Endocrine Active Compound Monitoring in Minnesota Lakes, 2009-2011: Lake Habitat and Land Use

Minnesota Pollution Control Agency

February 2013

Legislative Charge

This study was authorized in the FY 2010-2011 biennial budget by the Minnesota Legislature and the Governor, enacted in 2009 Session Laws Chapter 172, Article 2 Clean Water Fund, Section 4(k): "$7,500,000 the first year and $7,500,000 the second year are for completion of 20 percent of the needed statewide assessments of surface water quality and trends. Of this amount, $175,000 the first year and $200,000 the second year are for monitoring and analyzing endocrine disruptors in surface waters."

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Abbreviations

APE	Alkylphenol ethoxylate – detergents that degrade to endocrine active alkylphenols such as nonylphenol or octylphenol
BCF	Body condition factor – the ratio of body length to body weight
EAC	Endocrine active chemical
GSI	Gonadosomatic index – the ratio of the weight of gonads to body weight
HSI	Hepatosomatic index – the ratio of the weight of the liver to body weight
LAS	Linear alkylbenzene sulfonate – detergents that are not endocrine active
PPCPs	Pharmaceuticals and personal care products
VTG	Vitellogenin – a protein normally produced in female fish associated with egg development
VNP	Voyageur's National Park

Units of Concentration

ppm, mg/L	Parts per million
ppb, µg/L	Parts per billion
ppt, ng/L	Parts per trillion

Summary

Several recent studies of Minnesota lakes and rivers have shown that endocrine active chemicals (EACs) and pharmaceuticals are surprisingly widespread in the state's surface water and sediment. Fish that were analyzed in these studies showed evidence of exposure to EACs.

This study, following the Statewide Endocrine Disrupting Compound Monitoring Study of Minnesota Lakes in 2008, was done to understand how endocrine disruption in fish might vary spatially within a single lake, and how lake "microhabitats" are influenced by localized sources of EACs to the surface water. In addition to studying fish from these habitats, surface water samples and samples of groundwater flowing into the lake were collected for chemical analysis from each microhabitat.

The results of this intensive investigation of a single lake suggest that fish undergo morphological (such as body size and weight), physiological (such as metabolic or enzyme functions), and genetic changes when they are exposed to habitats influenced by septic fields, urban runoff, and agricultural sources of contaminants. Analysis of groundwater entering the lake and lake surface water revealed the presence of a variety of endocrine active alkylphenols, hormones, and other chemicals.

Water samples were also taken from lakes in very remote locations. Results of this sampling revealed that although remote lakes in Voyageur's National Park (VNP) and Itasca State Park did contain some contaminants, they appear to contain fewer chemicals than were discovered in lakes in more developed watersheds.

Introduction and Rationale for Study

In 2008, the Minnesota Pollution Control Agency (MPCA) collaborated with the U.S. Geological Survey (USGS) and St. Cloud State University (SCSU) to study pharmaceuticals and personal care products (PPCPs) and other contaminants of emerging concern in 12 Minnesota lakes and four streams (Figure 1). The study included the analysis of endocrine disruption in fish in those waters (Ferrey et al. 2008; Writer et al. 2010). That study, together with previous investigations (Lee et al. 2010), demonstrated that several PPCPs and EACs are widespread in Minnesota's lakes and rivers, sometimes occurring in areas without obvious sources of contamination. It also showed that fish from these lakes and streams – some considered relatively pristine – were exhibiting signs of endocrine disruption.

While limited sampling of lakes and rivers has shown that these contaminants are present in the aquatic environment, several questions remain unanswered. First, it is unclear if EACs and PPCPs are present in lakes that are not exposed to obvious sources of contamination (such as septic fields, wastewater treatment plants, or agricultural areas). Second, the variability in contaminant concentration across a lake is not known. It is unclear if fish populations within a lake are exposed to contaminants uniformly or are more heavily exposed to contaminants in close proximity to identifiable sources, such as residential septic systems. Finally, it is not known if fish populations exhibit a variation in endocrine disrupting effects within a given lake depending on location.

In this study, four microhabitats in one lake (Sullivan Lake in Wright County) were investigated for:

a) The presence and concentration of PPCPs and EACs in the surface water.
b) The presence of detergents in the groundwater entering the lake at these four locations.
c) Temporal variability in PPCP and EAC concentrations in lake surface water.
d) Evidence of endocrine disruption in native fish at these locations.
e) Evidence of endocrine disruption in fish that were caged for a limited time at each microhabitat.

In addition, four remote lakes in northern Minnesota – Ryan and Beast Lakes in Voyageur's National Park, and Bohall and Elk Lakes in Itasca State Park (Figure 1) – were sampled and analyzed for EACs and other chemicals of emerging concern to determine what contaminants are present in pristine and undeveloped lakes that lack nearby sources of these chemicals.

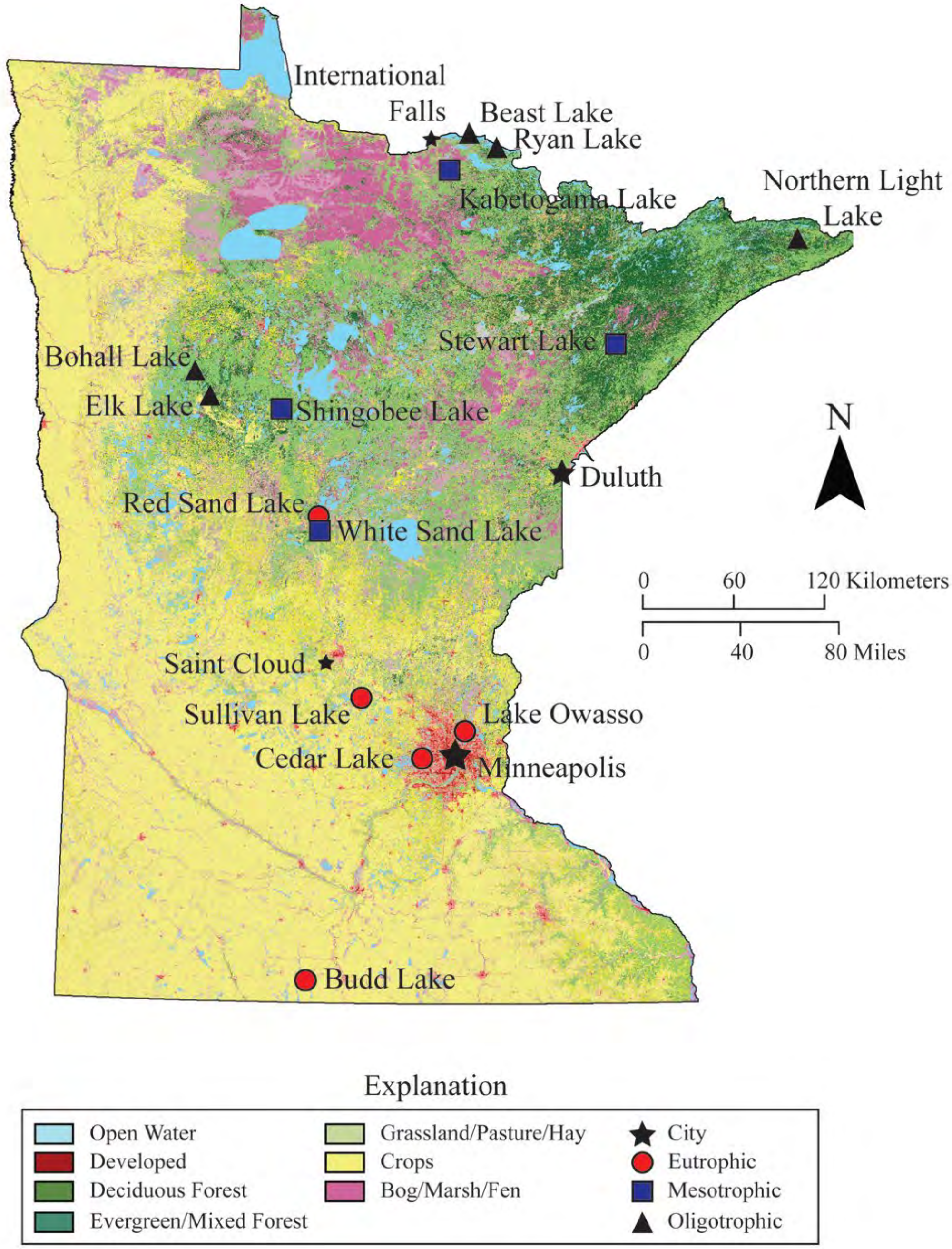

Figure 1. Lakes sampled for the 2008 Statewide Endocrine Disrupting Compound Study and this study.

Approach

Focused lake study. Sullivan Lake in Wright County, Minnesota, was selected for the focused lake study because the lake is influenced by a variety of land use: residential septic systems, runoff from a boat landing (road runoff), and agricultural activity (Figure 2). Aquatic microhabitats near these potential sources of contamination were studied to determine how particular land use affects nearby aquatic environments.

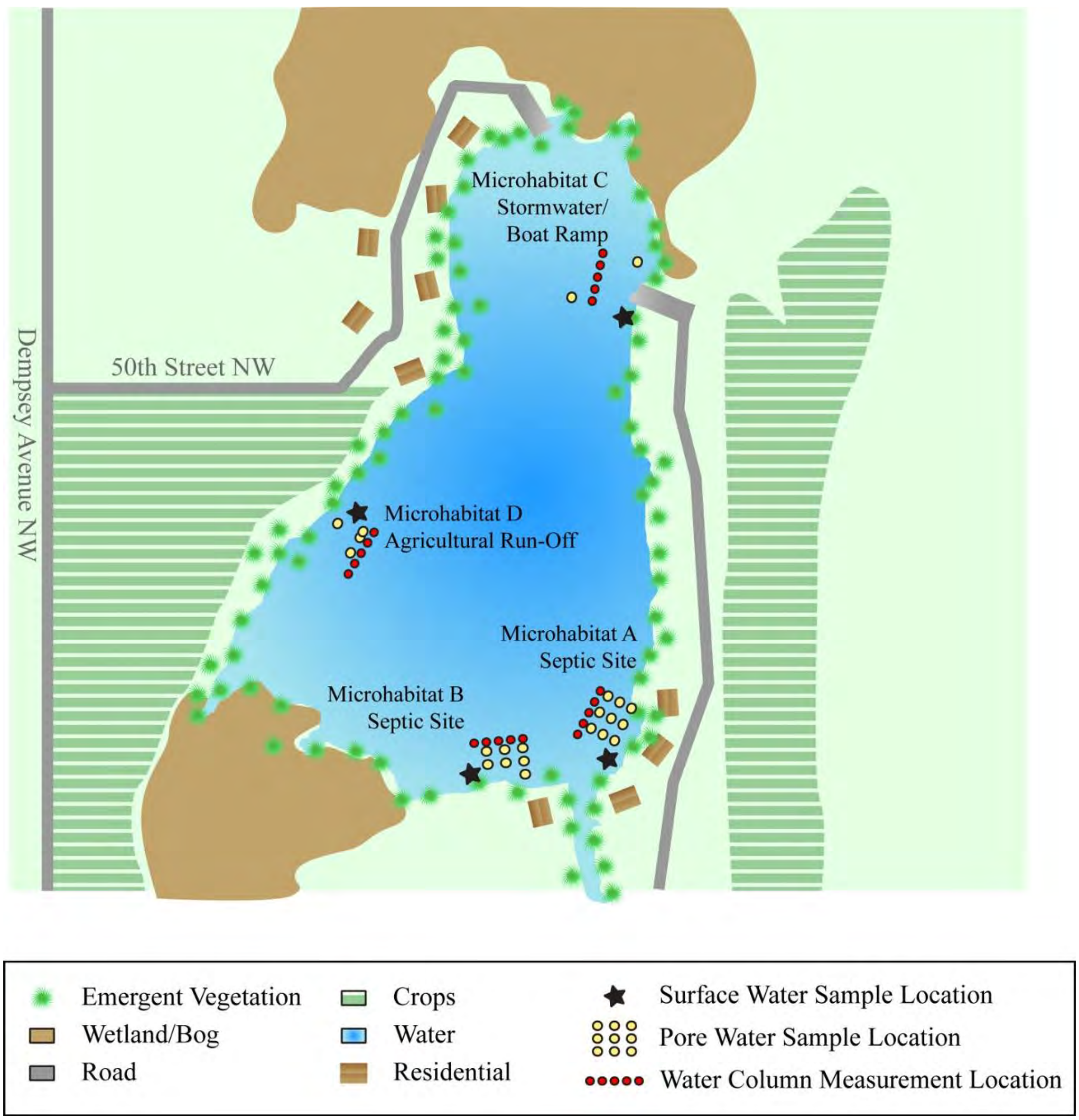

Figure 2. Locations selected for sampling on Sullivan Lake corresponding to land use.

Sullivan Lake surface water and groundwater sampling. Locations of groundwater discharge to Sullivan Lake were identified using temperature gradients and inorganic chemical measurements (e.g., nitrate) that indicate where groundwater flows upward through near-shore lake sediment and into the lake. Two areas of groundwater discharge to the lake near residences were chosen as locations where residential septic systems may be impacting the lake habitat (Figure 2). Groundwater was sampled as it passed upward through the lake sediment (Figure 3) and was analyzed for linear alkylbenzene sulfonates (LAS) and alkylphenol ethoxylates (APEs).

Surface water was sampled at the four lake locations shown in Figure 2 – areas influenced by road runoff, agricultural activities, and two locations influenced by residential septic drainfields – and analyzed for a broad suite of contaminants. Note that not all the chemicals analyzed for this study have endocrine active properties. For a complete list of analytes, see Appendix A.

Fish studies. Twenty native sunfish from each microhabitat location were collected during the spawning season and studied for evidence of endocrine disruption, including vitellogenin (VTG) production in male fish, morphological/body condition changes, and tissue analysis of gonads and liver. These analyses were also done on laboratory-raised sunfish that were caged at each microhabitat for 21 days. Results were compared to baseline or control fish that were maintained in the laboratory during the course of the experiment. Fathead minnow larvae in the laboratory were exposed to sediment pore-water that was collected from the two locations of groundwater upwelling into the lake near residential septic drainfields. After a 21-day exposure to this groundwater, the larvae were tested for behavioral changes using a predator escape performance assay in the laboratory.

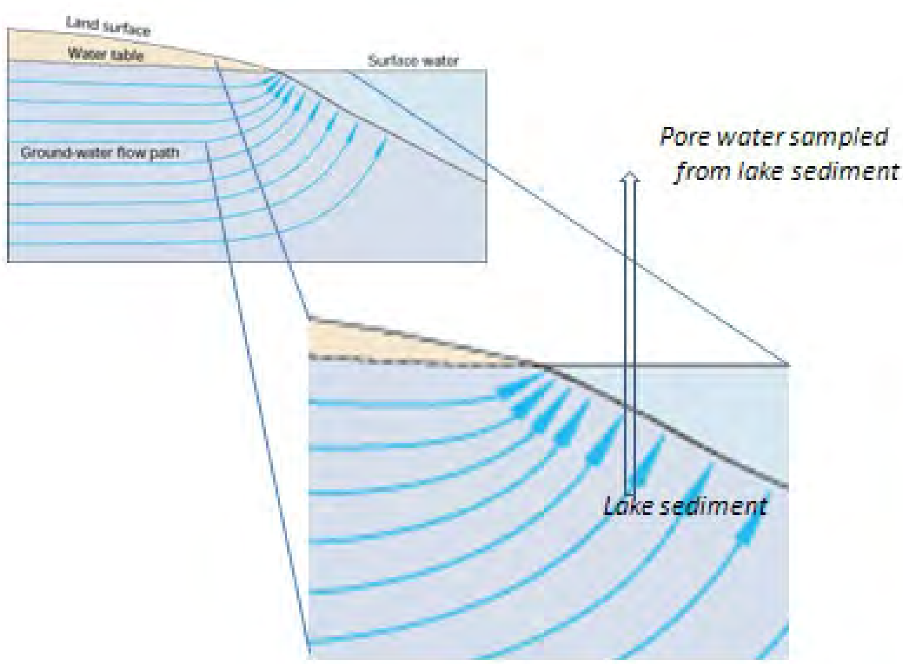

Figure 3. Diagram of groundwater flow to surface water. Groundwater samples can be taken as it flows upward through the lake sediment by inserting a narrow, hollow tube into the lake sediment and withdrawing water from the tube with a syringe *(Diagram modified from USGS*).

After exposure to the sediment pore water from the two septic-influenced sites (Sites A and B), the road runoff location (Site C), and the agriculture influenced location (Site D), fathead minnow larvae were analyzed for alterations in the expression of two genes – the estrogen receptor (ER) gene and the steroidogenic acute regulatory protein (StAR) gene – to measure the genetic effects of exposure to contaminants.

Remote lake sampling. Two remote lakes in Voyageur's National Park (VNP) – Ryan Lake and Beast Lake – and two lakes in Itasca State Park – Bohall Lake and Elk Lake (Figure 1) – were sampled in October 2010. The surface water was analyzed for a variety of hormones and industrial chemicals, several of which are EACs (for a complete list of the analytes, see Appendix A). Ryan, Beast, and Bohall Lakes are remote and receive very little human impact. Elk Lake, situated near Lake Itasca, is undeveloped, but is located on an access road and is exposed to light recreational traffic. Elk Lake was included in the previous sampling of 12 lakes in the Statewide Endocrine Disrupting Compound Study of 2008.

Results

Effect of land use on water quality

Sullivan Lake surface water analysis

Summer 2010

- EDTA (a metal binding agent and food preservative), nitrilotriacetic acid, nonylphenolmonoethoxycarboxylic acid (a detergent), di-butyl benzoquinone (a breakdown product of BHT), DEET (insect repellent), and methylphenol were detected at every site (Tables 1, 3).
- Bisphenol A (the chemical used to make polycarbonate plastic and a known EAC) and nonylphenol (a breakdown product of APE detergents and a known EAC) were detected in surface water at both septic-influenced sites A and B (Table 3).
- Caffeine was detected at site A (septic influenced), which also had the highest levels of DEET and nonylphenol (Table 3).
- Cholesterol, coprostanol (a metabolite of cholesterol), and estrone (a steroid hormone) were detected at all sites. The hormone androstenedione was detected at all sites except site A (Table 5).
- The pesticide 2,4-D and the pesticide metabolites acetochlor ESA, acetochlor OXA, alachlor ESA, metolachlor ESA, and hydroxyatrazine were detected at low parts per trillion (Table 15).

Fall 2010

- Cholesterol and coprostanol were detected at all sites. Estrone was detected at site D (Table 6).
- EDTA was detected at every location (Table 2). Caffeine and bisphenol A were detected at the two septic influenced locations A and B, while nonylphenol was detected at septic location A. Dibutylbenzoquinone was detected at every site, and DEET were detected at sites A, B, and D (Table 4).

Sullivan Lake microhabitat sediment pore water (ground water) analysis

Microhabitat A (septic–influenced location, Table 7)

- LAS concentrations in pore water samples ranged from <0.02 to 0.92 mg/L.
- There was one detection of APE (0.14 mg/L).

Microhabitat B (septic-influenced location, Table 9)

- LAS concentrations ranged from <0.02 to 0.12 mg/L.
- APEs were detected up to 0.11 mg/L.

Microhabitat C (road-runoff location, Table 11)

- LAS was detected at 0.03 mg/L.
- APE was detected at 0.11 mg/L.

Microhabitat D (agricultural-influenced location, Table 13)

- LAS was detected at concentrations ranging from 0.02 to 0.11 mg/L.

Remote lake surface water (Elk, Beast, Bohall, and Ryan Lakes; Appendix C Tables 1-4)

- EDTA was detected in Elk Lake (0.53 µg/L), Bohall Lake (0.06 µg/L), and in Ryan Lake (0.40 µg/L).
- Di-butyl benzoquinone was detected in Elk Lake (50 ng/L), Beast Lake (40 ng/L), and Bohall Lake (20 ng/L).
- Methylphenol was found (10 ng/L) in Beast and Ryan Lakes.
- Cholesterol was detected in all of the remote lakes sampled. Coprostanol (a biological metabolite of cholesterol) was detected in every lake except for Ryan Lake. These chemicals can occur due to the presence of either humans or wildlife.
- The hormone estrone was detected in Elk Lake (0.1 ng/L), Beast Lake (0.1 ng/L), Ryan Lake (0.8 ng/L), and Bohall Lake (0.1 ng/L). Estrone is a metabolite of 17β-estradiol, which can be due to the presence of either wildlife or humans.

Sullivan Lake microhabitat fish studies

- Caged male sunfish at one septic-influenced location produced more VTG than those caged at the urban/road runoff site. (VTG production in male fish is evidence of endocrine disruption, since it is a protein required for egg development in female fish and is not normally found in males.)
- No differences were found in the hepatosomatic index (HSI) between caged and native fish at any of the locations. (HSI is a ratio of liver to body weight and is an indicator of physiological stress.) However, the livers from sunfish that were caged at one of the septic-influenced locations showed more signs of stress (vacuoles in the liver tissue) than the livers from native fish.
- Native sunfish had a significantly different gonadosomatic index (GSI) between the two septic locations. Change in GSI is an indication of endocrine disruption.
- Reproductive organs from wild male and female fish from one of the septic locations showed decreased maturity compared to those of the baseline/control fish.
- There were no measurable effects on predator escape responses in fathead minnow larvae that were exposed to sediment pore water that was collected from any of the locations.
- Minnow larvae that were exposed to sediment pore water from the septic-influenced locations and the road-runoff locations showed increases in the activity of ER and StAR genes.

Discussion

In 2008, the Minnesota Legislature funded the Statewide Endocrine Disrupting Compound Study of Minnesota lakes (Writer et al. 2010), which revealed the widespread presence of PPCPs and EACs in lakes that often lacked obvious sources of contaminants. That study (of 12 lakes and four rivers) also showed that endocrine disruption in fish is a common phenomenon in Minnesota's lakes and streams.

The results of this study indicate that land use is important in determining the type and concentration of contaminant in the surface water and incoming groundwater. In Sullivan Lake, surface water from every location was found to contain nonylphenol ethoxylate, nonylphenol, EDTA, DEET, di-butyl benzoquinone, cholesterol, and estrone. The highest concentrations of DEET in surface water were found at one of the septic-influenced locations.

Groundwater entering the lake from the residential/septic influenced areas contained LAS up to 1 mg/L as well as nonylphenol and octylphenol, indicating that groundwater associated with a particular land use can be a significant source of EACs to lake surface water. By contrast, lakes without surrounding development or human impact appear to contain very few of the contaminants that are present in lakes in a more developed watershed. Of the chemicals that are solely attributable to human impact, the remote Ryan Lake in VNP contained only the food preservative EDTA, while Beast, Bohall, and Elk Lakes contained di-butyl benzoquinone. Although these lakes also contained cholesterol, coprostanol, and estrone, the presence of these chemicals may be attributable to wildlife.

Not all the chemicals analyzed for this study are endocrine active. However, several of the chemicals detected in this study are known to elicit hormone-like effects in fish. Nonylphenol, for example, is an established EAC that is weakly estrogenic with effects on the behavior of fish at concentrations in the parts per trillion range(Schoenfuss et al. 2008). Surface water standards have not been established for endocrine active compounds. Regardless, the chemicals detected in the surface water in this study are similar in concentration to what has been found in previous investigations. The results reported here are consistent with earlier work that suggests EACs and other chemicals of emerging concern are widespread in our aquatic environment.

This is the first study of the influence of land use on "microhabitats" in an individual lake. The results suggest that endocrine disruption in fish varies spatially within a lake, and is associated with the proximity to sources of contaminants. Subtle but clear differences in exposure effects between caged and resident sunfish, the variation in estrogenic responses in fish, and differences in body condition factors all suggest that fish are exposed to different stressors that can be location specific. For example, decreased BCF was the only adverse effect that indicated exposure to EACs in the habitat associated with agricultural land use, while no adverse effects were found on sunfish in the habitat exposed to road runoff. Fish from both septic influenced sites, however, showed more evidence of exposure to estrogenic chemicals, and changes in reproductive organs were observed in fish from these habitats. In addition to a decreased BCF, male sunfish caged at septic site A had higher levels of VTG, while fish caged at septic site B were found to have an increase in HSI and decreased sexual maturity.

Fathead minnow larvae exposed to groundwater that contained estrogenic contaminants did not exhibit behavioral changes in the predator avoidance tests that have been previously observed following exposure to estrogenic compounds. However, alterations of these fish on a genetic level (alterations in the expression of the ER and StAR genes) indicate that chemicals were exerting estrogen-like effects at the locations where this groundwater was entering the lake.

Studies in Minnesota and elsewhere have demonstrated that exposure to EACs in surface water can elicit changes in patterns of gene expression (Lee et al. 2011), the unnatural production of VTG in male fish of several species (Writer et al. 2010), and the collapse of fish populations and disruption of aquatic ecosystems (Kidd et al. 2007).This study demonstrates that effects of EACs apparently extend across different life stages (larvae and adult fish) as well as across species (sunfish and fathead minnows). Moreover, the study shows that the physiology of individual organisms can also be affected, including alterations of:

- gene expression (such as the ER and StAR gene expression changes)
- protein expression (such as vitellogenin production in male fish)
- tissues (the observed changes in liver structure)
- reproductive condition (observed changes in gonad tissues in wild fish)

These results are consistent with those of previous studies in suggesting that EACs released into our aquatic environments may be having adverse effects on fish and wildlife, and heighten concern over how fish populations – and other wildlife – may be affected over the long term.

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Appendix A

Chemicals analyzed in this study

Hormone	Description
11-ketotestosterone	The oxidized form of testosterone, the primary male sex hormone in fish
17α-estradiol	A form of estradiol that has no estrogenic properties used for the treatment of hair loss
17β-estradiol	One of three naturally occurring estrogens; a female hormone
Androstenedione	Precursor to testosterone and estrogen, the male and female sex hormones
Androsterone	Steroidal hormone with weak androgenic activity
Equilenin	An estrogen used in hormone replacement therapy
Equilin	An estrogen used in hormone replacement therapy
Estriol	One of three naturally occurring estrogens; a female hormone
Estrone	One of three naturally occurring estrogens; a female hormone
Ethinylestradiol	Synthetic oral contraceptive
Mestranol	An estrogen used in oral contraceptives, converted to ethinylestradiol
Progesterone	A female steroid hormone
Testosterone	A male sex hormone and anabolic steroid

Organic Wastewater Compound	Application/description
1,3-dichlorobenzene	Deodorizer
1,4-dichlorobenzene	Pesticide; deodorizer
2,6-di-tert-butyl-1,4-benzoquinone (di-butyl benzoquinone)	Metabolite of 2,6-di-tert-butylphenol and BHT (an antioxidant food additive)
2,6-di-tert-butyl-4-methylphenol (butylated hydroxytoluene)	BHT. Antioxidant food additive, also used in fuels, cosmetics, pharmaceuticals, and embalming fluid.
2,6-di-tert-butylphenol	A UV stabilizer and an antioxidant for hydrocarbon-based products. Prevents gumming in aviation fuel.
3β-coprostanol	Metabolite of cholesterol formed in the gut of animals; indicator of fecal matter in the environment
4-ethylphenol	A naturally occurring degradation product of lignocellulose
4-methylphenol (cresol)	Wood preservative; can also be a naturally occurring chemical in the environment
4-*tert*-octylphenol	Nonionic surfactant; an alkylphenol with endocrine active properties
4-n-octylphenol	An alkylphenol with endocrine active properties; a breakdown product of octylphenol ethoxylate
Organic Wastewater Compound (continued)	**Application/description**

4-*tert*-octylphenol monoethoxylate; 4-OP1EO	Nonionic detergent
4-*tert*-octylphenol diethoxylate; 4-OP2EO	Nonionic detergent
4-*tert*-octylphenol tetraethoxylate; 4-OP4EO	Nonionic detergent
4-*tert*-octylphenol pentaethoxylate; 4-OP5EO	Nonionic detergent
4-*tert*-pentylphenol	An alkylphenol with estrogenic properties
4-nonylphenol	An alkylphenol with estrogenic properties; breakdown product of nonylphenol ethoxylate detergent
4-nonylphenol monoethoxylate; 4-NP1EO	Nonionic detergent
4-nonylphenol diethoxylate; 4-NP2EO	Nonionic detergent
4-propylphenol	An alkylphenol used in flavor and fragrance chemistry
4-tert-butylphenol	An alkylphenol precursor to other chemicals such as antioxidants and UV protection chemicals.
4-tert-pentylphenol	An alkylphenol used in the formation of antioxidants and UV protection chemicals
5-methyl-1H-benzotriazole	Anticorrosive; rust inhibitor
AHTN (Acetylhexamethyltetrahydronaphthalene)	Fragrance chemical
APECs (Alkylphenol ethoxycarboxylates)	Synthetic detergents that can break down to endocrine active alkylphenols
Bisphenol A	Monomer used to synthesize polycarbonate plastic
DEET (N,N-diethyl-*meta*-toluamide)	Pesticide (insect repellant)
EDTA (Ethylenediaminetetraacetic acid)	Chemical that binds metal ions in solution; widespread industrial and commercial application
HHCB (Hexahydrohexamethylcyclopentabenzopyran)	Fragrance chemical
NTA (Nitrilotriacetic acid)	Chemical that binds metal ions in solution; widespread industrial and commercial application
Triclosan	A widely used antimicrobial chemical

Pharmaceutical	Description
Caffeine	Stimulant; an indicator of wastewater

Appendix B

Sullivan Lake

Surface water and sediment pore water data

Table 1. Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on July 8, 2010.

Table 2. Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010.

Table 3. Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn. on July 8, 2010.

Table 4. Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010.

Table 5. Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on July 8, 2010.

Table 6. Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010.

Table 7. Site characterization of Sullivan Lake microhabitat A (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010.

Table 8. Water column water-quality measurements for Sullivan Lake microhabitat A (residential/septic influence) collected from 1:30 to 3:00 PM on July 21, 2010.

Table 9. Site characterization of Sullivan Lake microhabitat B (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010.

Table 10. Water column water-quality measurements for Sullivan Lake microhabitat B (residential/septic influence) collected 10:30 AM to 2:00 PM on July 21, 2010.

Table 11. Site characterization of Sullivan Lake microhabitat C (stormwater/boat-ramp influence) and water-quality values for pore-water samples collected July 8, 2010.

Table 12. Water column water-quality measurements for Sullivan Lake microhabitat C (stormwater/boat ramp influence) collected 12:00 to 1:30 PM on July 21, 2010.

Table 13. Site characterization of Sullivan Lake microhabitat D (agricultural influence) and water-quality values for pore-water samples collected July 9, 2010.

Table 14. Water column water-quality measurements for Sullivan Lake microhabitat D (agricultural influence) collected 9:00 to 10:30 AM on July 21, 2010.

Table 15. Pesticide data for Sullivan Lake.

Table 1. **Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minnesota, on July 8, 2010. [Microhabitats A and B, residential/septic influenced, microhabitat C, stormwater/boat-ramp influenced, imicrohabitat D, agriculture influenced, µg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NM, no measurement]**

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS Percent	Site B µg/L	Site B-Dup µg/L	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Blank µg/L	Blnk µg/L	Blank µg/L
Ethylenediaminetetraacetic acid	0.2	0.2	63	0.2	0.2	0.2	0.2	68.6	0.6	0.6	<0.05	<0.05	<0.05
Nitrilotriacetic acid	.1	.1	101	.1	.1	.1	.1	98.4	.1	.1	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	.6	.4	90	.4	.4	.4	.5	90.9	.4	.4	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	.5	.1	95	< .1	< .1	< .1	< .1	90.0	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	80	< .1	< .1	< .1	< .1	78.1	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	84	< .1	< .1	< .1	< .1	80.0	< .1	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)													
4-*normal*-Nonylphenoldiethoxycarboxylic acid	69	66	95	68	72	68	87	85	79	75	98	NM	75

Table 2. **Summary of acidic organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than; Dup, duplicate sample]**

Compound	Site A µg/L	Site A Dup µg/L	Site B µg/L	Site C µg/L	Site D µg/L
Ethylenediaminetetraacetic acid	0.3	0.3	0.2	0.2	0.3
Nitrilotriacetic acid	.1	.1	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	.2	.1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)					
4-*normal*-Nonylphenoldiethoxycarboxylic acid	64	46	55	70	52

Table 3. Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn. on July 8, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NM, not measured]

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
Acetylhexamethyltetrahydronaphthalene	<0.01	<0.01	111	<0.01	<0.01	114	<0.01	<0.01	131	<0.01	<0.01	124
Bisphenol A	.03	.02	67	.05	.01	68	< .01	< .01	85	< .01	< .01	84
2[3]-*tert*-Butyl-4-methylphenol	< .01	< .01	118	< .01	< .01	92	< .01	< .01	131	< .01	< .01	123
4-*tert*-butylphenol	< .01	< .01	111	< .01	< .01	116	< .01	< .01	129	< .01	< .01	129
Caffeine	.01	.02	31	< .01	< .01	31	< .01	< .01	30	< .01	< .01	41
2,6-Di-*tert*-butyl-1,4-benzoquinone	.03	.03	NM	.04	.03	NM	.04	.03	NM	.03	.03	NM
2,6-Di-*tert*-butyl-4-methylphenol	.04	.02	NM	.02	.01	NM	.03	.04	NM	.02	< .01	NM
2,6-Di-*tert*-butylphenol	< .01	< .01	136	< .01	< .01	98	< .01	< .01	146	< .01	< .01	143
1,2-Dichlorobenzene	< .01	< .01	99	< .01	< .01	98	< .01	< .01	88	< .01	< .01	111
1,3-Dichlorobenzene	< .01	< .01	98	< .01	< .01	96	< .01	< .01	88	< .01	< .01	110
1,4-Dichlorobenzene	< .01	< .01	98	< .01	< .01	96	< .01	< .01	87	< .01	< .01	110
N,*N*-Diethyl-*meta*-toluamide	.15	.14	107	.13	.13	118	.07	.08	117	.06	.06	125
4-Ethylphenol	< .01	< .01	73	< .01	< .01	69	.01	< .01	68	< .01	< .01	84
Hexahydrohexamethylcyclopentabenzopyran	< .01	< .01	107	< .01	< .01	107	< .01	< .01	124	< .01	< .01	118
5-Methyl-1H-Benzotriazole	< .01	< .01	<10	< .01	< .01	<10	< .01	< .01	<10	< .01	< .01	<10
4-Methylphenol	.02	.03	43	.03	.03	39	.03	.04	41	.03	.03	54
4-Nonylphenol	.09	.09	122	.05	.05	131	< .05	< .05	163	< .05	< .05	159
4-Nonylphenolmonoethoxylate	.28	.09	134	.09	.08	137	< .05	< .05	161	< .05	< .05	152
4-Nonylphenoldiethoxylate	< .05	< .05	110	< .05	< .05	117	< .05	< .05	137	< .05	< .05	129
4-*normal*-Octylphenol	< .01	< .01	12	< .01	< .01	12	< .01	< .01	15	< .01	< .01	14
4-*tert*-Octylphenol	.04	.02	137	.02	.01	138	.02	.01	162	< .01	< .01	157
4-*tert*-Octylphenolmonoethoxylate	.02	< .01	113	< .01	< .01	115	< .01	< .01	133	< .01	< .01	128
4-*tert*-Octylphenoldiethoxylate	< .01	< .01	109	< .01	< .01	108	< .01	< .01	122	< .01	< .01	119
4-*tert*-Octylphenoltriethoxylate	< .01	< .01	95	< .01	< .01	83	< .01	< .01	91	< .01	< .01	91
4-*tert*-Octylphenoltetraethoxylate	< .01	< .01	70	< .01	< .01	72	< .01	< .01	73	< .01	< .01	101
4-*tert*-Octylphenolpentaethoxylate	< .01	< .01	47	< .01	< .01	34	< .01	< .01	45	< .01	< .01	55
4-*tert*-Pentylphenol	< .01	< .01	122	< .01	< .01	128	< .01	< .01	147	< .01	< .01	142

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
4-Propylphenol	< .01	< .01	115	< .01	< .01	114	< .01	< .01	122	< .01	< .01	128
Triclosan	< .01	< .01	100	< .01	< .01	87	< .01	< .01	100	< .01	< .01	102
Surrogate Recovery (percent)												
d6-Bisphenol A	72	78		78	77		96	99		85	81	
d21-2,6-Di-tert-butyl-4-methylphenol	49	71		59	61		60	89		65	70	
4-normal-Nonylphenol	52	57		53	52		69	76		55	53	
4-normal-Nonylphenolmonoethoxylate	47	52		52	51		74	77		56	53	
4-normal-Nonylphenoldiethoxylate	29	39		41	40		59	63		45	40	

Table 4. Summary of neutral organic compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010. [Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than; Dup, duplicate sample]

Compound	**Site A µg/L**	**Site A- µg/L**	**Site B µg/L**	**Site B- µg/L**	**Site C µg/L**	**Site D µg/L**	**Site D- µg/L**
Acetylhexamethyltetrahydronaphthalene	0.02	0.01	0.01	0.02	<0.01	<0.01	<0.01
Bisphenol A	.01	.01	.15	.02	< .01	< .01	< .01
2[3]-*tert*-Butyl-4-methylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Butylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Caffeine	.02	.05	.04	.01	< .01	< .01	< .01
2,6-Di-*tert*-butyl-1,4-benzoquinone	.06	.09	.04	.06	.04	< .01	.04
2,6-Di-*tert*-butyl-4-methylphenol	.01	.01	< .01	.01	.01	.02	.02
2,6-Di-*tert*-butylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
1,2-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
1,3-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
1,4-Dichlorobenzene	< .01	< .01	< .01	< .01	< .01	< .01	< .01
N,*N*-Diethyl-*meta*-toluamide	.09	.08	.09	.07	< .01	< .01	.02
4-Ethylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Hexahydrohexamethylcyclopentabenzopyran	.01	.02	< .01	.02	< .01	< .01	< .01
5-Methyl-1H-benzotriazole	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-Methylphenol	< .01	< .01	.02	.01	< .01	< .01	< .01
4-Nonylphenol	.06	.05	< .05	< .05	< .05	< .05	< .05
4-Nonylphenolmonoethoxylate	.11	.08	< .05	< .05	< .05	< .05	< .05
4-Nonylphenoldiethoxylate	< .05	< .05	< .05	< .05	< .05	< .05	< .05
4-*normal*-Octylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Octylphenol	.09	.06	.05	.03	< .01	< .01	< .01
4-*tert*-Octylphenolmonoethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoldiethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoltriethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoltetraethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Octylphenolpentaethoxylate	< .01	< .01	< .01	< .01	< .01	< .01	< .01
4-*tert*-Pentylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01

Compound	**Site A µg/L**	**Site A- µg/L**	**Site B µg/L**	**Site B- µg/L**	**Site C µg/L**	**Site D µg/L**	**Site D- µg/L**
4-Propylphenol	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Triclosan	< .01	< .01	< .01	< .01	< .01	< .01	< .01
Surrogate Recovery (percent)							
d6-Bisphenol A	19	40	44	40	39	40	41
d21-2,6-Di-*tert*-butyl-4-methylphenol	20	31	21	38	57	56	49
4-*normal*-Nonylphenol	26	47	37	45	36	43	43
4-*normal*-Nonylphenolmonoethoxylate	52	53	38	43	35	39	39
4-*normal*-Nonylphenoldiethoxylate	44	53	47	53	43	49	49

Table 5. Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on July 8, 2010.
[Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than; Dup, duplicate sample; MS, matrix spike recovery in percent; NQ, not quantifiable]

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
4-Androstene-3,17-dione	<0.0001	<0.0001	138	0.0009	0.0005	118	0.0005	0.0005	134	0.0014	0.0012	133
cis-Androsterone	< .0001	< .0001	150	< .0001	< .0001	139	< .0001	< .0001	130	< .0001	< .0001	150
Cholesterol	5.7	NQ	348	7.6	4.4	266	30	11	-664	23	NQ	327
Coprostanol	.029	.065	104	.042	.025	103	.053	.090	86	.20	.97	89
Diethylstilbestrol	< .0001	< .0001	97	< .0001	< .0001	66	< .0001	< .0001	97	< .0001	< .0001	97
Equilenin	< .0001	< .0001	105	< .0001	< .0001	86	< .0001	< .0001	101	< .0001	< .0001	107
Equilin	< .0001	< .0001	98	< .0001	< .0001	78	< .0001	< .0001	97	< .0001	< .0001	76
17α-Estradiol	< .0001	< .0001	117	< .0001	< .0001	132	< .0001	< .0001	112	< .0001	< .0001	119
17β-Estradiol	< .0001	< .0001	107	< .0001	< .0001	106	< .0001	< .0001	108	< .0001	< .0001	111
Estriol	< .0001	< .0001	115	< .0001	< .0001	73	< .0001	< .0001	62	< .0001	< .0001	235
Estrone	.0007	.0008	120	.0010	.0009	111	.0011	.0009	110	.0008	.0010	107
17α-Ethynylestradiol	< .0001	< .0001	110	< .0001	< .0001	100	< .0001	< .0001	103	< .0001	< .0001	104
Mestranol	< .0001	< .0001	107	< .0001	< .0001	99	< .0001	< .0001	106	< .0001	< .0001	106
Norethindrone	< .0001	< .0001	106	< .0001	< .0001	99	< .0001	< .0001	104	< .0001	< .0001	106
Progesterone	< .0001	< .0001	15	< .0001	< .0001	26	< .0001	< .0001	27	< .0001	< .0001	22
Testosterone	< .0001	< .0001	124	< .0001	< .0001	114	< .0001	< .0001	121	< .0001	< .0001	124
dihydro-Testosterone	< .0001	< .0001	130	< .0001	< .0001	109	< .0001	< .0001	123	< .0001	< .0001	113
epi-Testosterone	< .0001	< .0001	128	< .0001	< .0001	121	< .0001	< .0001	120	< .0001	< .0001	132
11-*keto*-Testosterone	< .0001	< .0001	81	< .0001	< .0001	69	< .0001	< .0001	95	< .0001	< .0001	91
Surrogate Recovery (percent)												
d7-Cholesterol	47	<10		37	41		19	10		40	<10	
d12-Chrysene	105	106		183	115		115	119		105	104	
d8-Diethylstibestrol	59	<10		47	18		42	18		34	<10	
$^{13}C_6$-Estradiol	80	18		56	33		36	28		28	<10	
d2-*epi*-Estriol	42	18		46	49		39	19		16	18	
$^{13}C_6$-Estrone	96	30		75	97		71	65		97	31	
d4-Ethynylestradiol	82	26		62	71		47	49		70	25	
d3-Medroxyprogesterone	19	12		45	33		44	35		28	21	

Compound	Site A µg/L	Site A-Dup µg/L	Site A-MS percent	Site B µg/L	Site B-Dup µg/L	Site B-MS percent	Site C µg/L	Site C-Dup µg/L	Site C-MS percent	Site D µg/L	Site D-Dup µg/L	Site D-MS percent
d4-Mestranol	83	30		63	70		48	48		71	28	
d3-Nandrolone	63	24		54	61		54	45		30	17	

Table 6. Summary of steroid and steroidal hormone compound data for water samples collected from four microhabitat sites in Sullivan Lake, Minn., on October 12, 2010.
[Microhabitats A and B, residential/septic influenced; microhabitat C, stormwater/boat-ramp influenced; microhabitat D, agriculture influenced; µg/L, microgram per liter; <, less than]

Compound	**Site A µg/L**	**Site B µg/L**	**Site C µg/L**	**Site D µg/L**
4-Androstene-3,17-dione	<0.0001	<0.0001	<0.0001	<0.0001
cis-Androsterone	< .0001	< .0001	< .0001	< .0001
Cholesterol	3.3	3.4	2.8	3.2
Coprostanol	.042	.017	.017	.030
Diethylstilbestrol	< .0001	< .0001	< .0001	< .0001
Equilenin	< .0001	< .0001	< .0001	< .0001
Equilin	< .0001	< .0001	< .0001	< .0001
17α-Estradiol	< .0001	< .0001	< .0001	< .0001
17β-Estradiol	< .0001	< .0001	< .0001	< .0001
Estriol	< .0001	< .0001	< .0001	< .0001
Estrone	< .0001	< .0001	< .0001	.0006
17α-Ethynylestradiol	< .0001	< .0001	< .0001	< .0001
Mestranol	< .0001	< .0001	< .0001	< .0001
Norethindrone	< .0001	< .0001	< .0001	< .0001
Progesterone	< .0001	< .0001	< .0001	< .0001
Testosterone	< .0001	< .0001	< .0001	< .0001
dihydro-Testosterone	< .0001	< .0001	< .0001	< .0001
epi-Testosterone	< .0001	< .0001	< .0001	< .0001
11-*keto*-Testosterone	< .0001	< .0001	< .0001	< .0001
Surrogate Recovery (percent)				
d7-Cholesterol	39	46	53	15
d12-Chrysene	82	94	94	104
d8-Diethylstibestrol	80	69	83	52
$^{13}C_6$-Estradiol	63	65	58	50
d2-epi-Estriol	85	98	97	72
$^{13}C_6$-Estrone	118	116	106	81
d4-Ethynylestradiol	91	99	92	75
d3-Medroxyprogesterone	55	43	47	42
d4-Mestranol	103	101	94	74
d3-Nandrolone	121	96	98	74

Compound	Site A µg/L	Site B µg/L	Site C µg/L	Site D µg/L
d4-Mestranol	103	101	94	74
d3-Nandrolone	121	96	98	74

Table 7. Site characterization of Sullivan Lake microhabitat A (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010.
[x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters;

		Location (x, y) in m			
Constituent		**(0, 1) (0, 3) (0, 5)**	**(5, 1) (5, 3) (5, 5)**	**(10, 1) (10, 3) (10, 5)**	**(15, 1) (15, 3) (15, 5)**
Dissolved organic carbon	mg/L	3.0	2.6	2.1	2.1
		5.9	4.0	2.1	3.4
		3.2	7.4	3.6	3.7
Linear alkylbenzene sulfonates	mg/L	< .02	.29	.04	.16
		< .02	.92	.13	.13
		.38	.15	.04	.10
Alkylphenolethoxylates	mg/L	.14	< .02	< .02	< .02
		< .02	< .02	< .02	< .02
		< .02	< .02	< .02	< .02
Hydraulic head	mm	0	NM	.8	.1
		15	2.1	.1	.3
		3.1	1.4	.7	2.8
$\text{Temperature}_{\text{water/sediment}}$	°C	24.3	25.4	24.6	23.8
		25.1	25.6	25.0	23.0
		25.6	25.5	25.9	23.1
$\text{Temperature}_{\text{30 cm}}$	°C	22.9	21.7	21.2	16.5
		24.0	23.7	22.4	19.2
		23.4	23.6	23.4	20.5

°C, degrees Celsius; <, less than; NM, no field measurement; cm, centimeters]

Table 8. Water column water-quality measurements for Sullivan Lake microhabitat A (residential/septic influence) collected from 1:30 to 3:00 PM on July 21, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; per centimeter; mg/L, mill per liter; standard units, **standard unit]**

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	42	10	29.1	390	8.9	8.5
		32	28.9	392	7.7	8.3
(3, 5)	39	10	29.1	388	7.9	8.6
		29	28.7	390	7.9	8.5
(5, 5)	42	10	29.1	384	9.1	8.7
		32	30.0	385	8.8	8.6
(10, 5)	44	10	29.1	386	7.8	8.6
		34	28.9	386	7.8	8.5
(15, 5)	48	10	29.0	385	8.4	8.5
		38	27.4	414	5.8	7.9

Table 9. Site characterization of Sullivan Lake microhabitat B (residential/septic influence) and water-quality values for pore-water samples collected July 8, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters; °C, degrees Celsius; <, less than; NM, no field measurement; cm, centimeters]

		Location (x, y) in m			
Constituent		**NM (0, 3) (0, 5)**	**NM (5, 3) (5, 5)**	**(10, 1) (10, 3) (10, 5)**	**(15, 1) (15, 3) (15, 5)**
Dissolved organic carbon	mg/L	NM	NM	7.6	9.4
		7.8	7.6	8.7	10.4
		10.3	10.0	11.4	10.1
Linear alkylbenzene sulfonates	mg/L	NM	NM	< .02	< .02
		.02	.04	< .02	< .02
		.12	.02	< .02	.03
Alkylphenolethoxylates	mg/L	NM	NM	< .02	< .02
		< .02	.10	< .02	.04
		.11	< .02	< .02	< .02
Hydraulic head	mm	NM	NM	-1	-3
		-2	0	-3	1
		3	-1	-1	0
$Temperature_{water/sediment}$	°C	NM	NM	NM	NM
		NM	NM	NM	NM
		NM	NM	NM	NM
$Temperature_{30\ cm}$	°C	NM	NM	NM	NM
		NM	NM	NM	NM
		NM	NM	NM	NM

Table 10. Water column water-quality measurements for Sullivan Lake microhabitat B (residential/septic influence) collected 10:30 AM to 2:00 PM on July 21, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; mS/cm , microsiemens per centimeter; mg/L, milligrams per liter; standard unit, standard unit]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	52	10	27.0	378	7.2	8.7
		26	26.8	378	8.0	8.7
		42	26.6	379	7.7	8.6
(3, 5)	54	10	27.1	377	7.8	8.7
		27	26.9	379	8.1	8.7
		44	26.8	378	8.7	8.7
(5, 5)	55	10	27.0	379	8.4	8.7
		28	26.8	378	8.7	8.8
		45	26.8	378	8.4	8.8
(10, 5)	60	10	27.2	378	8.8	8.7
		30	27.0	378	9.5	8.8
		50	26.9	378	8.9	8.7
(15, 5)	65	10	27.2	378	9.2	8.7
		22	27.0	378	9.3	8.8
		55	26.7	378	9.6	8.8

Table 11. Site characterization of Sullivan Lake microhabitat C (stormwater/boat-ramp influence) and water-quality values for pore-water samples collected July 8, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; °C, degrees Celsius; <, less than; NM, not measured; cm, centimeters]

		Location (x, y) in m	
		(0, 1)	**NM**
Constituent		**NM**	**(15, 5)**
Dissolved organic carbon	mg/L	11.2	NM
		NM	7.3
Linear alkylbenzene sulfonates	mg/L	< .02	NM
		NM	.03
Alkylphenolethoxylates	mg/L	.11	NM
		NM	< .02
Temperature$_{\text{water/sediment}}$	°C	23.7	NM
		NM	28.6
Temperature$_{\text{30 cm}}$	°C	19.4	NM
		NM	17.7

Table 12. Water column water-quality measurements for Sullivan Lake microhabitat C (stormwater/boat ramp influence) collected 12:00 to 1:30 PM on July 21, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; total depth, distance from water surface to bed sediments; cm, centimeters; °C, degrees Celsius; mS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	82	10	27.1	384	9.2	8.4
		41	26.3	387	7.8	8.1
		72	26.2	392	5.2	7.8
(3, 5)	69	10	27.4	383	7.7	8.4
		35	26.8	385	7.8	8.3
		59	26.3	384	7.5	8.3
(5, 5)	68	10	27.4	381	8.4	8.4
		34	26.6	384	7.8	8.2
		58	26.2	389	6.9	7.7
(10, 5)	73	10	27.5	379	9.6	8.5
		37	26.5	384	9.1	8.1
		63	26.3	388	7.3	7.8
(15, 5)	71	10	26.9	379	10.1	8.5
		36	26.5	384	8.0	8.1
		61	26.1	394	5.9	7.5

Table 13. Site characterization of Sullivan Lake microhabitat D (agricultural influence) and water-quality values for pore-water samples collected July 9, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure 1 for specific sampling-grid orientation; m, meters; mg/L, milligrams per liter; mm, millimeters; °C, degrees Celsius; <, less than; NM, no measurement; * positive bias due to high inorganic carbon content; cm, centimeters]

		Location (x, y) in m	
		(0, 1)	NM
		(0, 3)	NM
Constituent		(0, 5)	(5, 5)
Dissolved organic carbon	mg/L	31.8*	NM
		12.6*	NM
		32.3*	5.0
Linear alkylbenzene sulfonates	mg/L	.02	NM
		.11	NM
		.02	.11
Alkylphenolethoxylates	mg/L	< .02	NM
		< .02	NM
		< .02	< .02
Hydraulic head	mm	1.2	NM
		NM	NM
		NM	NM
$\text{Temperature}_{\text{water/sediment}}$	°C	22.9	NM
		23.8	NM
		21.7	19.9
$\text{Temperature}_{30\text{ cm}}$	°C	18.5	NM
		18.8	NM
		17.4	16.7

Table 14. Water column water-quality measurements for Sullivan Lake microhabitat D (agricultural influence) collected 9:00 to 10:30 AM on July 21, 2010. [x, distance parallel to shore; y, distance perpendicular to shore; see Figure. 1 for specific sampling-grid orientation; m, meters; cm, centimeters; °C, degrees Celsius; □S/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit; NM, no measurement]

Location (x, y) in m	Total depth cm	Depth from water surface cm	Temperature C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
(0, 5)	70	10	25.1	388	4.1	7.4
		35	25.2	388	3.7	7.5
		50	25.1	389	3.6	7.5
(3, 5)	72	10	25.5	880	4.3	7.6
		36	25.1	406	3.2	6.9
		52	25.0	443	2.9	6.7
(5, 5)	76	10	25.4	389	4.4	7.5
		38	25.2	450	2.9	6.7
		56	25.0	540	1.9	6.7
(10, 5)	71	10	25.8	385	5.1	7.9
		36	25.4	472	2.5	6.7
		51	NM	252	1.9	6.6
(15, 5)	78	10	26.1	384	5.6	7.9
		39	25.6	387	4.8	7.7
		58	25.2	451	3.0	6.8

Table 15. Pesticide data

Microhabitat	Acetochlor (µg/L)	Alachlor (µg/L)	Atrazine (µg/L)	Boscalid (µg/L)	Chlorothalonil (µg/L)	Chlorpyrifos (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Clomazone (µg/L)	Cyanazine (µg/L)	Cyfluthrin (µg/L)	Deisopropylatrazine (µg/L)	Desethylatrazine (µg/L)	Diazinon (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Dimethenamid (µg/L)	Dimethoate (µg/L)	Disulfoton (µg/L)	EPTC (µg/L)	Esfenvalerate (µg/L)	Ethafluralin (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Fonofos (µg/L)	Lambda Cyhalothrin (µg/L)	Malathion (µg/L)	Metolachlor (µg/L)	Metribuzin (µg/L)	Metribuzin DA (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Metribuzin DADK (µg/L)	Metribuzin DK (µg/L)	Myclobutanil (µg/L)	Oxadiazon (µg/L)	Parathion, Methyl (µg/L)	Pendimethalin (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Phorate (µg/L)	Prometon (µg/L)	Propachlor (µg/L)	Propazine (µg/L)	Propiconazole (µg/L)	Pyraclostrobin (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	NA
C	ND	ND	ND	ND	ND	NA
D	ND	ND	ND	ND	ND	NA

Microhabitat	Simazine (µg/L)	Tebuconazole (µg/L)	Tebuprimiphos (µg/L)	Terbufos (µg/L)	Tetraconazole (µg/L)	Triallate (µg/L)
A	NA	NA	NA	NA	NA	NA
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Trifluralin (µg/L)	Zeta Cypermethrin (µg/L)	2,4,5-T (ng/L)	2,4,5-TP (ng/L)	2,4-D (ng/L)	2,4-DB (ng/L)
A	NA	NA	ND	ND	79.2	ND
B	ND	ND	ND	ND	67.1	ND
C	ND	ND	ND	ND	79.4	ND
D	ND	ND	ND	ND	67.8	ND

Microhabitat	Acetochlor ESA (ng/L)	Acetochlor OXA (ng/L)	Alachlor ESA (ng/L)	Alachlor OXA (ng/L)	Bentazon (ng/L)	Clopyralid (ng/L)
A	108	52.3	61.6	ND	ND	ND
B	81.6	45.3	ND	ND	ND	ND
C	95.4	51.6	45.3	ND	ND	ND
D	86.7	54.3	51.2	ND	ND	ND

Microhabitat	Dicamba (ng/L)	Dichlorprop (ng/L)	Dimethenamid ESA (ng/L)	Dimethenamid OXA (ng/L)	Flufenacet OXA (ng/L)	Isoxaflutole Deg (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	MCPA (ng/L)	MCPB (ng/L)	MCPP (ng/L)	Mesotrione (ng/L)	Metolachlor ESA (ng/L)	Metolachlor OXA (ng/L)
A	ND	ND	ND	ND	20.9	ND
B	ND	ND	ND	ND	17.3	ND
C	ND	ND	ND	ND	18.0	ND
D	ND	ND	ND	ND	19.1	ND

Microhabitat	Picloram (ng/L)	Propachlor ESA (ng/L)	Propachlor OXA (ng/L)	Tembotrione (ng/L)	Triclopyr (ng/L)	Acetamiprid (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Aldicarb sulfone (ng/L)	Aldicarb sulfoxide (ng/L)	Azoxystrobin (ng/L)	Bensulfuron Methyl (ng/L)	Bromacil (ng/L)	Carbaryl (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Carbofuran (ng/L)	Chlorimuron Ethyl (ng/L)	DEDI Atrazine (ng/L)	Disulfoton Sulfone (ng/L)	Diuron (ng/L)	Halosulfuron Methyl (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Hexazinone (ng/L)	Hydroxyatrazine (ng/L)	Imazamethabenz Acid (ng/L)	Imazamethabenz Methyl (ng/L)	Imazamox (ng/L)	Imazapic (ng/L)
A	ND	149	ND	ND	ND	ND
B	ND	139	ND	ND	ND	ND
C	ND	140	ND	ND	ND	ND
D	ND	148	ND	ND	ND	ND

Microhabitat	Imazapyr (ng/L)	Imazaquin (ng/L)	Imazethapyr (ng/L)	Imidacloprid (ng/L)	Isoxaflutole (ng/L)	Linuron (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Metalaxyl (ng/L)	Metsulfuron methyl (ng/L)	Neburon (ng/L)	Nicosulfuron (ng/L)	Norflurazon (ng/L)	Prometryn (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Propoxur (ng/L)	Saflufenacil (ng/L)	Siduron (ng/L)	Sulfometuron methyl (ng/L)	Thiamethoxam (ng/L)	Thifensulfuron Methyl (ng/L)
A	ND	ND	ND	ND	ND	ND
B	ND	ND	ND	ND	ND	ND
C	ND	ND	ND	ND	ND	ND
D	ND	ND	ND	ND	ND	ND

Microhabitat	Thiobencarb (ng/L)	Triasulfuron (ng/L)
A	ND	ND
B	ND	ND
C	ND	ND
D	ND	ND

NA: Not Analyzed
ND: Not Detected

Samples collected: 8/23/2010

PCA/DNR SITE ID: 86-0119

MDA SITE ID: 1SUL

Appendix C

Remote lakes

Beast, Ryan, Bohall, and Elk Lakes

Surface water data

Table 1. Summary of field measurements for water samples collected from four Minnesota lakes during 2010.

Table 2. Summary of acidic organic compound data for water samples collected from four Minnesota lakes during 2010.

Table 3. Summary of neutral organic compound data for water samples collected from four Minnesota lakes during 2010.

Table 4. Summary of steroid and steroidal hormone compound data for water samples collected from four Minnesota lakes during 2010.

Table 1. Summary of field measurements for water samples collected from four Minnesota lakes during 2010. [°C, degrees Celsius; µS/cm, microsiemens per centimeter; mg/L, milligrams per liter; std unit, standard unit]

	Temperature °C	Specific conductance µS/cm	Dissolved oxygen mg/L	pH std unit
Elk	11.8	294	8.6	8.4
Beast	10.7	23	9.5	6.9
Bohall	9.6	182	10.0	8.0
Ryan	10.7	23	9.8	6.8

Table 2. Summary of acidic organic compound data for water samples collected from four Minnesota lakes during 2010. [µg/L, microgram per liter; <, less than]

Compound	Elk µg/L	Beast µg/L	Bohall µg/L	Ryan µg/L
Ethylenediaminetetraacetic acid	0.53	<0.05	0.06	0.40
Nitrilotriacetic acid	< .05	< .05	< .05	< .05
4-Nonylphenolmonoethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoldiethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoltriethoxycarboxylic acid	< .1	< .1	< .1	< .1
4-Nonylphenoltetraethoxycarboxylic acid	< .1	< .1	< .1	< .1
Surrogate Recovery (percent)				
4-*normal*-Nonylphenoldiethoxycarboxylic acid	61	59	67	54

Table 3. **Summary of neutral organic compound data for water samples collected from four Minnesota lakes during 2010. [µg/L, microgram per liter; <, less than; --, not quantifiable]**

Compound	Elk µg/L	Beast µg/L	Bohall µg/L	Ryan µg/L
Acetylhexamethyltetrahydronaphthalene	<0.01	<0.01	<0.01	<0.01
Bisphenol A	< .01	< .01	< .01	< .01
2[3]-*tert*-Butyl-4-methylphenol	< .01	< .01	< .01	< .01
4-*tert*-Butylphenol	< .01	< .01	< .01	< .01
Caffeine	< .01	< .01	< .01	< .01
2,6-Di-*tert*-butyl-1,4-benzoquinone	.05	.04	.02	< .01
2,6-Di-*tert*-butyl-4-methylphenol	.01	< .01	< .01	< .01
2,6-Di-*tert*-butylphenol	< .01	< .01	< .01	< .01
1,2-Dichlorobenzene	< .01	< .01	< .01	< .01
1,3-Dichlorobenzene	< .01	< .01	< .01	< .01
1,4-Dichlorobenzene	< .01	< .01	< .01	< .01
N,N-Diethyl-*meta*-toluamide	< .01	< .01	< .01	< .01
4-Ethylphenol	< .01	< .01	< .01	< .01
Hexahydrohexamethylcyclopentabenzopyran	< .01	< .01	< .01	< .01
5-Methyl-1H-benzotriazole	< .01	< .01	< .01	< .01
4-Methylphenol	< .01	.01	< .01	.01
4-Nonylphenol	< .05	< .05	< .05	< .05
4-Nonylphenolmonoethoxylate	< .05	< .05	< .05	< .05
4-Nonylphenoldiethoxylate	< .05	< .05	< .05	< .05
4-*normal*-Octylphenol	< .01	< .01	< .01	< .01
4-*tert*-Octylphenol	< .01	< .01	< .01	< .01
4-*tert*-Octylphenolmonoethoxylate	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoldiethoxylate	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoltriethoxylate	< .01	< .01	< .01	< .01
4-*tert*-Octylphenoltetraethoxylate	< .01	< .01	< .01	< .01
4-*tert*-Octylphenolpentaethoxylate	< .01	< .01	< .01	< .01
4-*tert*-Pentylphenol	< .01	< .01	< .01	< .01

Compound	Elk µg/L	Beast µg/L	Bohall µg/L	Ryan µg/L
4-Propylphenol	< .01	< .01	< .01	< .01
Triclosan	< .01	< .01	< .01	< .01
Surrogate Recovery (percent)				
d6-Bisphenol A	27	49	52	32
d21-2,6-Di-*tert*-butyl-4-methylphenol	53	85	21	63
4-*normal*-Nonylphenol	38	56	10	37
4-*normal*-Nonylphenolmonoethoxylate	38	51	47	37
4-*normal*-Nonylphenoldiethoxylate	46	63	--	34

Table 4. Summary of steroid and steroidal hormone compound data for water samples collected from four Minnesota lakes during 2010. [µg/L, microgram per liter; <, less than]

Compound	Elk µg/L	Beast µg/L	Bohall µg/L	Ryan µg/L
4-Androstene-3,17-dione	<0.0001	<0.0001	<0.0001	< .0001
cis-Androsterone	< .0001	< .0001	< .0001	< .0001
Cholesterol	1.4	1.2	1.2	2.4
Coprostanol	.06	.08	.01	< .005
Diethylstilbestrol	< .0001	< .0001	< .0001	< .0001
Equilenin	< .0001	< .0001	< .0001	< .0001
Equilin	< .0001	< .0001	< .0001	< .0001
17α-Estradiol	< .0001	< .0001	< .0001	< .0001
17β-Estradiol	< .0001	< .0001	< .0001	< .0001
Estriol	< .0001	< .0001	< .0001	< .0001
Estrone	.0001	.0001	.0001	.0008
17α-Ethynylestradiol	< .0001	< .0001	< .0001	< .0001
Mestranol	< .0001	< .0001	< .0001	< .0001
Norethindrone	< .0001	< .0001	< .0001	< .0001
Progesterone	< .0001	< .0001	< .0001	< .0001
Testosterone	< .0001	< .0001	< .0001	< .0001
dihydro-Testosterone	< .0001	< .0001	< .0001	< .0001
epi-Testosterone	.0001	< .0001	< .0001	< .0001
11-*keto*-Testosterone	< .0001	< .0001	< .0001	< .0001
Surrogate Recovery (percent)				
d7-Cholesterol	64	40	48	54
d12-Chrysene	101	100	99	98
d8-Diethylstibestrol	75	78	83	90
$^{13}C_6$-Estradiol	57	60	76	82
d2-*epi*-Estriol	96	90	66	94
$^{13}C_6$-Estrone	115	109	95	105
d4-Ethynylestradiol	94	90	83	91

Compound	Elk µg/L	Beast µg/L	Bohall µg/L	Ryan µg/L
d3-Medroxyprogesterone	76	67	56	37
d4-Mestranol	95	90	82	90
d3-Nandrolone	102	93	86	83