3	na
Cotinine	na	6.1
DEET	30.5	16.5
Estrone	0.7	<4.2
Gyburide	na	2.9
Iopamidol	na	238
Methylphenol	19.5	na

Table 10. Elk Lake

Chemical	2008	2013
17- β -estradiol	0.41	<4.0
2,6-di-tert-butyl-4-methylphenol	18.4	na
Androstenedione	0.81	<2.1
Benzoquinone	76.5	na
BPA	<10.0	28.6
Caffeine	10.7	<14.9
Cholesterol	8279	na
Colchicine	na	8.5
Coprostanol	51	na
Cotinine	na	2.1
DEET	217.8	16.6
Estrone	1.1	<4.2
Metformin	na	3
Methylphenol	14.4	na

Table 11. Stewart Lake

Chemical	2008	2013
Androstenedione	0.33	<2.81
BPA	20	<2
Caffeine	13.8	<15.2
Cholesterol	1364	na
Clinafloxacin	na	66.6
Coprostanol	162	na
Cotinine	na	1.83
DEET	51.2	33.2
Estrone	1.45	<3.96

Iopamidol	na	302
Methylphenol	16.9	na
Octylphenol	10.2	na
Sertraline	na	12.7
Sulfachloropyridazine	na	3.83
Trimethoprim	na	3.63

Table 12. Shingobee

Chemical	2008	2013
Amphetamine	na	2.91
Androstenedione	0.54	<2
BPA	19.9	B2.96
Caffeine	12.8	<15
Cholesterol	1370	na
Coprostanol	418.5	na
DEET	579.4	10.6
Diphenhydramine	35.7	<0.6
Estrone	1.08	<4
Iopamidol	na	118
Metformin	na	18
Methylphenol	13.8	na
Nonylphenol	110.8	B1.57
Nonylphenol monoethoxylate	86.3	na
Nonylphenol diethoxylate	170.5	<2.67
Nonylphenol triethoxylate	123	na
Octylphenol	10.2	<0.55
Octylphenol diethoxylate	33.7	na
Octylphenol triethoxylate	13.8	na

Table 13. Lake Kabetogama

Chemical	2008	2013
Androstenedione	0.5	<2.57
Cholesterol	1976	na
Coprostanol	25.3	na
DEET	17.6	B 5.3
Estrone	0.02	<4.15
Iopamidol	na	510
Methylphenol	18.6	na
Sertraline	na	12.7

Table 14. Northern Light Lake

Chemical	2008	2013
17- β -estradiol	0.45	<4.09
2,6-di-tert-butyl-4-methylphenol	12	na
BPA	12.4	35.8
Caffeine	19.3	<16
Cholesterol	931	na
Ciprofloxacin	na	36.8
Clinafloxacin	na	184
Coprostanol	14.8	na
DEET	50.7	13.8
Diltiazem	na	1.19
Erythromycin-H2O	na	3.33
Methylphenol	41.4	na
Nonylphenol	213.8	B2.7
Nonylphenol monoethoxylate	106	<2.63
Nonylphenol diethoxylate	65.8	<3.45
Octylphenol diethoxylate	42.7	na
Trimethoprim	na	2.87

Table 15. Lake contaminants sorted by Anatomical Therapeutic Classification

Pharmaceutical detected	ATC designation	ATC category description (abbreviated)
Metformin	A10BA02	Alimentary Tract and Metabolism
Diltiazem	C08DB01	Cardiovascular System
Gemfibrozil	C10AB04	Cardiovascular System
Thiabendazole	D01AC06	Dermatologicals
Erythromycin-H2O	D10AF02	Dermatologicals
Naproxen	G02CC02	Genito Urinary System and Sex Hormones
Estrone	G03CA07	Genito Urinary System and Sex Hormones
Sulfachloropyridazine	J01E	Antiinfectives for Systemic Use
Trimethoprim	J01EA01	Antiinfectives for Systemic Use
Erythromycin-H2O	J01FA01	Antiinfectives for Systemic Use
Ciprofloxacin	J01MA02	Antiinfectives for Systemic Use
Naproxen	M01AE02	Musculo-Skeletal System
Naproxen	M02AA12	Musculo-Skeletal System
Colchicine	M04AC01	Musculo-Skeletal System
Amitriptyline	N06AA09	Tricyclic Antidepressant
Sertraline	N06AB06	Nervous System
Caffeine	N06BC01	Nervous System
Glyburide	No Data	

Norverapamil	No Data	
Cotinine	No Data	
Thiabendazole	P02CA02	Antiparasitic Products
Erythromycin-H2O	S01AA17	Sensory Organs, Ophthalmologicals
Clinafloxacin	S01AE	Sensory Organs, Ophthalmologicals
Iopamidol	V08AB04	Various, Contrast Media, X-Ray Contrast Media

Table 16. Contaminants detected in proximity of wastewater treatment plants sorted by Anatomical Therapeutic Classification

Pharmaceutical detected	ATC designation	Category description
Cimetidine	A02BA01	Alimentary Tract and Metabolism
Ranitidine	A02BA02	Alimentary Tract and Metabolism
Metformin	A10BA02	Alimentary Tract and Metabolism
Furosemide	C03CA01	Cardiovascular System
Triamterene	C03DB02	Cardiovascular System
Propranolol	C07AA05	Cardiovascular System
Metoprolol	C07AB02	Cardiovascular System
Atenolol	C07AB03	Cardiovascular System
Verapamil	C08DA01	Cardiovascular System
Diltiazem	C08DB01	Cardiovascular System
Valsartan	C09CA03	Cardiovascular System
Gemfibrozil	C10AB04	Cardiovascular System
Thiabendazole	D01AC06	Dermatologicals
Virginiamycin M1	D06AX10	Dermatologicals
Methylprednisolone	D07AA01	Dermatologicals
Erythromycin-H2O	D10AF02	Dermatologicals
Naproxen	G02CC02	Genito Urinary System And Sex Hormones
Trimethoprim	J01EA01	Antiinfectives for Systemic Use
Tylosin	J01EA01	Antiinfectives for Systemic Use
Sulfamethazine	J01EB03	Antiinfectives for Systemic Use
Sulfamethoxazole	J01EC01	Antiinfectives for Systemic Use
Sulfadimethoxine	J01ED01	Antiinfectives for Systemic Use
Erythromycin-H2O	J01FA01	Antiinfectives for Systemic Use
Clarithromycin	J01FA09	Antiinfectives for Systemic Use
Ofloxacin	J01MA01	Antiinfectives for Systemic Use
Moxifloxacin	J01MA14	Antiinfectives for Systemic Use
Amphetamine	J05AF01	Antiinfectives for Systemic Use
Naproxen	M01AE02	Musculo-Skeletal System
Cocaine	N01BC01	Nervous System
Oxycodone	N02AA05	Nervous System
Carbamazepine	N03AF01	Nervous System

Alprazolam	N05BA12	Nervous System
Meprobamate	N05BC01	Nervous System
Fluoxetine	N06AB03	Nervous System
Citalopram	N06AB04	Nervous System
Sertraline	N06AB06	Nervous System
Venlafaxine	N06AX16	Nervous System
Caffeine	N06BC01	Nervous System
Thiabendazole	P02CA02	Antiparasitic Products
Cocaine	R02AD03	Respiratory System
Albuterol	R03AC02	Respiratory System
Theophylline	R03DA04	Respiratory System
Hydrocodone	R05DA03	Respiratory System
Codeine	R05DA04	Respiratory System
Diphenhydramine	R06AA02	Respiratory System
Ofloxacin	S01AE01	Sensory Organs, Ophthalmologicals
Cocaine	S01HA01	Sensory Organs, Ophthalmologicals
Ofloxacin	S02AA16	Sensory Organs, Otologicals
Cocaine	S02DA02	Sensory Organs, Otologicals
Ciprofloxacin	S03AA07	Sensory Organs, Ophthalmological And Otological
Diatrizoic acid	V08AA01	Various, Contrast Media, X-Ray Contrast Media
Iopamidol	V08AB04	Various, Contrast Media, X-Ray Contrast Media
Pseudoephedrine/Ephedrine	No Data	
Dehydronifedipine	No Data	
Amitriptyline	No Data	
Hydrochlorothiazide	No Data	
Desmethyldiltiazem	No Data	

Figure 1. Sampling locations



Figure 2. Frequency of contaminant detection in lakes in 2013

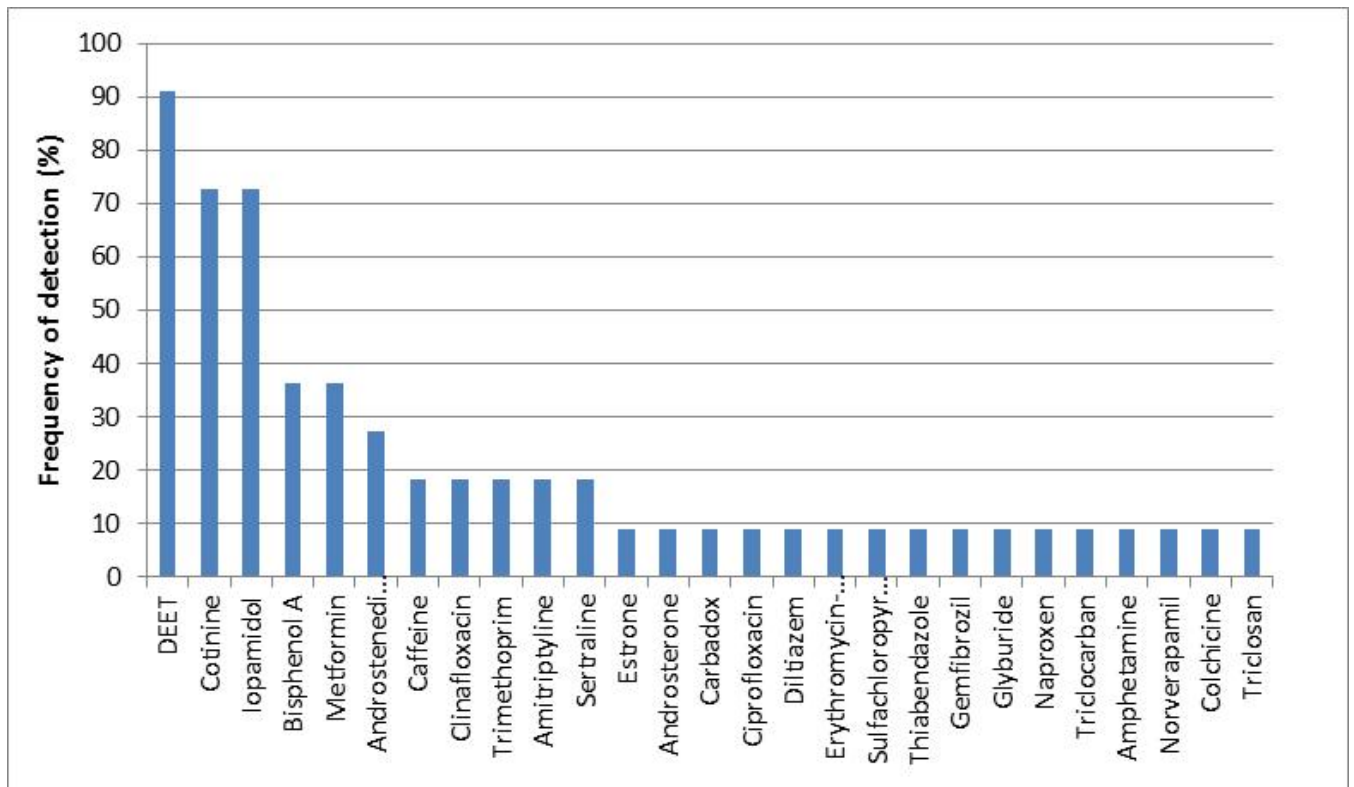


Figure 3. Fraction of locations downstream of wastewater treatment plants where chemicals were detected in 2013

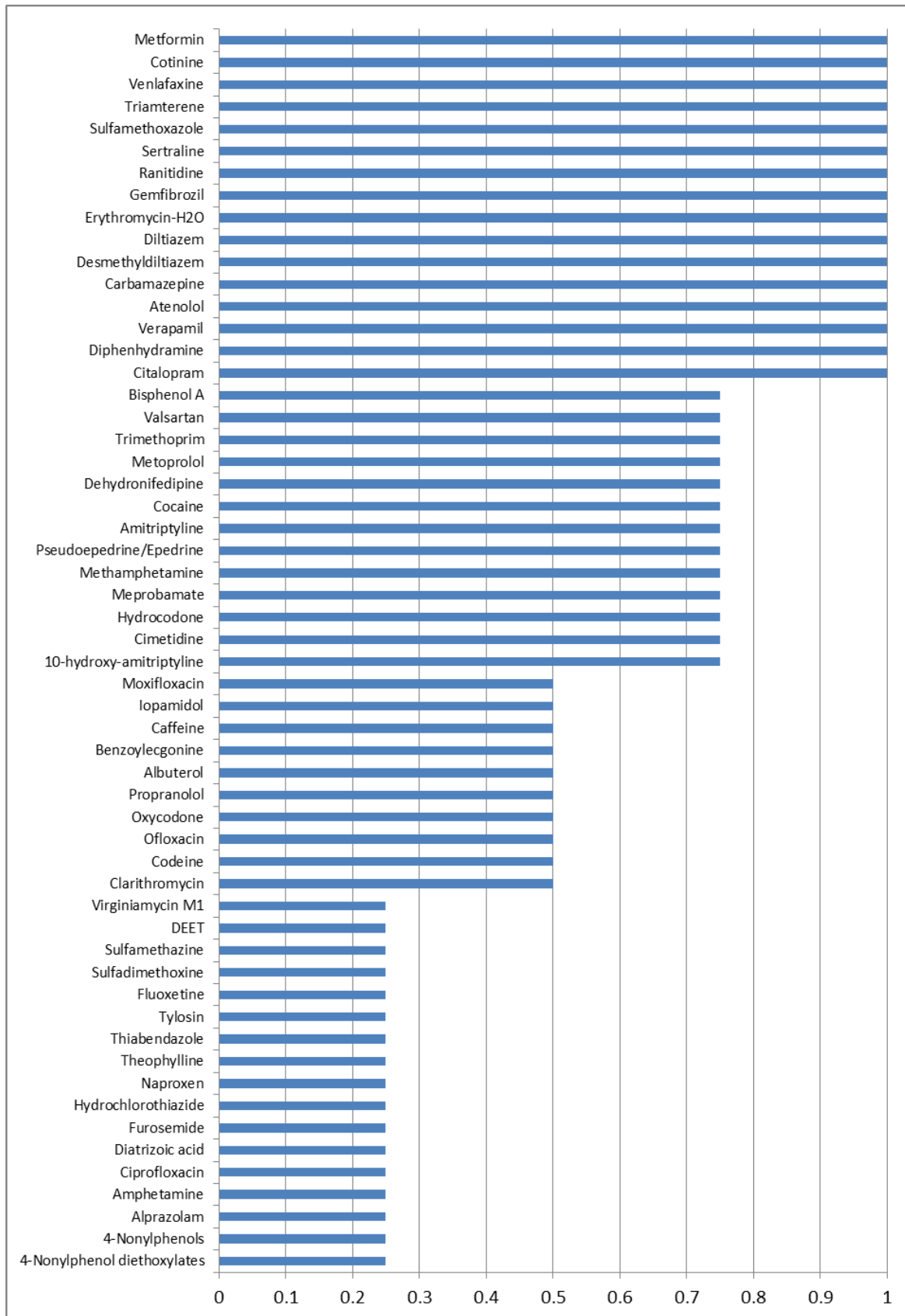


Figure 4. Fraction of locations upstream of wastewater treatment plants where chemicals were detected in 2013

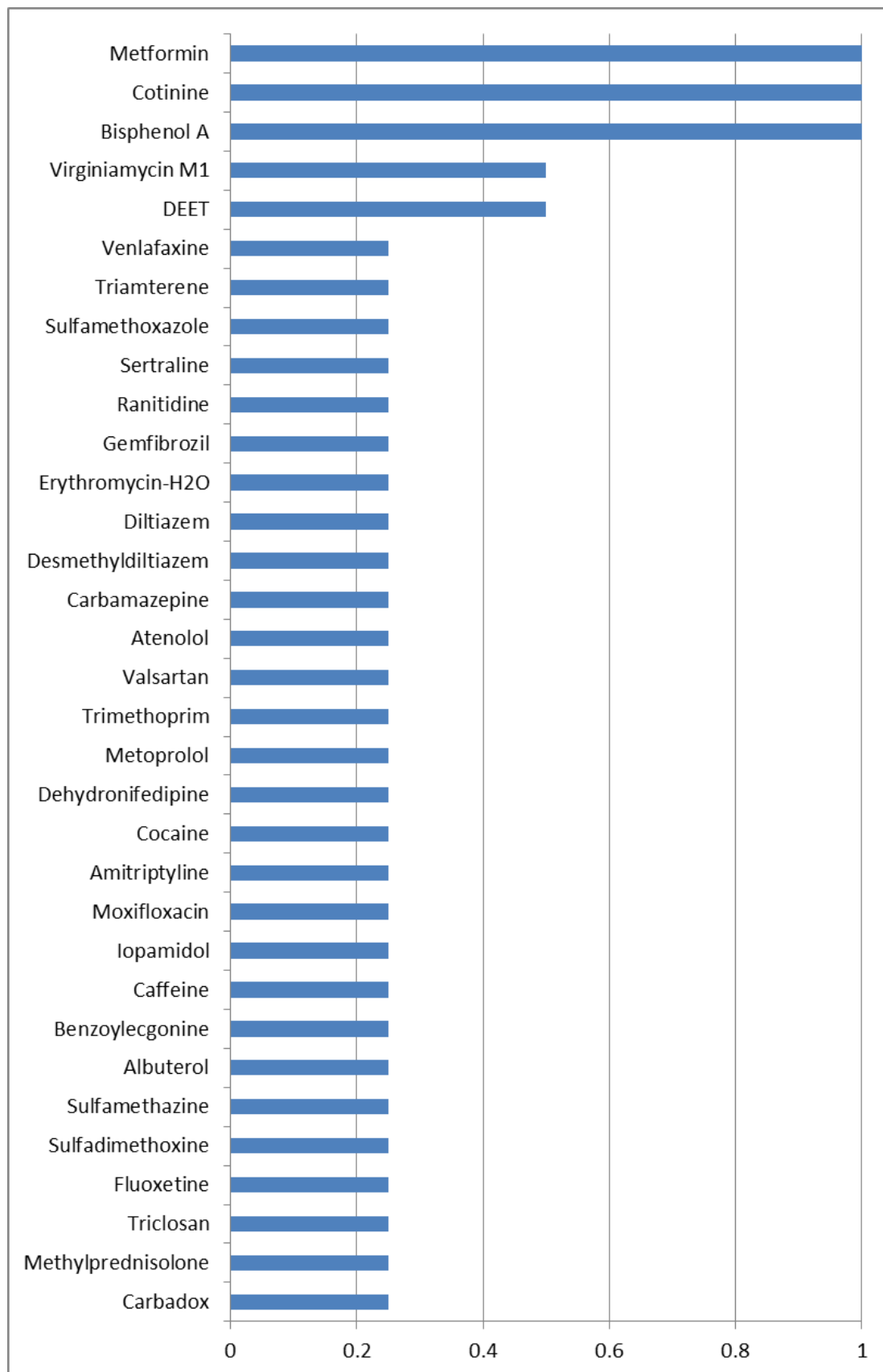
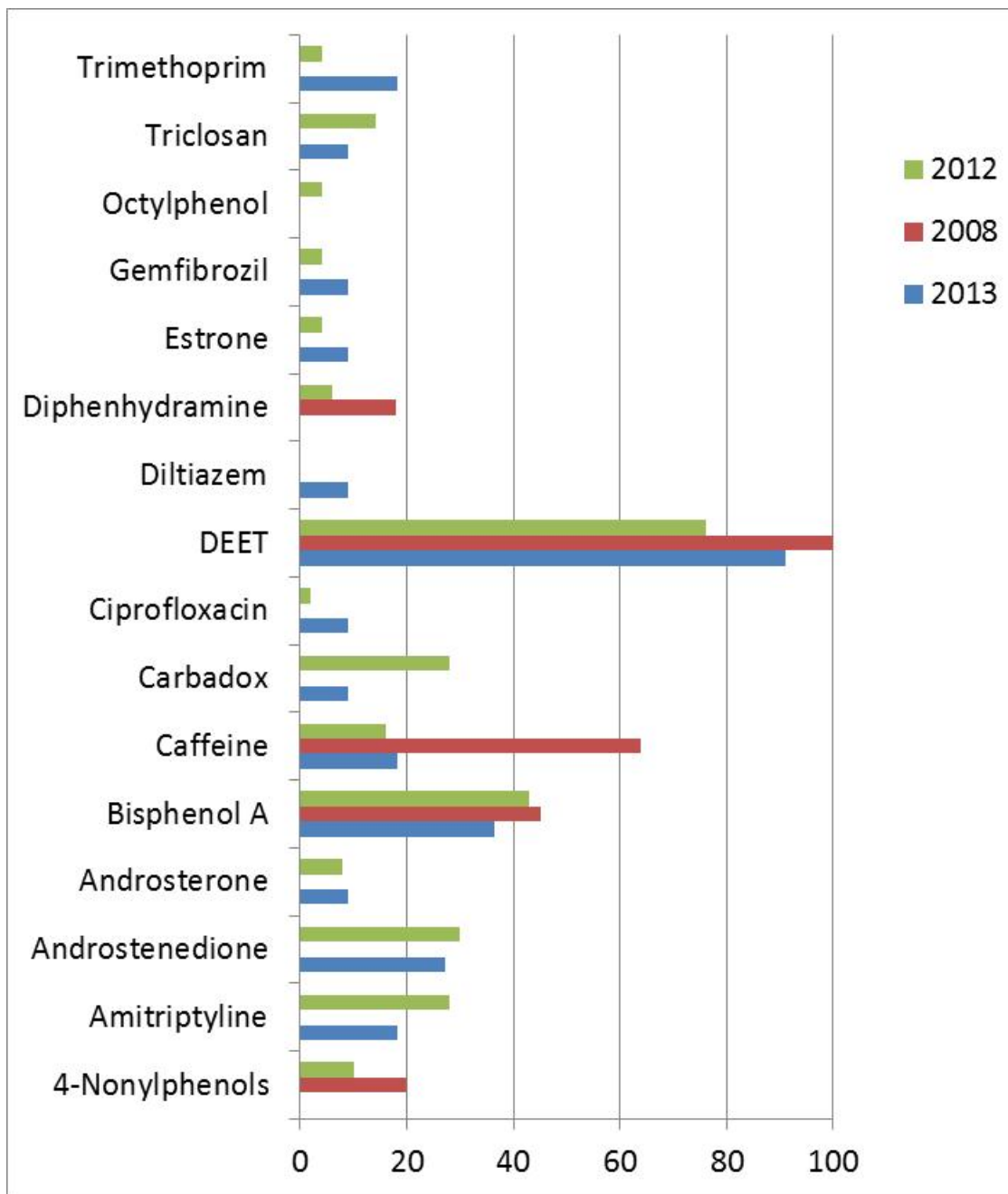


Figure 5. Percent of lake locations where chemicals were detected for 2008, 2012, and 2013



Appendix A

Lake and wastewater treatment plant descriptions and location information

Lakes

Lake name	County	MPCA/MDNR site ID	Trophic state	Lake type	Littoral area MDNR 15 feet standard (acres)	Lake surface area (acres)	Percent littoral
Budd	Martin	46-0030	Eutrophic	Urban, sewerred	168.56	218.38	77.19%
Cedar	Hennepin	27-0039	Eutrophic	Urban, sewerred	60.06	163.79	36.67%
Elk	Clearwater	15-0010	Mesotrophic	Reference	77.74	305.36	25.46%
Kabetogama	St. Louis	69-0845	Oligotrophic	Septic input	7168.96	24032.72	29.83%
Northern Light	Cook	16-0089	Oligotrophic	Reference	433.00	453.32	95.52%
Owasso	Ramsey	62-0056	Eutrophic	Urban, sewerred	284.18	374.96	75.79%
Red Sand	Crow Wing	18-0386	Mesotrophic	Septic input	502.00	502.00	100.00%
Shingobee	Hubbard	29-0043	Oligotrophic	Septic input	43.00	171.93	25.01%
Stewart	Crow Wing	38-0744	Oligotrophic	Septic input	237.11	237.72	99.74%
Sullivan	Wright	86-0119	Mesotrophic	Septic input	39.43	70.58	55.87%
White Sand	Crow Wing	18-0379	Mesotrophic	Septic input	282.54	413.22	68.38%

Percentage of land cover types within 1 km of lake shoreline

Lake name	Mean depth (feet)	Open water	Marsh	High-intensity urban	Low-intensity urban	Deciduous forest
Budd	11.1	7.82%	0.52%	22.85%	21.98%	3.63%
Cedar	19.62	7.38%	0.04%	44.34%	42.73%	2.67%
Elk	34.25	2.34%	2.12%	0.00%	0.00%	84.51%
Kabetogama	24.75	1.99%	2.45%	0.00%	0.00%	66.85%
Northern Light	n/a	0.21%	0.63%	0.00%	0.00%	87.66%
Owasso	10.9	4.02%	0.54%	2.23%	90.88%	1.14%
Red Sand	n/a	18.28%	7.89%	0.00%	0.00%	31.99%
Shingobee	n/a	0.37%	0.24%	0.00%	0.00%	92.84%
Stewart	6.42	0.17%	0.00%	0.00%	0.00%	97.26%
Sullivan	14.94	0.04%	0.64%	0.00%	0.00%	0.11%
White Sand	9.91	13.84%	4.49%	4.48%	1.68%	20.23%

Percentage of land cover types within 1 km of lake shoreline

Lake name	Conifer forest	Mixed forest	Shrubland	Grassland	Cropland	Barren
Budd	0.00%	0.00%	0.00%	2.28%	40.93%	0.00%
Cedar	1.38%	0.00%	0.22%	0.58%	0.66%	0.00%
Elk	10.21%	0.00%	0.83%	0.00%	0.00%	0.00%
Kabetogama	18.37%	0.00%	7.46%	1.20%	0.08%	1.60%
Northern Light	8.21%	0.18%	2.79%	0.33%	0.00%	0.00%
Owasso	0.15%	0.00%	0.29%	0.47%	0.28%	0.00%
Red Sand	24.44%	0.00%	2.52%	11.80%	3.09%	0.00%
Shingobee	1.87%	0.00%	0.30%	1.53%	2.86%	0.00%
Stewart	0.70%	0.00%	1.86%	0.00%	0.00%	0.00%
Sullivan	0.11%	0.00%	1.13%	2.10%	95.87%	0.00%
White Sand	43.32%	0.00%	1.03%	7.28%	3.66%	0.00%

Wastewater treatment plants

	Sauk Centre WWTP Sauk Centre, MN	Marshall WWTP Marshall, MN	Metro Plant (WWTP) St. Paul, MN	Hinckley WWTP Hinckley, MN
Design flow (Mgal/d)	0.88	4.5	251	0.5
Design flow (cms)	0.0385	0.1971	10.99	0.0219
Type	Continuous	Continuous	Continuous	Continuous
Treatment process	Activated sludge, contact stabilization, conventional, step feed	Trickling filter, Activated sludge, contact stabilization, conventional, step feed, sand filter	Activated sludge, contact stabilization, conventional, step feed	Activated sludge, extended aeration
No. trickling filters	0	2	0	0
disinfection	Chl/DEChl	UV	Chl/DEChl	UV
Population served	4111	13000	1,800,000	1438
Percent domestic	100	40	na	100
Percent industrial	0	60	na	0

Sampling locations

Lakes

Lake	Lat	Long
Budd	43°38'20.97"N	94°28'1.57"W
Cedar	44°57'44.41"N	93°19'16.39"W
Elk	47°11'28.04"N	95°13'7.61"W
Kabetogama	48°28'59.42"N	93° 1'35.33"W
Northern Light	47°54'19.67"N	90°14'21.86"W
Owasso	45° 1'37.33"N	93° 7'58.43"W
Red Sand	46°22'40.08"N	94°17'15.08"W
Shingobee	47° 0'5.92"N	94°41'26.38"W
Stewart	47°11'16.48"N	91°45'16.03"W
Sullivan	45°13'19.13"N	93°56'30.47"W
White Sand	46°21'3.33"N	94°17'18.17"W

Wastewater treatment plants

Samples were collected upstream and downstream of the WWTP outfalls.

WWTP	Lat	Long
Sauk Centre upstream	45°43'8.18"N	94°56'23.93"W
Sauk Centre downstream	45°42'51.04"N	94°56'17.75"W
Marshall upstream	44°28'45.50"N	95°46'33.79"W
Marshall downstream	44°29'12.10"N	95°45'57.47"W
Metro Plant upstream	44°55'11.04"N	93° 3'4.46"W
Metro Plant downstream	44°54'5.90"N	93° 2'17.60"W
Hinckley upstream	46° 1'6.86"N	92°54'33.46"W
Hinckley downstream	46° 0'48.40"N	92°53'23.57"W

Appendix B

Sample collection

Samples were collected in one liter amber glass or high-density polyethylene (HDPE) bottles, depending on the specific analysis for which the sample was intended. All sample bottles were provided by AXYS Analytical Services in Sidney, British Columbia, Canada. Field staff did not apply fragrances, insect repellent (DEET), or sunscreen prior to sampling and wore disposable powder-free, nitrile gloves while sampling. Sample bottles were transported to the site in re-sealable plastic bags and coolers.

For lakes, mid-lake grab samples were collected from the bow of the watercraft, holding the collection bottle at arm's length ahead of the boat which was maneuvered slowly in an upwind direction in an effort to prevent any possible airborne or waterborne contamination from either the watercraft or the field staff. Sample bottles were removed from the re-sealable plastic bags and uncapped for sample collection only with gloved hands and only when these sampling conditions were achieved. Samples were not collected during rainfall.

For wastewater treatment plants, grab samples were collected from locations upstream and downstream of the effluent outfall. Except for the Twin Cities Metro WWTP, samples were collected midstream, reached by wading into the stream flow, and from an area of the stream judged to be the highest flow. For samples collected from the Mississippi River above and below the Twin Cities Metro WWTP, the sampling procedure was the same as for lakes. For the other three WWTP locations (Sauk, Marshall, and Hinckley), samples were collected at arm's length from upstream relative to the field staff. Sample bottles were removed from the plastic re-sealable bags and uncapped for sample collection only with gloved hands and only when these sampling conditions were achieved. Samples were not collected during rainfall. Sample collection was not flow-weighted.

For all sample collection events, sample bottles were rinsed with the surface water three times by filling and emptying the bottle completely. Bottles were immersed below the surface so as not to allow exposed skin above the gloved hand to come into contact with the surface water. The final sample was collected without headspace in the bottle. Once filled, bottles were re-capped, re-sealed in the plastic bags, and chilled in the accompanying coolers on ice.

Field blanks were collected by transporting duplicate sample bottles to a location where samples were collected. Field blank water, also supplied by the laboratory in identical glass or HDPE bottles, was transported to the site. Field blank water bottles were transported in identical re-sealable plastic bags and in coolers to replicate the procedure used for actual samples. Using gloved hands and facing upwind, the field blank water was poured from the lab-supplied bottles into empty sample bottles without rinsing. The field blank samples were then re-capped, re-sealed in plastic bags, and treated identically to the surface water samples.

After returning from the field, samples were refrigerated at four degrees Celsius. All samples were kept in the original re-sealable plastic bags and were shipped overnight to AXYS Analytical Services for chemical analysis. The maximum holding time for samples, from the date of collection to their extraction in the laboratory, was seven days.

Appendix C

Laboratory analytical methods

Five separate analyses were performed to target the 125 compounds (see [Table 1](#)) analyzed in this study as described below.

PPCPs

The pharmaceuticals and personal care products (PPCPs) selected for analysis were based on the U.S. Environmental Protection Agency (EPA) Method 1694 with additional compounds incorporated into List 3 and an additional run for List 5 compounds. This expanded EPA 1694 analyte list represents those PPCP compounds identified by the EPA and other AXYS clients (AXYS Analytical Services, Sidney, British Columbia, Canada) as priorities for assessment based on annual consumption, expected toxicity, and persistence. For analysis of List 1, 2, 3, and 5 compounds ([Table C1](#)), a 1.0 L sample was filtered (1.6 µm), adjusted to pH 2 by addition of HCl, spiked with a suite of isotopically labeled internal standards and extracted by solid phase extraction using an Oasis HLB cartridge. The extract was analyzed by liquid chromatography-tandem mass spectrometry (LC/MS/MS) operated in the ESI positive mode for List 1, 2, 5 and 6 compounds and analyzed in the ESI negative mode for List 3 compounds. Separate analysis runs and conditions were used for each of the lists of target analytes.

Hormones

A 1.0 L sample was filtered (1.6 µm), adjusted to pH 2 by addition of HCl, spiked with a suite of isotopically labeled internal standards, and extracted by solid phase extraction using an Oasis HLB cartridge. The extract was analyzed by LC/MS/MS operated in the ESI positive mode for the ESI+ hormones and by LC/MS/MS operated in the ESI negative mode for the ESI- hormones ([Table C1](#)).

Alkylphenols

An unfiltered 1.0 L sample was spiked with isotopically labeled internal standards, adjusted to pH 11-12 by the addition of potassium carbonate solution, and acetylated with acetic anhydride. The extract was then acidified to pH 6 with HCl and extracted with hexane. Extracts were cleaned up by silica column chromatography and analyzed by gas chromatography-mass spectrometry (GC/MS) operated in the multiple ion detection mode.

Bisphenol A

A 0.5 L sample was filtered (1.6 µm), adjusted to pH 2 by addition of HCl, and spiked with deuterated bisphenol A internal standard and extracted by solid phase extraction using an Oasis HLB cartridge. The extract was analyzed by LC/MS/MS operated in the ESI negative mode.

Triclosan

A 0.5 L sample was filtered (1.6 µm), adjusted to pH 2 by addition of HCl, spiked with [¹³C]-labeled triclosan, and extracted by solid phase extraction using an Oasis HLB cartridge. The extract was analyzed by LC/MS/MS operated in the ESI negative mode.

Quality control and assurance

All analytes were quantified either by isotope dilution internal standard quantification or, when an isotopically labeled analog of the analyte was unavailable, by internal standard methods using a related labeled compound. This produces accurate results that are recovery-corrected for losses during the analysis procedure and compensated for LC/MS/MS suppression or enhancement due to sample matrix. For each batch of samples analyzed, a lab blank was included to demonstrate that detected analytes

were not due to lab background or other external contamination. Results for lab blanks were compliant with AXYS's statistically determined blank control limits (mean plus three standard deviations of about 30 blanks) and any detects were used to censor field sample results. Each analysis batch also included a "known" or quality control sample to demonstrate the accuracy of the method for each analyte. Recoveries of all added labeled standards were monitored to ensure that analyses were in control and meeting regular method specifications.

Table C1.

PPCP List 1	PPCP List 2	PPCP List 5
Acetaminophen	Anhydrochlortetracycline	Alprazolam
Azithromycin	Anhydrotetracycline	Amitriptyline
Caffeine	Chlortetracycline	Amlodipine
Carbadox	Demeclocycline	Benzoylcegonine
Carbamazepine	Doxycycline	Benztropine
Cefotaxime	4-Epianhydrochlortetracycline	Betamethasone
Ciprofloxacin	4-Epianhydrotetracycline	Cocaine
Clarithromycin	4-Epichlortetracycline	DEET
Clinafloxacin	4-Epioxytetracycline	Desmethyldiltiazem
Cloxacillin	4-Epitetracycline	Diazepam
Dehydronifedipine	Isochlortetracycline	Fluocinonide
Diphenhydramine	Minocycline	Fluticasone propionate
Diltiazem	Oxytetracycline	Hydrocortisone
Digoxin	Tetracycline	10-hydroxy-amitriptyline
Digoxigenin		Meprobamate
Enrofloxacin	PPCP List 3	Methylprednisolone
Erythromycin-H2O	Bisphenol A	Metoprolol
Flumequine	Furosemide	Norfluoxetine
Fluoxetine	Gemfibrozil	Norverapamil
Lincomycin	Glipizide	Paroxetine
Lomefloxacin	Glyburide	Prednisolone
Miconazole	Hydrochlorothiazide	Prednisone
Norfloxacin	2-Hydroxy-ibuprofen	Promethazine
Norgestimate	Ibuprofen	Propoxyphene
Ofloxacin	Naproxen	Propranolol
Ormetoprim	Triclocarban	Sertraline
Oxacillin	Triclosan	Simvastatin

(PPCP List 1, cont.)	(PPCP List 3, cont.)	(PPCP List 5, cont.)
Oxolinic Acid	Warfarin	Theophylline
Penicillin G		Trenbolone
Penicillin V	Hormones ESI +	Trenbolone acetate
Roxithromycin	Allyl Trenbolone	Valsartan
Sarafloxacin	Androstenedione	Verapamil
Sulfachloropyridazine	Androsterone	
Sulfadiazine	Desogestrel	Alkylphenols
Sulfadimethoxine	Estriol	4-Nonylphenol monoethoxylates
Sulfamerazine	Mestranol	4-Nonylphenol diethoxylates
Sulfamethazine	Norethindrone	4-Nonylphenols
Sulfamethizole	Norgestrel	Octylphenol
Sulfamethoxazole	Progesterone	
Sulfanilamide	Testosterone	Bisphenol A
Sulfathiazole		
Thiabendazole	Hormones ESI-	Triclosan
Trimethoprim	17 alpha-Dihydroequilin	
Tylosin	Equilenin	
Virginiamycin	Equilin	
1,7-Dimethylxanthine	17 beta-Estradiol	
	17 alpha-Estradiol	
	Estrone	
	17 alpha-Ethinyl-Estradiol	

Appendix D

Analytical

Data flags and definitions

Flag	Definition
B	Analyte found in the sample and the associated laboratory blank
U	Analyte not detected at reporting limit (RL)
K	Peak detected but did not meet quantification criteria, the result reported represents the estimated maximum possible concentration
N	Authentic recovery is not within the method/contract control limits
TIC	Compound identity and concentration are estimated
V	Surrogate recovery is not within method/contract control limits
H	Analyte concentration is estimated
NQ	Data is not quantifiable
T	The result was recalculated against alternate labeled compound(s) or internal standard
MAX	Analyte concentration is an estimated maximum value
D	Dilution data

All data are reported in ng/L, or parts per trillion

Alkylphenols

Location	4-Nonylphenols	4-Nonylphenol monoethoxylates	4-Nonylphenol diethoxylates	Octylphenol
Lake				
Budd Lake	B 7.06	U 2.84	U 2.89	U 0.905
Cedar Lake	B 5.29	U 3.26	U 4.04	U 0.815
Elk Lake	U 1.99	U 2.16	U 1.86	U 0.976
Lake Kabetogama	B 19.9	U 1.95	U 2.79	U 0.98
Lake Owasso	B 5.56	U 2.95	U 4.44	U 0.738
Northern Light Lake	B 2.7	U 2.63	U 3.45	U 1.01
Red Sand Lake	B 1.45	U 1.42	U 2.73	U 0.534
Shingobee Lake	B 1.57	U 3.69	U 2.67	U 0.556
Stewart Lake	B 5.65	U 2.81	U 2.31	U 0.805
Sullivan Lake	B 3.28	U 2.68	U 2.89	U 1.01
White Sand Lake	B 1.71	U 1.26	U 2.2	U 0.652
WWTP				
Hinckley-down	B 14.7	U 4.63	U 4.03	U 1.22
Hinckley-up	B 7.42	U 9.91	U 14.6	U 1.68
Marshall-down	B 29.1	U 2.97	13	U 0.97
Marshall-up	B 20.1	U 5.29	U 7.25	U 0.88
Metro Plant-down	30.1	U 3.99	7.01	U 1.01
Metro Plant-up	B 6.03	U 3.17	U 2.45	U 0.763
Sauk-down	B 8.29	U 7.41	U 9.92	U 1.2
Sauk-up	B 5.13	U 8.25	U 11.5	U 1.69
Field Blank 1	B 8.66	U 2.79	U 2.53	U 0.654
Field Blank 2	B 24.1	U 2.86	U 2.85	U 1.13

Bisphenol A

Location	Bisphenol A
Lake	
Budd Lake	U 2.02
Cedar Lake	6.4
Elk Lake	28.6
Lake Kabetogama	U 2.03
Lake Owasso	3.6
Northern Light Lake	35.8
Red Sand Lake	U 2.07
Shingobee Lake	B 2.96
Stewart Lake	U 2
Sullivan Lake	U 1.98
White Sand Lake	U 2.07
WWTP	
Hinckley-down	5.5
Hinckley-up	3.29
Marshall-down	6.98
Marshall-up	4.1
Metro Plant-down	B 3.81
Metro Plant-up	237
Sauk-down	5.23
Sauk-up	3.47
Field Blank 1	B 2.99
Field Blank 2	U 2.36

Triclosan

Location	Triclosan
Lake	
Budd Lake	U 4.91
Cedar Lake	U 4.89
Elk Lake	U 5.78
Lake Kabetogama	U 5.31
Lake Owasso	U 5.03
Northern Light Lake	U 5.01
Red Sand Lake	5.75
Shingobee Lake	U 5.13
Stewart Lake	U 5.03
Sullivan Lake	U 4.97
White Sand Lake	U 4.99
WWTP	
Hinckley-down	U 4.31
Hinckley-up	9.35
Marshall-down	U 4.99
Marshall-up	U 4.91
Metro Plant-down	U 5.02
Metro Plant-up	U 4.82
Sauk-down	U 4.76
Sauk-up	U 4.27
Field Blank 1	U 5.13
Field Blank 2	U 5

Hormones-negative

Location	17 alpha-Dihydroequilin	Equilenin	Equilin	17 beta-Estradiol	17 alpha-Estradiol	Estrone	17 alpha-Ethinylestradiol
Lake							
Budd Lake	U 4.29	U 0.858	U 8.58	U 4.29	U 4.29	U 4.29	U 5.36
Cedar Lake	U 4.21	U 0.841	U 8.41	U 4.21	U 4.21	24.6	U 5.26
Elk Lake	U 5.71	U 0.808	U 8.08	U 4.04	U 4.04	U 4.04	U 5.05
Lake Kabetogama	U 4.15	U 0.869	U 8.29	U 4.15	U 4.15	U 4.15	U 5.18
Lake Owasso	U 4.22	U 0.844	U 8.44	U 4.22	U 4.22	U 4.22	U 5.27
Northern Light Lake	U 4.09	U 0.817	U 8.17	U 4.09	U 4.09	U 4.09	U 5.11
Red Sand Lake	U 3.96	U 0.792	U 7.92	U 3.96	U 3.96	U 3.96	U 4.95
Shingobee Lake	U 5.88	U 1.54	U 7.98	U 3.99	U 3.99	U 3.99	U 4.99
Stewart Lake	U 3.96	U 0.791	U 7.91	U 3.96	U 3.96	U 3.96	U 4.95
Sullivan Lake	U 4.23	U 0.845	U 8.45	U 4.23	U 4.23	U 4.23	U 5.28
White Sand Lake	U 6.5	U 0.803	U 9.04	U 4.01	U 4.01	U 4.01	U 5.02
WWTP							
Hinckley-down	U 4.04	U 1.93	U 8.09	U 4.04	U 4.04	U 4.04	U 5.74
Hinckley-up	U 4.62	U 1.3	U 8.09	U 4.05	U 4.05	U 4.05	U 5.06
Marshall-down	U 3.94	U 0.788	U 7.88	U 3.94	U 3.94	U 3.94	U 4.93
Marshall-up	U 3.87	U 0.774	U 7.74	U 3.87	U 3.87	U 3.87	U 4.84
Metro Plant-down	U 3.99	U 0.798	U 7.98	U 3.99	U 3.99	U 3.99	U 4.98
Metro Plant-up	U 3.99	U 0.798	U 7.98	U 3.99	U 3.99	U 3.99	U 4.99
Sauk-down	U 4.03	U 0.805	U 8.05	U 4.03	U 4.03	U 4.03	U 9.13
Sauk-up	U 4.08	U 0.922	U 8.15	U 4.08	U 4.08	U 4.08	U 5.09
Field Blank 1	U 4.02	U 0.804	U 8.04	U 4.02	U 4.02	U 4.02	U 5.02
Field Blank 2	U 4.21	U 0.842	U 8.42	U 4.21	U 4.21	U 4.21	U 5.26

Hormones-positive

Location	Allyl Trenbolone	Androstenedione	Androsterone	Desogestrel	Estriol	Mestranol	Norethindrone	Norgestrel	Progesterone	Testosterone
Lake										
Budd Lake	U 1.04	5	U 27.1	U 208	U 71	U 70.8	U 4.29	U 4.29	U 1.22	U 1.62
Cedar Lake	U 1.21	3.96	U 40.5	U 1500	U 105	K 226	U 4.21	U 4.67	U 1.66	U 1.8
Elk Lake	U 0.96	U 2.06	U 56.6	U 303	U 167	K B 208	U 4.83	U 4.04	U 0.808	U 1.83
Lake Kabetogama	U 1.36	U 2.57	U 20.7	U 648	U 16.6	U 69.4	U 4.63	U 4.15	U 0.829	U 3.28
Lake Owasso	U 1.5	2.62	128	U 600	U 106	U 50.4	U 4.22	U 4.22	U 0.844	U 2.43
Northern Light Lake	U 1.42	U 2.22	U 107	U 306	U 251	K B 269	U 4.09	U 4.43	U 0.817	U 2.46
Red Sand Lake	U 1.2	U 2.25	U 19.8	U 297	U 31.7	K B 389	U 5.64	U 6.03	U 0.838	U 4.24
Shingobee Lake	U 0.798	U 2	U 40.5	U 332	U 139	K B 106	U 3.99	U 3.99	U 0.798	U 1.6
Stewart Lake	U 1.39	U 2.81	U 68.7	U 297	U 325	K B 220	U 4.24	U 6.59	U 0.791	U 3.08
Sullivan Lake	U 1.06	U 2.11	U 30.5	TIC 402	U 64.6	U 34	U 4.23	U 4.27	U 0.845	U 1.23
White Sand Lake	U 0.803	U 2.28	U 20.1	U 301	U 32.1	K B 191	U 5.02	U 4.62	U 1.07	U 2.59
WWTP										
Hinckley-down	U 0.929	U 2.02	U 91	U 448	U 236	K B 214	U 4.24	U 4.04	U 0.809	U 2.09
Hinckley-up	U 0.891	U 2.02	U 40.5	U 422	U 305	K B 219	U 5	U 4.05	U 0.809	U 0.809
Marshall-down	U 0.808	U 1.97	U 82.8	U 324	U 172	K B 129	U 3.94	U 3.94	U 1.22	U 2.03
Marshall-up	U 0.979	U 1.94	U 108	U 498	U 31	K B 114	U 3.87	U 4.43	U 0.965	U 2.23
Metro Plant- down	U 1.91	U 2.39	U 72.6	U 299	U 370	K B 294	U 3.99	U 4.78	U 0.798	U 1.88
Metro Plant-up	U 1.19	U 2.71	U 68.2	U 299	U 308	K B 83.6	U 4.35	U 5.1	U 0.798	U 2.51
Sauk-down	U 1.17	U 2.01	U 143	U 319	U 240	K B 282	U 4.48	U 4.03	U 0.805	U 1.87
Sauk-up	U 0.991	U 2.04	U 72.1	U 334	U 310	K B 299	U 4.41	U 4.08	U 0.815	U 1.52
Field Blank 1	U 0.985	U 2.01	U 60.1	U 301	U 32.2	K B 88.4	U 4.02	U 4.02	U 0.804	U 0.804
Field Blank 2	U 0.842	U 2.1	U 31.2	U 316	U 33.7	K B 49.3	U 4.21	U 4.21	U 0.842	U 0.869

Illicit drugs

Location	3,4-methylenedioxy-N-methylamphetamine (MDMA)	Amphetamine	Benzoyllecgonine	Cocaine	Methamphetamine	Morphine	Pseudo ephedrine/Ephedrine
Lake							
Budd Lake	U 0.39	U 1.10	U 0.50	U 0.47	U 0.75	U 9.10	U 0.67
Cedar Lake	U 0.41	U 1.8	U 0.50	U 0.47	U 0.75	U 9.8	U 0.69
Elk Lake	U 0.43	U 1.6	U 0.50	U 0.47	U 0.75	U 9.6	U 0.71
Lake Kabetogama	U 0.46	U 1.3	U 0.50	U 0.47	U 0.75	U 9.3	U 0.74
Northern Light Lake	U 0.44	U 1.5	U 0.50	U 0.47	U 0.75	U 9.5	U 0.72
Lake Owasso	U 0.42	U 1.7	U 0.50	U 0.47	U 0.75	U 9.7	U 0.70
Red Sand Lake	U 0.48	U 1.1	U 0.50	U 0.47	U 0.75	U 9.1	U 0.76
Shingobee Lake	U 0.47	U 1.2	U 0.50	U 0.47	U 0.75	U 9.2	U 0.75
Stewart Lake	U 0.45	U 1.4	U 0.50	U 0.47	U 0.75	U 9.4	U 0.73
Sullivan Lake	U 0.40	U 1.9	U 0.50	U 0.47	U 0.75	U 9.9	U 0.68
White Sand Lake	U 0.49	U 1.0	U 0.50	U 0.47	U 0.75	U 9.0	U 0.77
WWTP							
Hinckley-down	U 0.51	U 1.2	U 0.50	U 0.47	U 0.75	U 9.2	U 0.79
Hinckley-up	U 0.50	U 1.1	U 0.50	U 0.47	U 0.75	U 9.1	U 0.78
Marshall-down	U 0.55	U 1.6	U 0.50	U 0.47	3	U 9.6	2.3
Marshall-up	U 0.54	U 1.5	U 0.50	U 0.47	U 0.75	U 9.5	U 0.82
Metro -down	U 0.57	U 1.8	2	U 0.47	2	U 9.8	1.5
Metro-up	U 0.56	U 1.7	U 0.50	U 0.47	U 0.75	U 9.7	U 0.84
Sauk-down	U 0.53	U 1.4	U 0.50	U 0.47	0.75	U 9.4	1.1
Sauk-up	U 0.52	U 1.3	U 0.50	U 0.47	U 0.75	U 9.3	U 0.80
Field Blank 1	U 0.58	U 1.9	U 0.50	U 0.47	U 0.75	U 9.9	U 0.86
Field Blank 2	U 0.59	U 1.10	U 0.50	U 0.47	U 0.75	U 9.10	U 0.87

Pharmaceuticals list 1

Location	Acetaminophen	Azithromycin	Caffeine	Carbadox	Carbamazepine	Cefotaxime	Ciprofloxacin	Clarithromycin	Clinafloxacin
Lake									
Lake Kabetogama	U 15.1	U 1.57	U 15.1	U 1.51	U 1.51	U 55	U 37.9	U 1.51	U 52.3
Budd Lake	U 16.2	U 2.12	U 16.2	U 23.8	U 1.62	U 21.6	U 31.7	U 1.62	U 124
Sullivan Lake	U 16	U 1.92	U 16	U 13.3	U 1.6	U 21.3	U 33.2	U 1.6	U 49.3
Cedar Lake	U 15.3	U 2.72	69.7	U 26.2	U 1.53	U 20.4	U 23.4	U 1.53	U 78.9
Lake Owasso	U 15.2	U 2.76	16.5	U 29	U 1.52	U 20.3	U 35.7	U 1.52	U 93.2
White Sand Lake	U 15	U 1.5	U 15	U 1.5	U 1.5	U 11.5	U 29.9	U 1.5	U 51.3
Red Sand Lake	U 15.2	U 1.52	U 15.2	5.45	U 1.52	U 21.1	U 38.7	U 1.52	U 67
Elk Lake	U 14.9	U 1.49	U 14.9	U 1.49	U 1.49	U 31.4	U 29.6	U 1.49	U 60
Shingobee Lake	U 15	U 1.5	U 15	U 1.5	U 1.5	U 24.4	U 20	U 1.5	U 52
Stewart Lake	U 15.2	U 1.64	U 15.2	U 6.39	U 1.52	U 22.5	U 16.7	U 1.52	66.6
Northern Light Lake	U 16	U 1.68	U 16	U 1.6	U 1.6	U 34.1	36.8	U 1.6	184
WWTP									
Hinckley-up	U 14.9	U 1.49	U 14.9	U 1.49	U 1.49	U 13.7	U 25.6	U 1.49	U 39.9
Sauk-up	U 14.9	U 1.49	U 15.3	U 1.49	U 1.49	U 11	U 24.2	U 1.49	U 47
Hinckley-down	U 14.9	U 1.49	U 14.9	U 1.49	11.1	U 17.2	U 23.7	U 1.49	U 94.4
Sauk-down	U 14.8	U 1.48	U 14.8	U 1.48	6.17	U 9.79	U 10.2	U 1.48	U 45.4
Marshall-up	U 15.6	U 1.56	37	U 1.56	U 1.56	U 8.89	U 16.7	U T 1.56	U 49.4
Marshall-down	U 15.5	U 1.55	80.1	U 1.55	35.7	U 17.5	U 15.4	7.95	U 47.1
Metro Plant-up	U 14.8	U 1.48	U 14.8	3.23	7.62	U 19.1	U 24.4	U 1.48	U 34.8
Metro Plant-down	U 14.9	U 1.85	17	U 7.53	28.9	U 22.7	22.6	1.81	U 39.8
Field Blank 1	U 14.9	U 1.49	U 14.9	U 1.49	U 1.49	U 7.47	U 5.95	U 1.49	U 5.95
Field Blank 2	U 16.2	U 5.4	U 54	U 1.62	U 1.62	U 6.48	U 6.48	U 1.62	U 6.48

Location	Cloxacillin	Dehydronifedipine	Diphenhydramine	Diltiazem	Digoxin	Digoxigenin	Enrofloxacin	Erythromycin-H2O	Flumequine
Lake									
Lake Kabetogama	U H 10.1	U 0.79	U 0.606	U 0.44	U 6.06	U 88.3	U 3.03	U 3.03	U 3.06
Budd Lake	U H 16.7	U 0.647	U 0.647	U 1.01	U 6.47	U 175	U 6.72	U 2.48	U 5.38
Sullivan Lake	U H 16	U 2.49	U 0.64	U 1.28	U 6.4	U 490	U 6.39	U 2.45	U 7.21
Cedar Lake	U H 13.6	U 0.612	U 0.612	U 2.22	U 6.12	U 376	U 3.06	B 3.08	U 7.87
Lake Owasso	U H 17.2	U 0.609	U 0.609	U 1.56	U 6.09	U 564	U 4.5	B 2.75	U 14.3
White Sand Lake	U H 3	U 0.601	U 2	U 0.3	U 6.01	U 226	U 3	U 2.3	U 1.58
Red Sand Lake	U H 3.04	U 0.609	U 2.03	U 0.304	U 6.09	U 209	U 3.12	U 2.33	U 1.82
Elk Lake	U H 9.93	U 0.596	U 0.596	U 0.298	U 5.96	U 166	U 3.51	U 2.28	U 1.49
Shingobee Lake	U H 10	U 0.601	U 0.601	U 0.348	U 6.01	U 120	U 3.1	U 2.3	U 1.5
Stewart Lake	U H 3.05	U 1.13	U 0.609	U 1.7	U 6.09	U 59.9	U 3.05	U 3.05	U 2.81
Northern Light Lake	U H 3.21	U 1.54	U 0.642	1.19	U 6.42	U 109	U 3.21	3.33	U 2.35
WWTP									
Hinckley-up	U H 29.9	U 0.597	U 0.597	U 0.299	U 5.97	U 146	U 2.99	U 2.29	U 1.49
Sauk-up	U H 29.9	U 0.598	U 0.598	U 0.299	U 5.98	U 156	U 2.99	U 2.29	U 1.49
Hinckley-down	U H 29.7	0.684	2.76	1.68	U 5.95	U 244	U 2.97	4.03	U 1.49
Sauk-down	U H 29.6	U 0.591	1.52	1.1	U 5.91	U 120	U 2.96	2.3	U 1.48
Marshall-up	U H 31.1	U 0.623	U 0.623	U 0.311	U 6.23	U 170	U 3.11	U 2.39	U 2.26
Marshall-down	U H 31	0.807	6.61	6.31	U 6.2	U 314	U 3.1	13.2	U 1.55
Metro Plant-up	U H 2.98	0.715	U 0.593	2.26	U 5.93	U 108	U 2.96	4.96	U 5.14
Metro Plant-down	U H 2.98	1.08	1.47	19.5	U 5.97	U 82.7	U 2.98	10.7	U 3.44
Field Blank 1	U H 2.97	U 0.595	U 0.595	U 0.991	U 5.95	U 5.95	U 2.97	3.21	U 1.58
Field Blank 2	U H 32.4	U 0.648	U 0.648	U 0.324	U 21.6	U 6.48	U 3.24	U 5.4	U 1.62

Location	Fluoxetine	Lincomycin	Lomefloxacin	Miconazole	Norfloxacin	Norgestimate	Ofloxacin	Ormetoprim	Oxacillin	Oxolinic Acid
Lake										
Lake Kabetogama	U 1.51	U 3.03	U 5.5	U 1.51	U 72.4	U 7.91	U 3.65	U 0.606	U H 3.03	U 0.932
Budd Lake	U 1.62	U 3.24	U 11.7	U 2.29	U 111	U 364	U 2.7	U 0.647	U H 8.78	U 5.07
Sullivan Lake	U 1.6	U 3.2	U 15.2	U 2.18	U 99	U 37	U 5.03	U 0.64	H 7.65	U 8.31
Cedar Lake	U 1.53	U 3.06	U 9.83	U 2.95	U 109	U 33.5	U 5.31	U 0.612	U H 6.41	U 3.05
Lake Owasso	U 1.52	U 3.05	U 8.52	U 3.03	U 170	U 38.3	U 4.61	U 0.609	U H 8.48	U 6.85
White Sand Lake	U 1.5	U 3	U 14.7	U 1.5	U 47.9	U 5.99	U 3.59	U 0.601	U H 3	U 2.42
Red Sand Lake	U 1.52	U 3.04	U 9.7	U 1.52	U 73.4	U 7.06	U 4.14	U 0.609	U H 3.04	U 2.38
Elk Lake	U 4.96	U 2.98	U 11.3	U 1.49	U 40	U 3.52	U 4.66	U 0.596	U H 2.98	U 2.35
Shingobee Lake	U 5.01	U 3.01	U 8.55	U 1.5	U 28.9	U 3.27	U 3.53	U 0.601	U H 3.01	U 0.601
Stewart Lake	U 1.52	U 3.05	U 3.05	U 1.52	U 45.5	U 3.05	U 1.94	U 0.609	U H 3.28	U 4.74
Northern Light Lake	U 1.6	U 3.21	U 5.22	U 1.6	U 158	U 3.21	U 1.96	U 0.642	U H 3.21	U 3.79
WWTP										
Hinckley-up	U 1.49	U 2.99	U 6.26	U 1.49	U 32	U 4.2	U 1.49	U 0.597	U H 2.99	U 2.01
Sauk-up	12.1	U 2.99	U 4.69	U 1.49	U 79.7	U 4.17	U 1.49	U 0.598	U H 2.99	U 6.76
Hinckley-down	U 1.49	U 2.97	U 13.7	U 1.49	U 104	U 6.78	U 3.5	U 0.595	U H 2.97	U 0.712
Sauk-down	7.73	U 2.96	U 7.22	U 1.48	U 61.4	U 4.86	U 1.48	U 0.591	U H 2.96	U 2.51
Marshall-up	U 1.56	U 3.11	U 3.64	U 1.56	U 53.3	U 3.72	U 1.56	U 0.623	U H 3.11	U 0.623
Marshall-down	U 1.55	U 3.1	U 7.31	U 1.55	U 56.8	U 4.82	23.4	U 0.62	U H 3.1	U 1.18
Metro Plant-up	U 1.48	U 2.96	U 3.6	U 1.48	U 49	U 2.96	U 1.62	U 0.593	U H 3.15	U 2.11
Metro Plant-down	U 1.49	U 2.98	U 4.29	U 1.49	U 74.1	U 2.98	6.48	U 0.597	U H 2.98	U 1.42
Field Blank 1	U 1.49	U 2.97	U 2.97	U 1.49	U 14.9	U 2.97	U 1.49	U 0.595	U H 2.97	U 0.595
Field Blank 2	U 1.62	U 3.24	B 6.66	U 1.62	U 16.2	U 3.24	U 1.62	U 0.648	U H 162	U 0.648

Location	Penicillin G	Penicillin V	Roxithromycin	Sarafloxacin	Sulfachloropyridazine	Sulfadiazine	Sulfadimethoxine	Sulfamerazine	Sulfamethazine
Lake									
Lake Kabetogama	U H 3.03	U 4.37	U 0.303	U 15.1	U 1.51	U 1.51	U 1.09	U 2.67	U 4.44
Budd Lake	U H 7.87	U 12.4	U 0.324	U 16.2	U 5.16	U 1.62	U 2.51	U 0.837	U 4.88
Sullivan Lake	U H 6.44	U 10.7	U 0.32	U 16	U 1.6	U 1.6	U 1.04	U 2.02	U 4.12
Cedar Lake	U H 4.08	U 14.6	U 0.306	U 15.3	U 1.53	U 1.53	U 1.57	U 1.21	U 4.35
Lake Owasso	U H 5.14	U 11.9	U 0.305	U 15.2	U 6	U 1.52	U 14.6	U 2.36	U 0.609
White Sand Lake	U H 3	U 3	U 0.3	U 15	U 1.5	U 1.5	U 0.3	U 0.601	U 2.31
Red Sand Lake	U H 3.04	U 3.04	U 0.304	U 16	U 1.52	U 1.52	U 0.519	U 1.08	U 2.74
Elk Lake	U H 2.98	U 2.98	U 0.298	U 14.9	U 3.28	U 1.49	U 1.32	U 1	U 1.99
Shingobee Lake	U H 3.01	U 3.01	U 0.301	U 15	U 2.66	U 1.5	U 0.301	U 0.779	U 0.76
Stewart Lake	U H 3.05	U 3.27	U 0.333	U 15.2	3.83	U 1.52	U 1.2	U 1.03	U 4.11
Northern Light Lake	U H 3.21	U 5.79	U 0.443	U 16	U 5.54	U 1.6	U 2.28	U 1.98	U 3.07
WWTP									
Hinckley-up	U H 29.9	U 2.99	U 0.299	U 14.9	U 1.49	U 1.49	U 0.365	U 0.629	U 1.02
Sauk-up	U H 29.9	U 2.99	U 0.299	U 14.9	U 1.49	U 1.49	U 0.299	U 0.598	U 1.55
Hinckley-down	U H 29.7	U 2.97	U 0.297	U 14.9	U 1.49	U 1.49	U 0.699	U 0.734	U 1.47
Sauk-down	U H 29.6	U 2.96	U 0.296	U 14.8	U 1.48	U 1.48	U 0.484	U 0.679	U 2.17
Marshall-up	U H 31.1	U 3.11	U T 0.311	U 15.6	NQ	NQ	NQ	NQ	U H 5.81
Marshall-down	U H 31	U 3.1	U 0.31	U 15.5	U 1.55	U 1.55	4.58	U 0.928	U 0.62
Metro Plant-up	U H 2.96	U 4.34	U 0.323	U 14.8	U 1.48	U 1.48	0.804	U 0.593	30.9
Metro Plant-down	U H 2.98	U 2.98	U 0.33	U 14.9	U 1.49	U 1.49	U 2.1	U 0.67	25.6
Field Blank 1	U H 2.97	U 2.97	U 0.297	U 14.9	U 1.49	U 1.49	U 0.297	U 0.595	U 0.595
Field Blank 2	U H 32.4	U 3.24	U 1.08	U 16.2	U 1.62	U 1.62	U 0.324	U 0.648	U 0.648

Location	Sulfamethizole	Sulfamethoxazole	Sulfanilamide	Sulfathiazole	Thiabendazole	Trimethoprim	Tylosin	Virginiamycin M1	1,7-Dimethylxanthine
Lake									
Lake Kabetogama	U 3.04	U 1.77	U 50.5	U 1.51	U 1.51	U 3.59	U 6.06	U 8.09	U 60.6
Budd Lake	U 1.38	U 1.49	U 16.2	U 2.29	U 1.62	U 2.93	U 6.47	U 39	U 64.7
Sullivan Lake	U 2.07	U 0.64	U 16	U 2.19	U 1.6	U 9.75	U 6.4	U 35.2	U 64
Cedar Lake	U 2.68	U 0.612	U 15.3	U 1.53	12.7	U 9.07	U 6.12	U 38.7	U 61.2
Lake Owasso	U 3.61	U 0.609	U 15.2	U 2.15	U 1.52	U 5.92	U 6.09	U 44.8	U 60.9
White Sand Lake	U 1.15	U 1.15	NQ	U 1.5	U 1.5	U 1.5	U 6.01	U 3	U 60.1
Red Sand Lake	U 1.09	U 2.42	NQ	U 1.52	U 1.52	U 1.52	U 6.09	U 15.6	U 60.9
Elk Lake	U 0.596	U 0.769	U 93.1	U 1.49	U 1.49	U 1.49	U 5.96	U 9.93	U 59.6
Shingobee Lake	U 0.601	U 0.601	U 83.9	U 1.5	U 1.5	U 1.5	U 6.01	U 14.3	U 60.1
Stewart Lake	U 1.99	U 0.609	U 15.2	U 1.58	U 1.52	3.63	U 6.09	U 14.5	U 60.9
Northern Light Lake	U 2.99	U 0.642	U 16	U 1.76	U 1.6	2.87	U 6.42	U 15.3	U 64.2
WWTP									
Hinckley-up	U 1.96	U 0.597	U 78.5	U 1.49	U 1.49	U 1.49	U 5.97	27.3	U 199
Sauk-up	U 0.812	U 0.598	U 80.5	U 1.49	U 1.49	U 1.49	U 5.98	U 15	U 199
Hinckley-down	U 1.07	14.6	U 67.5	U 1.49	U 1.49	4.68	U 5.95	U 19	U 198
Sauk-down	U 2.64	12.3	U 60.2	U 1.48	U 1.48	3.84	U 5.91	U 13.3	U 197
Marshall-up	NQ	U H 0.623	NQ	NQ	U 1.56	U 1.56	U T 6.23	U 14.5	U 208
Marshall-down	U 0.709	72.7	U 119	U 1.55	U 1.55	U 1.55	8.1	U 21	U 207
Metro Plant-up	U 1.58	52.8	U 14.8	U 1.48	U 1.48	9.37	U 5.93	67.6	U 59.3
Metro Plant-down	U 1.8	186	U 14.9	U 1.49	1.81	38.1	U 5.97	34	U 59.7
Field Blank 1	1.05	U 0.595	U 14.9	U 1.49	U 1.49	U 1.49	U 5.95	U 2.97	U 59.5
Field Blank 2	U 0.648	U 0.648	U 16.2	U 1.62	U 1.62	U 1.62	U 6.48	U 3.24	U 64.8

Pharmaceuticals list 2

Location	Anhydrochlortetracycline [ACTC]	Anhydrotetracycline [ATC]	Chlortetracycline [CTC]	Demeclocycline	Doxycycline
Lake					
Lake Kabetogama	U 50.5	U 50.5	U 10.6	U 15.2	U 7.61
Budd Lake	U 16.2	U 16.2	U 6.47	U 16.2	U 6.47
Sullivan Lake	U 16	U 16	U 10.1	U 16	U 6.4
Cedar Lake	U 15.3	U 15.3	U 6.56	U 15.3	U 6.12
Lake Owasso	U 15.2	U 15.2	U 6.09	U 15.2	U 6.09
Stewart Lake	B 26.1	U 53.2	U 53.6	U 24.5	U 22.9
Northern Light Lake	U 28.3	U 58.4	U 59.1	U 28.4	U 24.9
White Sand Lake	U 17.6	U 19	U 25.2	U 23.5	U 7.62
Red Sand Lake	U 22.6	U 19.9	U 25.3	U 25.1	U 7.84
Elk Lake	U 14.9	U 18	U 28.8	U 14.9	U 6.58
Shingobee Lake	U 15	U 15	U 25.7	U 15	U 6.15
WWTP					
Hinckley-up	U 14.9	U 14.9	U 19.9	U 21.2	U 6.69
Sauk-up	U 17.1	U 14.9	U 19.9	U 14.9	U 6.67
Hinckley-down	U 17.7	U 14.9	U 19.8	U 16.7	U 6.98
Sauk-down	U 17.1	U 14.8	U 19.7	U 18.3	U 6.18
Marshall-up	U 130	U 19.1	U 10.9	U 15.6	U 6.66
Marshall-down	U 129	U 17.2	U 12.4	U 15.9	U 6.2
Metro Plant Up	B 18.5	U 55.2	U 52.5	U 19.7	U 22.6
Metro Plant Down	B 17.8	U 64.3	U 52.6	U 14.9	U 23
Field Blank 1	B 17.1	B 51.2	U 50.5	U 14.9	U 21.8
Field Blank 2	U 16.5	U 54	U 6.48	U 16.2	U 21.6

Location	4-Epianhydrochlortetracycline [EACTC]	4-Epianhydrotetracycline [EATC]	4-Epichlortetracycline [ECTC]	4-Epioxytetracycline [EOTC]	4-Epitetracycline [ETC]
Lake					
Lake Kabetogama	U 202	U 50.5	U 22.6	U 11	U 8.33
Budd Lake	U 64.7	U 16.2	U 12.5	U 6.47	U 6.54
Sullivan Lake	U 64	U 16	U 20.7	U 6.4	U 6.4
Cedar Lake	U 61.2	U 15.3	U 14.1	U 6.12	U 6.12
Lake Owasso	U 60.9	U 15.2	U 13.2	U 6.09	U 6.09
Stewart Lake	B 71.5	U 18.4	U 23	U 14.6	U 9.31
Northern Light Lake	B 84.9	U 27.6	U 40.1	U 27.4	U 12.4
White Sand Lake	U 60.1	U 23.4	U 25.1	U 21.9	U 10.6
Red Sand Lake	U 60.9	U 25	U 24.6	U 29.3	U 11.5
Elk Lake	U 59.6	U 14.9	U 24.2	U 9.61	U 6.43
Shingobee Lake	U 60.1	U 15	U 15	U 6.4	U 6.01
WWTP					
Hinckley-up	U 59.7	U 14.9	U 21.4	U 14.1	U 10.6
Sauk-up	U 59.8	U 16.5	U 20.7	U 10.7	U 13.3
Hinckley-down	U 59.5	U 18.5	U 19.6	U 16.4	U 10.9
Sauk-down	U 59.1	U 19.2	U 14.8	U 13	U 13.8
Marshall-up	U 62.3	U 19.9	U 17.8	U 8.81	U 8.65
Marshall-down	U 62	U 16	U 21.2	U 16.3	U 7.32
Metro Plant Up	B 69.6	U 31	U 25	U 22.3	U 6.65
Metro Plant Down	U 59.7	U 63.6	U 23.6	U 14.7	U 8.3
Field Blank 1	U 59.5	U 14.9	U 14.9	U 5.95	U 5.95
Field Blank 2	U 64.8	U 16.2	U 54	U 6.48	U 21.6

Location	Isochlortetracycline [ICTC]	Minocycline	Oxytetracycline [OTC]	Tetracycline [TC]
Lake				
Lake Kabetogama	U 10.8	U 60.6	U 7.25	U 7.32
Budd Lake	U 6.47	U 64.7	U 6.47	U 6.47
Sullivan Lake	U 7.43	U 64	U 6.4	U 6.4
Cedar Lake	U 6.12	U 61.2	U 6.12	U 6.12
Lake Owasso	U 7.5	U 60.9	U 6.09	U 6.09
Stewart Lake	U 8.17	U 77	U 50.8	U 50.8
Northern Light Lake	U 13	U 102	U 53.5	U 53.5
White Sand Lake	U 7.32	U 60.1	U 20	U 21.8
Red Sand Lake	U 8.41	U 60.9	U 24.3	U 22.5
Elk Lake	U 10.4	U 59.6	U 19.9	U 20
Shingobee Lake	U 6.13	U 60.1	U 20	U 20
WWTP				
Hinckley-up	U 5.97	U 64.9	U 9.67	U 9.17
Sauk-up	U 6.7	U 73.3	U 7.78	U 11.2
Hinckley-down	U 6.34	U 70.1	U 11	U 9.39
Sauk-down	U 5.91	U 63.8	U 9.05	U 11.5
Marshall-up	U 6.23	U 78.7	U 7.81	U 8.26
Marshall-down	U 6.2	U 62	U 11.8	U 7.34
Metro Plant Up	U 13.1	U 106	U 49.4	U 49.4
Metro Plant Down	U 10.7	U 105	U 49.7	U 49.7
Field Blank 1	U 5.95	U 59.5	U 49.6	U 49.6
Field Blank 2	U 6.48	U 64.8	U 21.6	U 6.48

Pharmaceuticals list 3

Location	Bisphenol A	Furosemide	Gemfibrozil	Glipizide	Glyburide	Hydrochlorothiazide
Lake						
Lake Kabetogama	U 505	U 40.4	U 5.05	U 6.06	U 3.03	U 20.2
Budd Lake	U 540	U 71	2.07	U 6.47	U 2.24	U 21.6
Cedar Lake	U 510	U 40.8	U 1.53	U 6.12	U 2.12	U 20.4
Elk Lake	U 496	U 39.7	U 1.49	U 5.96	U 2.98	U 19.9
Lake Owasso	U 508	U 40.6	U 1.52	U 6.09	U 2.11	U 20.3
Northern Light Lake	U 535	U 42.8	U 5.35	U 6.42	U 3.21	U 21.4
Red Sand Lake	U 507	U 40.6	U 1.52	U 6.09	U 3.04	U 20.3
Shingobee Lake	U 501	U 40.1	U 1.5	U 6.01	U 3.01	U 20
Stewart Lake	U 508	U 40.6	U 5.08	U 6.09	U 3.05	U 20.3
Sullivan Lake	U 534	U 42.7	U 1.6	U 6.4	2.92	U 21.3
White Sand Lake	U 504	U 40.3	U 1.51	U 6.05	U 3.03	U 20.2
WWTP						
Hinckley-up	U 498	U 39.8	2.33	U 5.97	U 2.99	U 199
Hinckley-down	U 496	U 39.7	2.3	U 5.95	U 2.97	U 198
Marshall-down	U 517	U 41.4	5.73	U 6.2	U 3.1	U 207
Marshall-up	U 519	U 41.5	U 1.56	U 6.23	U 3.11	U 208
Metro Plant Down	U 497	67	11.1	U 5.97	U 2.98	25.4
Metro Plant Up	U 494	U 39.5	U 4.94	U 5.93	U 2.96	U 19.8
Sauk-down	U 493	U 39.4	1.49	U 5.91	U 2.96	U 197
Sauk-up	U 498	U 39.8	U 1.49	U 5.98	U 2.99	U 199
Field Blank 1	U 496	U 39.7	U 4.96	U 5.95	U 2.97	U 19.8
Field Blank 2	U 540	U 43.2	U 1.62	U 6.48	U 3.24	U 13

Location	2-Hydroxy-ibuprofen	Ibuprofen	Naproxen	Triclocarban	Triclosan	Warfarin
Lake						
Lake Kabetogama	U 97.4	U 15.1	U 14.7	U 3.03	U 60.6	U 1.51
Budd Lake	U 86.3	U 16.2	U 10.8	3.28	U 64.7	U 1.62
Cedar Lake	U 81.6	U 15.3	U 10.2	U 3.06	U 61.2	U 1.53
Elk Lake	U 79.4	U 14.9	U 2.98	U 2.98	U 59.6	U 1.49
Lake Owasso	U 81.3	U 15.2	U 10.2	U 3.05	U 60.9	U 1.52
Northern Light Lake	U 85.6	U 16	U 10.7	U 3.21	U 64.2	U 1.6
Red Sand Lake	U 81.1	U 15.2	U 3.04	U 3.04	U 60.9	U 1.52
Shingobee Lake	U 80.1	U 15	U 3.01	U 3.01	U 60.1	U 1.5
Stewart Lake	U 81.2	U 15.2	U 10.2	U 3.05	U 60.9	U 1.52
Sullivan Lake	U 85.4	U 16	U 10.7	U 3.2	U 64	U 1.6
White Sand Lake	U 80.7	U 15.1	3.85	U 3.03	U 60.5	U 1.51
WWTP						
Hinckley-up	U 79.7	U 14.9	U 2.99	U 9.96	U 59.7	U 1.49
Hinckley-down	U 79.3	U 14.9	U 2.97	U 9.92	U 59.5	U 1.49
Marshall-down	U 82.7	U 15.5	9.75	U 3.1	U 62	U 1.55
Marshall-up	U 83	U 15.6	U 3.11	U 3.11	U 62.3	U 1.56
Metro Plant Down	U 79.6	U 14.9	U 9.95	U 2.98	U 59.7	U 1.49
Metro Plant Up	U 79	U 14.8	U 9.88	U 2.96	U 59.3	U 1.48
Sauk-down	U 78.8	U 14.8	U 2.96	U 9.85	U 59.1	U 1.48
Sauk-up	U 79.7	U 17.2	U 2.99	U 9.96	U 59.8	U 1.49
Field Blank 1	U 79.3	U 16.1	U 9.91	U 2.97	U 59.5	U 1.49
Field Blank 2	U 86.4	U 16.2	U 10.8	U 3.24	U 64.8	U 1.62

Pharmaceuticals list 4

Location	Albuterol	Amphetamine	Atenolol	Atorvastatin	Cimetidine	Clonidine	Codeine
Lake							
Budd Lake	U 0.857	K B 2.61	U 1.88	U 1.57	U 0.629	U 1.57	U 3.15
Cedar Lake	U 0.322	K B 5.2	U 1.14	U 1.76	U 0.622	U 2.49	U 4.63
Elk Lake	B 0.399	U 1.5	U 0.6	U 1.62	U 0.648	U 1.62	U 3.24
Lake Kabetogama	U 0.303	U 1.52	U 0.606	U 1.52	U 0.606	U 1.52	U 3.03
Lake Owasso	U 0.302	K B 6.37	U 1.08	U 1.51	U 0.604	U 1.66	U 8.6
Northern Light Lake	U 0.296	U 1.48	U 0.592	U 1.48	U 0.592	U 1.48	U 2.96
Red Sand Lake	U 0.296	U 1.48	U 0.731	U 1.48	U 0.592	U 1.48	U 2.96
Shingobee Lake	B 0.484	2.91	U 0.607	U 1.52	U 0.607	U 1.52	U 3.04
Stewart Lake	U 0.305	U 1.53	U 0.61	U 1.53	U 0.61	U 1.53	U 3.05
Sullivan Lake	U 0.3	K B 7.27	U 1.31	U 1.5	U 0.6	U 1.5	U 6.32
White Sand Lake	U 0.299	U 1.5	U 0.655	U 1.5	U 0.598	U 1.5	U 2.99
WWTP							
Marshall-up	U 0.312	U 1.96	U 0.651	U 1.56	U 0.625	U 1.56	U 3.12
Hinckley-down	U 0.295	U 1.48	4.18	U 1.48	0.737	U 1.48	U 2.95
Hinckley-up	U 0.298	U 1.49	U 0.596	U 1.49	U 0.596	U 1.49	U 2.98
Marshall-down	0.573	U 2.42	26.3	U 1.5	7.1	U 1.5	3.15
Metro Plant Down	1.9	U 1.49	8.99	U 1.49	U 0.597	U 1.49	4.4
Metro Plant Up	0.314	U 1.51	1.67	U 1.51	U 0.605	U 1.51	U 3.02
Sauk-down	U 0.301	1.65	1.4	U 1.51	0.662	U 1.51	U 3.01
Sauk-up	U 0.299	U 1.5	U 0.599	U 1.5	U 0.599	U 1.5	U 2.99
Field Blank 1	U 0.299	U 1.5	U 0.598	U 1.5	U 0.598	U 1.5	U 2.99
Field Blank 2	U 0.318	U 1.59	U 0.636	U 1.59	U 0.636	U 1.59	U 3.18

Location	Cotinine	Enalapril	Hydrocodone	Metformin	Oxycodone	Ranitidine	Triamterene
Lake							
Budd Lake	12.8	U 0.349	U 1.57	U 12.4	U 3.32	U 1.22	U 1.15
Cedar Lake	42.1	U 0.558	U 1.56	U 6.27	U 5.18	U 0.843	U 1.19
Elk Lake	2.13	U 0.343	U 1.62	3.01	U 1.71	U 0.648	B 0.454
Lake Kabetogama	U 1.52	U 0.303	U 1.52	U 3.03	U 0.606	U 0.606	U 0.303
Lake Owasso	15.6	U 0.302	U 1.51	U 6.35	U 4.51	U 0.701	U 0.877
Northern Light Lake	U 1.48	U 0.296	U 1.48	U 2.96	U 1.17	U 0.592	U 0.296
Red Sand Lake	3.61	U 0.298	U 1.48	8.41	U 0.592	U 0.592	U 0.306
Shingobee Lake	U 1.52	U 0.317	U 1.52	18	U 1.32	U 0.607	U 0.304
Stewart Lake	1.83	U 0.305	U 1.53	U 3.05	U 0.656	U 0.61	U 0.305
Sullivan Lake	6.06	U 0.594	U 1.5	U 10	U 1.13	U 0.826	U 1.05
White Sand Lake	6.87	U 0.299	U 1.5	4.06	U 0.598	U 0.598	U 0.335
WWTP							
Marshall-up	5.19	U 0.312	U 1.56	31.8	U 0.848	U 2.08	U 0.312
Hinckley-down	3.91	U 0.295	2.96	52.4	2.75	2.04	9.86
Hinckley-up	1.74	U 0.298	U 1.49	4.02	U 0.596	U 0.596	U 0.362
Marshall-down	8.32	U 0.301	1.94	434	U 1.45	21.2	10.9
Metro Plant Down	5.74	U 0.298	5.2	188	8.8	24.2	18.1
Metro Plant Up	4.09	U 0.302	U 1.51	53.9	U 0.915	0.744	3.19
Sauk-down	3.6	U 0.301	U 1.51	83.3	U 0.602	2.73	1.83
Sauk-up	3.18	U 0.299	U 1.5	5.32	U 0.599	U 0.599	U 0.364
Field Blank 1	U 1.5	U 0.299	U 1.5	U 2.99	U 0.598	U 0.598	U 0.299
Field Blank 2	U 1.59	U 0.318	U 1.59	U 3.18	U 0.636	U 0.636	U 0.318

Pharmaceuticals list 5

Location	Alprazolam	Amitriptyline	Amlodipine	Benzoylcegonine	Benztropine	Betamethasone	Cocaine
Lake							
Budd Lake	U 0.324	U 1.15	U 1.62	U 0.618	U 0.755	U 7.21	U 0.225
Cedar Lake	U 0.306	U 1.23	U 1.53	U 0.852	U 0.714	U 3.28	U 0.27
Elk Lake	U 0.298	U 0.365	U 1.49	U 0.298	U 0.695	U 1.49	U 0.149
Lake Kabetogama	U 0.303	U 0.318	U 1.51	U 0.434	U 0.707	U 21.3	U 0.157
Lake Owasso	U 0.305	U 1.11	U 1.52	U 0.746	U 0.711	U 20	U 0.423
Northern Light Lake	U 0.321	U 0.554	U 1.6	U 0.321	U 0.749	U 5.35	U 0.16
Red Sand Lake	U 0.304	0.503	U 1.52	U 0.403	U 0.71	U 5.07	U 0.152
Shingobee Lake	U 0.301	U 0.301	U 1.5	U 0.301	U 0.701	U 1.5	U 0.15
Stewart Lake	U 0.305	U 0.458	U 1.52	U 0.342	U 0.711	U 12	U 0.152
Sullivan Lake	U 0.32	U 0.666	U 1.6	U 0.864	U 0.747	U 10.2	U 0.362
White Sand Lake	U 0.3	0.493	U 1.5	U 0.333	U 0.701	U 5	U 0.15
WWTP							
Hinckley-up	U 0.299	0.471	U 1.49	U 0.299	U 0.697	U 2.32	U 0.149
Hinckley-down	U 0.297	0.724	U 1.49	U 0.297	U 0.694	U 4.96	0.172
Marshall-down	U 0.31	0.454	U 1.55	0.43	U 0.724	U 1.55	0.291
Marshall-up	U 0.311	U 0.311	U 1.56	U 0.311	U 0.727	U 1.56	0.203
Metro Plant Down	0.339	U 1.39	U 1.49	4.14	U 0.696	U 4.97	0.173
Metro Plant Up	U 0.296	U 0.296	U 1.48	0.469	U 0.691	U 4.94	U 0.148
Sauk-down	U 0.296	0.664	U 1.48	U 0.296	U 0.69	U 4.93	U 0.148
Sauk-up	U 0.299	U 0.44	U 1.49	U 0.348	U 0.697	U 4.98	U 0.155
Field Blank 1	U 0.297	U 0.562	U 1.49	U 0.297	U 0.694	U 4.96	U 0.149
Field Blank 2	U 0.324	U 0.324	U 1.62	U 0.324	U 0.756	U 1.62	U 0.162

Location	DEET	Desmethyldiltiazem	Diazepam	Fluocinonide	Fluticasone propionate	Hydrocortisone	10-hydroxy-amitriptyline
Lake							
Budd Lake	18.3	U 0.162	U 0.515	U 6.47	U 3.46	U 99	U 0.18
Cedar Lake	103	U 0.153	U 0.929	U 6.87	U 3.66	U 84.5	U 0.264
Elk Lake	16.6	U 0.149	U 0.298	U 8.18	U 1.99	U 59.6	U 0.149
Lake Kabetogama	B 5.29	U 0.151	U 0.303	U 6.06	U 2.02	U 60.6	U 0.151
Lake Owasso	52	U 0.152	U 0.465	U 6.3	U 3.34	U 99.7	U 0.167
Northern Light Lake	13.8	U 0.163	U 0.333	U 11.7	U 2.45	U 77.3	U 0.16
Red Sand Lake	37.1	U 0.152	U 0.304	U 7.08	U 2.84	U 60.9	U 0.152
Shingobee Lake	10.6	U 0.15	U 0.301	U 6.38	U 2	U 60.1	U 0.15
Stewart Lake	33.2	U 0.152	U 0.305	U 6.09	U 2.03	U 60.9	U 0.152
Sullivan Lake	16.5	U 0.16	U 0.398	U 7.46	U 2.47	U 81	U 0.235
White Sand Lake	69.3	U 0.15	U 0.3	U 6.08	U 2.33	U 60.1	U 0.15
WWTP							
Hinckley-up	B 16	U 0.149	U 0.342	U 6.45	U 1.99	U 66.2	U 0.149
Hinckley-down	B 18.1	0.362	U 0.297	U 5.95	U 1.98	U 97.5	U 0.149
Marshall-down	20.3	1.27	U 0.31	U 8.05	U 2.67	U 93.8	1.45
Marshall-up	7.17	U 0.156	U 0.311	U 8.83	U 2.47	U 102	U 0.156
Metro Plant Down	B 9.55	3.33	U 0.332	U 6.25	U 2	U 69.7	0.681
Metro Plant Up	11.2	0.273	U 0.296	U 7.6	U 2.21	U 85.5	U 0.148
Sauk-down	B 20.3	0.378	U 0.296	U 5.91	U 1.97	U 115	0.157
Sauk-up	B 26.4	U 0.149	U 0.299	U 6.44	U 1.99	U 111	U 0.149
Field Blank							
Field Blank 1	B 8.48	U 0.149	U 0.297	U 5.95	U 1.98	U 59.5	U 0.149
Field Blank 2	0.951	U 0.168	U 0.324	U 6.48	U 2.16	U 216	U 0.162

Location	Meprobamate	Methylprednisolone	Metoprolol	Norfluoxetine	Norverapamil	Paroxetine	Prednisolone
Lake							
Budd Lake	U 4.49	U 30.2	U 16.8	U 1.62	U 0.172	U 4.32	U 6.47
Cedar Lake	U 4.22	U 22.1	U 19.6	U 1.53	0.187	U 4.08	U 6.12
Elk Lake	U 3.97	U 6.32	U 5.32	U 1.49	U 0.149	U 3.97	U 25.2
Lake Kabetogama	U 4.04	U 10	U 6.4	U 1.51	U 0.151	U 4.04	U 25.9
Lake Owasso	U 4.06	U 17	U 27.3	U 1.52	U 0.152	U 4.06	U 6.09
Northern Light Lake	U 4.28	U 14.6	U 6.88	U 1.6	U 0.16	U 4.28	U 45.1
Red Sand Lake	U 4.06	U 4.06	U 11.2	U 1.52	U 0.152	U 4.06	U 6.09
Shingobee Lake	U 4.01	U 8.26	U 5.46	U 1.5	U 0.15	U 4.01	U 22.5
Stewart Lake	U 4.06	U 9.24	U 7.23	U 1.52	U 0.152	U 4.06	U 30.9
Sullivan Lake	U 4.41	U 31.2	U 19.4	U 1.6	U 0.186	U 4.27	U 6.4
White Sand Lake	U 4	U 4	U 8.28	U 1.5	U 0.15	U 4	U 6.01
WWTP							
Hinckley-up	U 5.18	U 7.92	U 9.68	U 1.49	U 0.149	U 3.98	U 36.8
Hinckley-down	14.9	U 26.9	16.4	U 1.49	U 0.153	U 3.97	U 21.5
Marshall-down	9.27	U 4.74	34.5	U 1.55	U 0.155	U 4.14	U 42.1
Marshall-up	U 4.15	6.06	U 6.57	U 1.56	U 0.156	U 4.15	U 21.5
Metro Plant Down	18.3	U 18.7	51	U 1.49	U 0.149	U 3.98	U 22.2
Metro Plant Up	U 10.8	U 7.48	7.79	U 1.48	U 0.148	U 3.95	U 18.2
Sauk-down	U 3.94	U 11.6	U 7.33	U 1.48	U 0.148	U 3.94	U 18.2
Sauk-up	U 3.98	U 44.3	U 9.02	U 1.49	U 0.149	U 3.98	U 47.6
Field Blank 1	U 3.97	U 3.97	U 1.49	U 1.49	U 0.149	U 3.97	U 5.95
Field Blank 2	U 4.32	U 4.32	U 1.62	U 1.62	U 0.162	U 4.32	U 6.72

Location	Prednisone	Promethazine	Propoxyphene	Propranolol	Sertraline	Simvastatin
Lake						
Budd Lake	U 166	U 0.432	U 0.324	U 2.16	U 0.432	U 21.6
Cedar Lake	U 153	U 0.408	U 0.306	U 2.04	U 0.408	U 20.4
Elk Lake	U 53	U 0.397	U 0.298	U 1.99	U 0.397	U 19.9
Lake Kabetogama	U 57.7	U 0.415	U 0.303	U 2.02	12.7	U 20.2
Lake Owasso	U 125	U 0.503	U 0.305	U 2.03	U 0.443	U 20.3
Northern Light Lake	U 97.4	U 0.428	U 0.321	U 2.14	U 0.428	U 21.4
Red Sand Lake	U 167	U 0.406	U 0.304	U 2.03	U 0.406	U 20.3
Shingobee Lake	U 51.5	U 0.401	U 0.301	U 2	U 0.401	U 20
Stewart Lake	U 81.1	U 0.406	U 0.305	U 2.03	12.7	U 20.3
Sullivan Lake	U 148	U 0.427	U 0.32	U 2.13	U 0.453	U 21.3
White Sand Lake	U 63.7	U 0.4	U 0.3	U 2	U 0.4	U 20
WWTP						
Hinckley-up	U 93.2	U 0.398	U 0.299	U 1.99	0.482	U 19.9
Hinckley-down	U 49.6	U 0.397	U 0.297	2.12	0.853	U 19.8
Marshall-down	U 78.9	U 0.414	U 0.31	U 2.07	0.659	U 20.7
Marshall-up	U 69.6	U 0.415	U 0.311	U 2.08	U 0.415	U 20.8
Metro Plant Down	U 113	U 0.398	U 0.298	8.6	3.48	U 19.9
Metro Plant Up	U 91.2	U 0.395	U 0.296	U 1.98	U 0.395	U 19.8
Sauk-down	U 52.9	U 0.394	U 0.296	U 1.97	0.409	U 19.7
Sauk-up	U 129	U 0.398	U 0.299	U 1.99	U 0.398	U 19.9
Field Blank 1	U 19.8	U 0.397	U 0.297	U 1.98	U 0.397	U 19.8
Field Blank 2	U 21.6	U 1.7	U 0.324	U 2.16	0.811	U 21.6

Location	Theophylline	Trenbolone	Trenbolone acetate	Valsartan	Verapamil
Lake					
Budd Lake	U 123	U 11.4	U 1.04	U 4.32	U 0.196
Cedar Lake	U 119	U 7.86	U 1.51	U 4.35	U 0.164
Elk Lake	U 59.6	U 3.97	U 0.418	U 3.97	U 0.149
Lake Kabetogama	U 60.6	U 4.04	U 0.337	U 4.04	U 0.151
Lake Owasso	U 101	U 6.12	U 0.928	U 4.06	U 0.31
Northern Light Lake	U 64.2	U 4.28	U 0.741	U 4.28	U 0.16
Red Sand Lake	U 101	U 5.59	U 0.375	U 4.06	U 0.152
Shingobee Lake	U 60.1	U 4.01	U 0.315	U 4.01	U 0.15
Stewart Lake	U 83.8	U 4.06	U 0.44	U 4.06	U 0.152
Sullivan Lake	U 125	U 5.31	U 0.695	U 4.27	U 0.16
White Sand Lake	U 85.3	U 4	U 0.369	U 4	U 0.15
WWTP					
Hinckley-up	U 199	U 3.98	U 0.332	U 3.98	U 0.149
Hinckley-down	U 119	U 4.69	U 0.297	7.6	0.521
Marshall-down	94.6	U 4.14	U 0.541	9.66	0.166
Marshall-up	U 79.8	U 5.37	U 0.54	U 4.15	U 0.156
Metro Plant Down	U 59.7	U 3.98	U 0.442	13.2	0.155
Metro Plant Up	U 59.3	U 3.95	U 0.423	4.44	U 0.148
Sauk-down	U 94.4	U 3.94	U 0.339	U 3.94	0.35
Sauk-up	U 133	U 4.88	U 0.398	U 3.98	U 0.149
Field Blank 1	U 59.5	U 3.97	U 0.297	U 3.97	U 0.149
Field Blank 2	U 64.8	U 4.32	U 0.324	U 4.32	U 0.162

Pharmaceuticals list 6

Location	Diatrizoic acid	Iopamidol	Citalopram	Tamoxifen	Cyclophosphamide	Venlafaxine	Amsacrine
Lake							
Budd Lake	U 43.2	U 184	U 0.861	U 0.432	U 1.73	U 0.432	U 0.863
Cedar Lake	U 40.8	212	U 1.52	U 0.408	U 1.63	U 0.408	U 0.816
Elk Lake	U 39.7	U 79.4	U 1.17	U 0.397	U 1.59	U 1.19	U 0.794
Lake Kabetogama	U 40.4	510	U 1.18	U 4.04	U 1.61	U 4.04	U 0.807
Lake Owasso	U 40.6	273	U 1.29	U 0.406	U 1.63	U 0.406	U 0.813
Northern Light Lake	U 42.8	U D 257	U 0.426	U 4.28	U 1.71	D 8.35	U 0.856
Red Sand Lake	U 40.6	339	U 2.78	U 0.406	U 1.62	U 4.06	U 0.811
Shingobee Lake	U 40.1	118	U 0.886	U 0.401	U 1.6	U 1.2	U 0.801
Stewart Lake	U 40.6	302	U 0.984	U 4.06	U 1.62	U 1.22	U 0.812
Sullivan Lake	U 42.7	238	U 0.999	U 0.427	U 1.71	U 0.427	U 0.854
White Sand Lake	U 40	140	U 1.16	U 0.4	U 1.6	U 4	U 0.801
WWTP							
Hinckley-down	U 39.7	U 79.3	8.5	U 0.397	U 1.59	30.5	U 0.793
Hinckley-up	U 39.8	U 79.7	U 0.389	U 0.398	U 1.59	U 1.19	U 0.797
Marshall-down	U 41.4	1230	7.3	U 0.414	U 1.65	50.4	U 0.827
Marshall-up	U 41.5	U 83	U 0.405	U 0.415	U 1.66	U 1.25	U 0.83
Metro Plant Down	91.2	522	3.87	U 3.98	U 1.59	45	U 0.796
Metro Plant Up	U 39.5	356	U 0.706	U 3.95	U 1.58	5.95	U 0.79
Sauk-down	U 39.4	U 78.8	3.41	U 0.394	U 1.58	5.9	U 0.788
Sauk-up	U 39.8	U 79.7	U 0.389	U 0.398	U 1.59	U 1.2	U 0.797
Field Blank 1	U 39.7	U 79.3	U 0.387	U 3.97	U 1.59	U 1.19	U 0.793
Field Blank 2	U 43.2	U 86.4	U 0.422	U 4.32	U 5.18	U 4.32	U 0.864

Location	Azathioprine	Busulfan	Carmustine	Chloramphenicol	Clotrimazole	Colchicine	Daunorubicin
Lake							
Budd Lake	U 8.63	U 25.9	U 86.3	U 941	U 2.16	U 2.16	U 17.3
Cedar Lake	U 8.16	U 24.5	U 81.6	U 890	U 2.04	U 2.04	U 16.3
Elk Lake	U 7.94	U 23.8	U 79.4	U 866	U 1.99	8.52	U 15.9
Lake Kabetogama	U 8.07	U 24.2	U 80.7	U 880	U 2.02	U 6.1	U 16.1
Lake Owasso	U 8.13	U 24.4	U 81.3	U 886	U 2.03	U 3.77	U 16.3
Northern Light Lake	U 8.56	U 25.7	U 85.6	U 1320	U 2.14	U 2.14	U 17.1
Red Sand Lake	U 8.11	U 24.3	U 81.1	U 884	U 2.03	U 2.03	U 16.2
Shingobee Lake	U 8.01	U 24	U 80.1	U 874	U 2	U 3.62	U 16
Stewart Lake	U 8.12	U 24.4	U 81.2	U 1680	U 2.03	U 2.03	U 16.2
Sullivan Lake	U 8.54	U 25.6	U 85.4	U 1190	U 2.13	U 2.13	U 17.1
White Sand Lake	U 8.01	U 24	U 80.1	U 873	U 2	U 4.07	U 16
WWTP							
Hinckley-down	U 7.93	U 23.8	U 79.3	U 865	U 1.98	U 3.15	U 15.9
Hinckley-up	U 7.97	U 23.9	U 79.7	U 868	U 1.99	U 1.99	U 15.9
Marshall-down	U 8.27	U 24.8	U 82.7	U 901	U 2.07	U 2.07	U 16.5
Marshall-up	U 8.3	U 24.9	U 83	U 905	U 2.08	U 2.08	U 16.6
Metro Plant Down	U 7.96	U 23.9	U 79.6	U 867	U 1.99	U 3.8	U 15.9
Metro Plant Up	U 7.9	U 23.7	U 79	U 1040	U 1.98	U 1.98	U 15.8
Sauk-down	U 7.88	U 23.6	U 78.8	U 859	U 1.97	U 1.97	U 15.8
Sauk-up	U 7.97	U 23.9	U 79.7	U 868	U 1.99	U 1.99	U 15.9
Field Blank 1	U 7.93	U 23.8	U 79.3	U 865	U 1.98	U 1.98	U 15.9
Field Blank 2	U 8.64	U 25.9	U 86.4	U 942	U 2.16	U 2.16	U 17.3

Location	Doxorubicin	Drospirenone	Etoposide	Lomustine	Medroxyprogesterone Acetate	Metronidazole
Lake						
Budd Lake	U 25.9	U 8.63	U 4.32	U 51.8	U 4.32	U 4.32
Cedar Lake	U 24.5	U 8.16	U 4.08	U 49	U 4.17	U 4.82
Elk Lake	U 23.8	U 7.94	U 3.97	U 47.7	U 3.97	U 11.9
Lake Kabetogama	U 24.2	U 8.07	U 4.04	U 48.4	U 4.04	U 4.04
Lake Owasso	U 24.4	U 8.13	U 4.06	U 48.8	U 4.06	U 5
Northern Light Lake	U 25.7	U 8.56	U 4.28	U 51.3	U 4.28	U 4.28
Red Sand Lake	U 24.3	U 8.11	U 4.06	U 48.7	U 4.06	U 4.06
Shingobee Lake	U 24	U 8.01	U 4.01	U 48.1	U 4.01	U 12
Stewart Lake	U 24.4	U 8.12	U 4.06	U 48.7	U 4.06	U 4.06
Sullivan Lake	U 25.6	U 8.54	U 4.27	U 51.2	U 4.27	U 4.4
White Sand Lake	U 24	U 8.01	U 4	U 48	U 4	U 4
WWTP						
Hinckley-down	U 23.8	U 7.95	U 3.97	U 47.6	U 3.97	U 3.97
Hinckley-up	U 23.9	U 7.97	U 3.98	U 47.8	U 3.98	U 3.98
Marshall-down	U 24.8	U 8.27	U 4.14	U 49.6	U 4.14	U 4.14
Marshall-up	U 24.9	U 8.3	U 4.15	U 49.8	U 4.15	U 4.15
Metro Plant Down	U 23.9	U 7.96	U 3.98	U 47.7	U 3.98	U 3.98
Metro Plant Up	U 23.7	U 7.9	U 3.95	U 47.4	U 3.95	U 3.95
Sauk-down	U 23.6	U 9.7	U 3.94	U 47.3	U 3.94	U 3.94
Sauk-up	U 23.9	U 7.97	U 3.98	U 47.8	U 3.98	U 3.98
Field Blank 1	U 23.8	U 7.93	U 3.97	U 47.6	U 3.97	U 3.97
Field Blank 2	U 25.9	U 8.64	NQ	U 51.8	U 4.32	U 8.36

Location	Moxifloxacin	Norethindrone	Oxazepam	Rosuvastatin	Teniposide	Zidovudine	Melphalan
Lake							
Budd Lake	U 15.1	U 69.1	U 17.3	U 17.3	U 8.63	U 51.8	U 124
Cedar Lake	U 7.54	U 65.3	U 16.3	U 16.3	U 8.16	U 49	U 217
Elk Lake	U 17.4	U 63.5	U 15.9	U 15.9	U 7.94	U 47.7	U 127
Lake Kabetogama	U 13.4	U 64.6	U 16.1	U 16.1	U 11.1	U 48.4	U 258
Lake Owasso	U 17.9	U 65	U 16.3	U 16.3	U 8.13	U 48.8	U 204
Northern Light Lake	U 9.71	U 68.4	U 17.1	U 17.1	U 9.6	U 51.3	U 132
Red Sand Lake	U 17	U 64.9	U 16.2	U 16.2	U 8.11	U 48.7	U 64.9
Shingobee Lake	U 9.53	U 64.1	U 16	U 16	U 8.01	U 48.1	U 152
Stewart Lake	U 7.67	U 65	U 16.2	U 16.2	U 8.12	U 48.7	U 232
Sullivan Lake	U 11.2	U 68.3	U 17.1	U 17.1	U 8.54	U 51.2	U 265
White Sand Lake	U 17.7	U 64.1	U 16	U 16	U 8.01	U 48	U 64.1
WWTP							
Hinckley-down	U 20.2	U 63.5	U 15.9	U 15.9	U 9.1	U 47.6	U 635
Hinckley-up	15.5	U 63.7	U 15.9	U 15.9	U 7.97	U 47.8	U 637
Marshall-down	6.94	U 66.2	U 16.5	U 16.5	U 8.27	U 49.6	U 662
Marshall-up	U 11.6	U 66.4	U 16.6	U 16.6	U 61	U 49.8	U 664
Metro Plant Down	7.63	U 63.7	U 15.9	U 15.9	U 7.96	U 47.7	U 97.9
Metro Plant Up	U 14.2	U 63.2	U 15.8	U 15.8	U 7.9	U 47.4	U 274
Sauk-down	U 9.5	U 63.1	U 15.8	U 15.8	U 7.88	U 47.3	U 631
Sauk-up	U 20.5	U 63.7	U 15.9	U 15.9	U 9.65	U 47.8	U 637
Field Blank 1	U 3.97	U 63.5	U 15.9	U 15.9	U 7.93	U 47.6	U 63.5
Field Blank 2	U 4.32	U 207	U 17.3	U 17.3	NQ	U 51.8	U 308