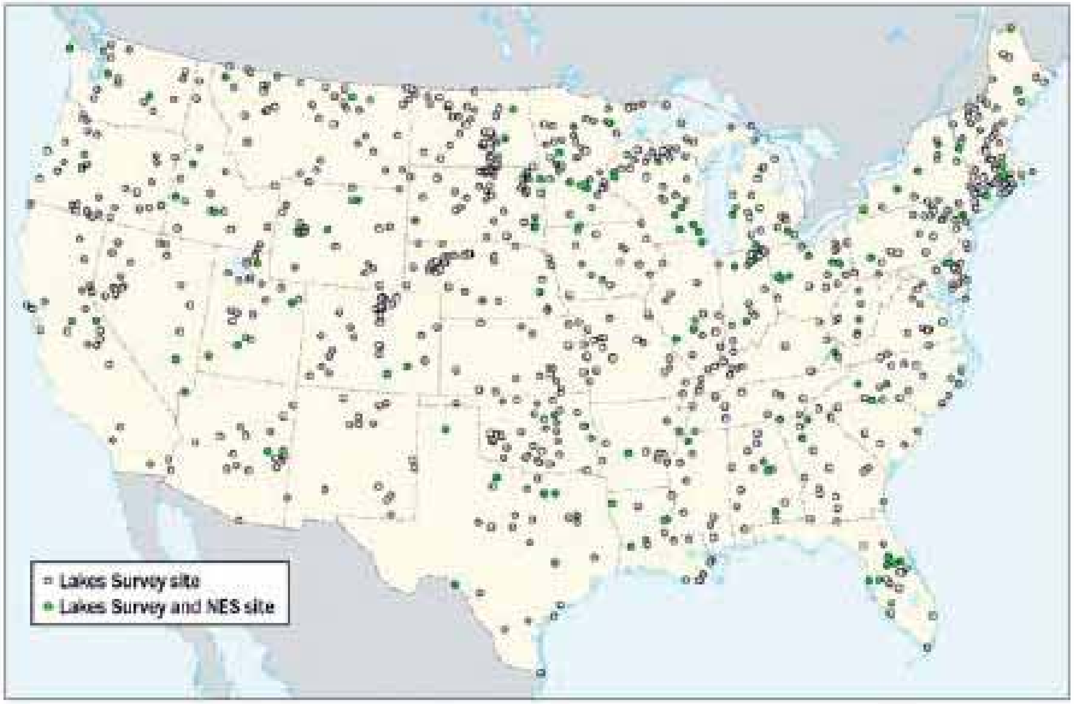

Minnesota National Lakes Assessment Project:

Pesticides in Minnesota Lakes

This report is part of a series based on Minnesota's participation in U.S. Environmental Protection Agency's 2007 National Lake Assessment

MINNESOTA DEPARTMENT OF AGRICULTURE
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Abstract

In 2007 the Minnesota Department of Agriculture (MDA) cooperated with the Minnesota Pollution Control Agency (MPCA) in a statewide assessment of pesticides in Minnesota lakes as part of the National Lakes Assessment Program (NLAP) coordinated by United States Environmental Protection Agency (USEPA). Fifty-three lakes were randomly selected and sampled by MPCA and USEPA staff from throughout the state. Samples were analyzed by the MDA laboratory for 37 commonly used agricultural pesticides and their degradates. Pesticides and/or degradates were detected in 91 percent of the samples collected. The corn herbicide atrazine was the most frequently detected compound. All pesticide detections were well below established water quality standards or USEPA reference values. Pesticide detections were widespread, including many non-agricultural areas of the state. Atmospheric deposition of pesticides is suspected as the primary transport mechanism in non-agricultural areas.

Keywords:
NLAP, Minnesota, Pesticides, Lakes, Atrazine, Degradates, Atmospheric Deposition

Introduction

Minnesota is known as “The Land of 10,000 Lakes” and has a history and heritage linked to its abundant water resources. These valued natural resources are used and promoted by a vibrant tourism industry. Minnesota is also known as a major agricultural state, ranking fourth nationally in the production of corn and soybeans. Both water-based recreation and agriculture are key contributors to Minnesota’s economy.

In recent years there has been an increase in corn acres planted in response to demand for corn-based ethanol (Hartwig, 2008). Along with this has come an associated increase in corn herbicide usage. In 2008, the first surface water impairment by a corn herbicide (acetochlor) in Minnesota was identified and placed on the 303(d) list of the state’s impaired waters maintained by the Minnesota Pollution Control Agency (MPCA).

This study focused on the presence of pesticides, and their associated breakdown products, or degradates, in lakes across Minnesota. This survey of 53 randomly selected lakes was completed in conjunction with the United States Environmental Protection Agency’s (USEPA) National Lake Assessment Program (NLAP). The lakes were sampled by MPCA and USEPA staff during the summer of 2007. The Minnesota Department of Agriculture (MDA) provided laboratory analytical services and data analysis for the pesticide samples.

Background

The MDA has an extensive statewide water monitoring program designed to evaluate the impact of pesticides on surface and ground water throughout the state. The surface water portion of the program has historically focused on streams and rivers, specifically systems which drain agricultural areas of Minnesota. Streams and rivers have been the focus of MDA pesticide monitoring efforts because they are generally regarded to be at greater risk than lakes from land-applied pesticides. Approximately 60 river and stream locations are sampled annually with varying levels of intensity. Lake pesticide sampling in Minnesota has been limited and is discussed in greater detail below.

Corn herbicide use estimates in Minnesota from 1990 through 2005 are shown in Figure 1. Figure 1 suggests an overall decline in pesticide usage on corn. With the increased use of the herbicide glyphosate on corn there has been a general decrease in use of historically popular corn herbicides such as atrazine and acetochlor. In addition to the general declining use of corn herbicides, there has been an expanded outreach and regulatory effort to educate Minnesota pesticide applicators on pesticide product label requirements and voluntary Best Management Practices (BMPs) for water-quality protection.

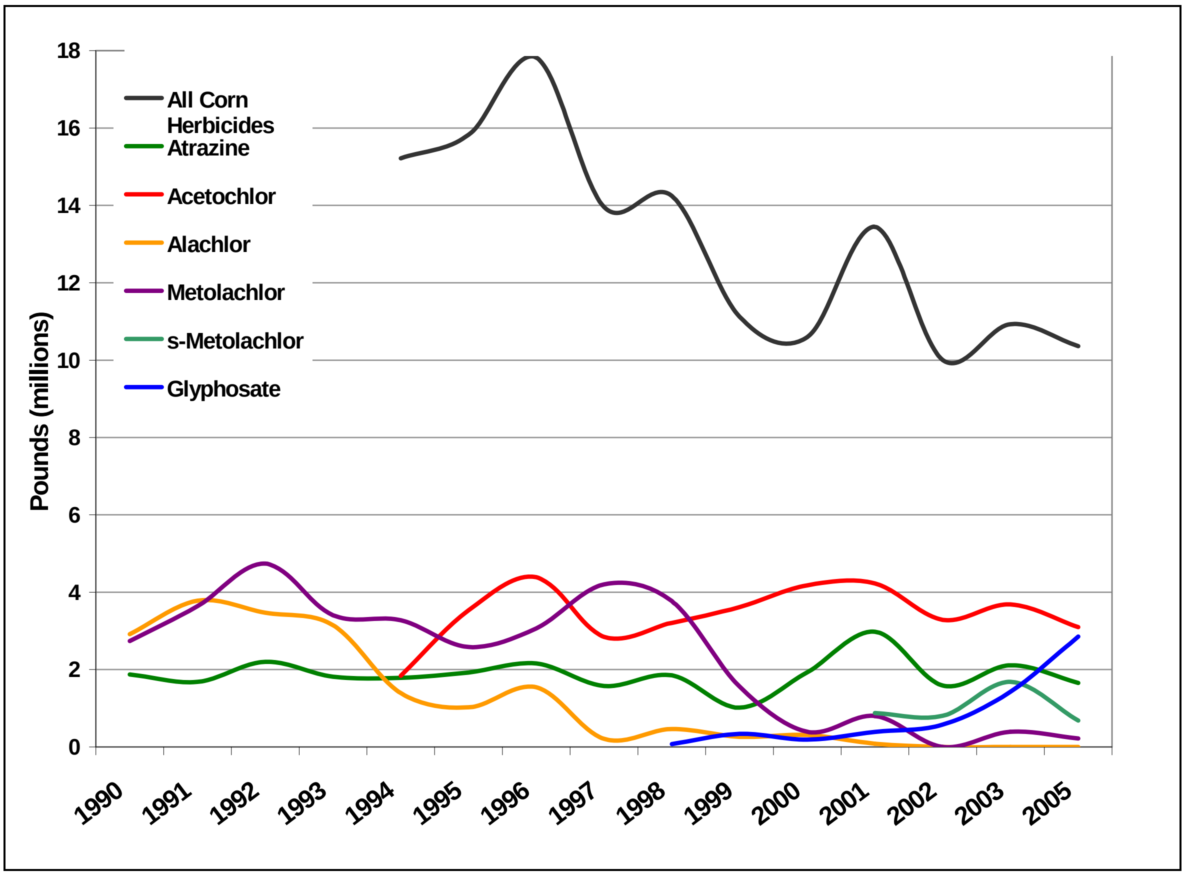

Figure 1.Total corn herbicide use estimates in Minnesota, 1990-2005: Pounds of all herbicides and major active ingredients by year (MDA and United States Department of Agriculture - National Agriculture Statistics Service (USDA-NASS)).

Previous Investigations

From 1992 through 1995 the MDA cooperated with the Minneapolis Park and Recreation Board, United States Geological Survey (USGS) and the Minnesota Grey Freshwater Institute in a study of Lake Harriet, an urban lake located in Minneapolis, Minnesota. The purpose of the study was to evaluate the presence of 26 commonly used urban and agricultural pesticides in the watershed. The study found that between 25 percent and 91 percent of lake samples collected from the epilimnion (upper 4 meters) contained detectable levels of atrazine (0.10 – 0.40 ug/L). Atrazine detections in samples collected from the hypolimnion were similar, ranging from 29 percent to 92 percent with concentrations ranging from 0.10 to 0.40 ug/L. During this period (1992 – 1995) no other agricultural, base-neutral pesticides were detected in Lake Harriet, however, several were detected in storm water runoff within the watershed. Rainfall analysis over the same period was conducted in the watershed and found alachlor, atrazine, cyanazine, and metolachlor present in 54 percent, 71 percent, 42 percent, and 46 percent of the samples respectively. Atmospheric deposition was identified as the primary source of agricultural pesticides in the urban area evaluated (Wotzka, 1998).

In a related study, the MDA cooperated with the USGS in evaluating wet atmospheric deposition of pesticides in Minnesota between 1989 and 1994 (Capel, 1994). More than 350 precipitation samples were collected at several sites across Minnesota during the study and all samples had detectable quantities of at least one pesticide. The most frequently detected compounds were alachlor, atrazine, cyanazine and metolachlor, all corn herbicides. Acetochlor was detected in precipitation in 1994, the first year of registration for use in Minnesota. Concentrations peaked following periods when the pesticides were applied and were highest in agricultural areas.

Figure 2 contains precipitation monitoring data collected by MDA from 1998 through 2005 in an agricultural area of southeastern Minnesota. The average monthly concentration for the four most frequently detected pesticides is presented. The figure shows strong seasonality in the relative concentration of these pesticides. This seasonal pattern corresponds to the herbicide's primary application periods in Minnesota.

These values are generally consistent with the findings of a USGS study that examined the atmospheric transport and deposition of commonly used herbicides in the "Corn Belt" of the United States (Starner, 1998). According to their report:

> *"Herbicides can be transported into the atmosphere by volatilization and entrainment on dust particles and then dispersed by air currents for possible re-deposition on watersheds at considerable distances from their application."*

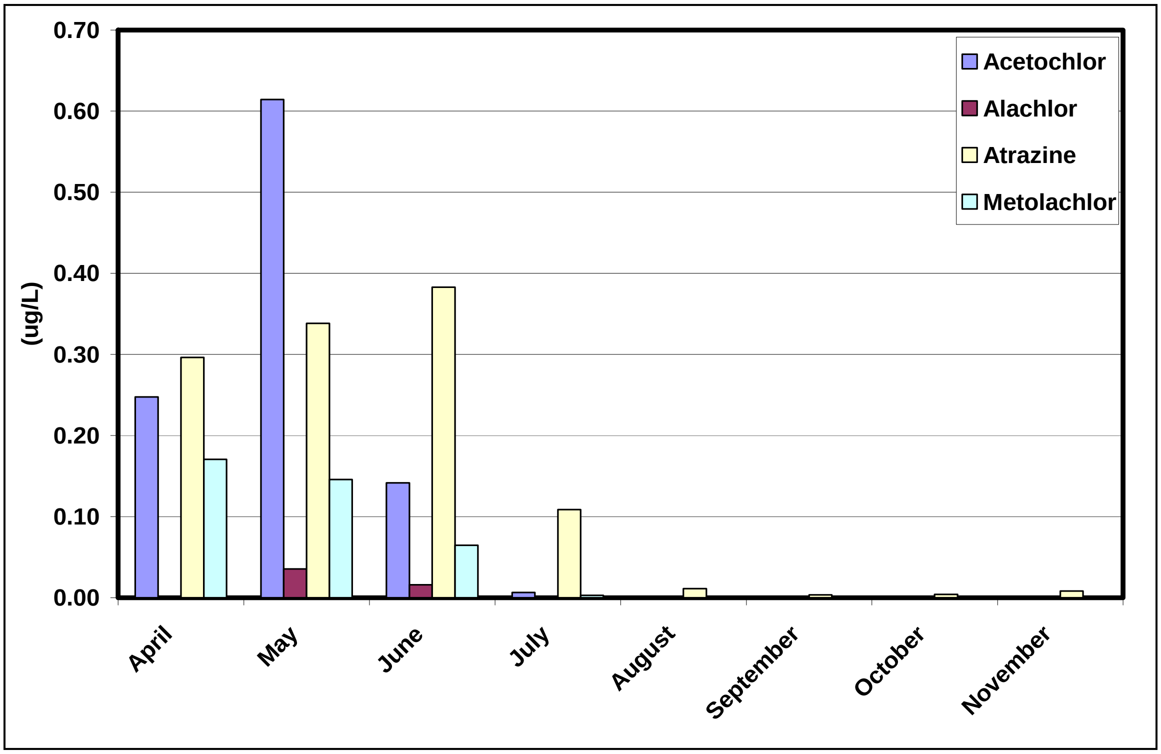

Figure 2. Average monthly pesticide concentration in precipitation samples collected from 1998 through 2005 in southeastern Minnesota (MDA data).

The USGS study analyzed 6,230 rainfall samples collected from sites located throughout the Midwest and Northeast United States. Immunoassay results of 5,927 samples found that 30.1 percent of samples had triazines and/or acetanilides present. Atrazine was present in 30.2 percent and alachlor was present in 19.2 percent of the samples (n=2,085 samples) screened by immunoassay and analyzed by gas chromatography/mass spectrometry (GC/MS) methods. These two compounds were detected most frequently. The concentrations of each pesticide measured in rainfall varied with climatic variability, but ultimately increased during periods of pesticide applications and were greatest in rain that fell during periods of low rainfall. Overall, rainfall-weighted average concentrations during the application period of mid-April through mid-July yielded concentrations of both atrazine and alachlor in rainfall of 0.2 to 0.4 ug/L. Higher concentrations of atrazine and alachlor were reported in areas of higher use (Iowa, Illinois, and Indiana) indicating a decrease in the amount of pesticides in the atmosphere as the distance from the source is increased (Starner, 1998).

A 2000 study was conducted by the University of Maryland and USDA in the Chesapeake Bay area to examine atmospheric deposition of pesticides into the Choptank River. The study concluded that 3 percent to 20 percent of metolachor in the Choptank River was deposited via wet atmospheric deposition. In addition to metolachor, pesticide compounds including chlorothalonil, atrazine, simazine, endosulfan, and chlorpyrifos were detected in both dry and wet deposition. The concentration of these chemicals, and their usage in the local area of this study, predicts atmospheric deposition is a significant source of agricultural pesticides in surface waters (Kuang, 2003).

One of the largest studies of pesticides in lakes of the Corn Belt region of the United States was conducted by the Acetochlor Registration Partnership (ARP). In 1995 the ARP implemented a multi-year, multi-analyte monitoring program to satisfy the conditions of the USEPA registration of acetochlor. The ARP study sampled surface water bodies used as drinking water sources throughout the Midwest and included lakes and reservoirs in Illinois, Indiana, Iowa, Kansas, Nebraska, Missouri, Ohio, and Wisconsin. Table 1 is based on all 31 ARP sites that had raw (pre-treatment) water samples and were categorized as either lakes or reservoirs. Watershed sizes for these 31 sites varied from 208 acres to more than 28 million acres. Land cover in these watersheds varied from 13 percent to 45 percent corn production (Acetochlor Registration Partnership, 2008).

There are seven years of monitoring data available for the three parent compounds of acetochlor, atrazine and metolachlor (1995-2001). There are three years of monitoring data available for their degradates (1999-2001). Fourteen samples were collected each year, biweekly through the spring and summer months, and quarterly during the fall and winter. Results indicate that detection frequencies for these compounds ranged from 100 percent for atrazine to 21 percent for acetochlor (Table 1).

Table 1. Acetochlor Registration Partnership (ARP) monitoring results in lakes/reservoirs (raw water only, N=31 sites, 14 samples per year).

Analyte (years monitored)	Limit of Detection (ug/L)	Detection Frequency
Acetochlor (1995-2001)	0.03	21%
Atrazine (1995-2001)	0.03	100%
Metolachlor (1995-2001)	0.03	61%
Acetochlor-ESA (1999-2001)	0.2	32%
Acetochlor-OXA (1999-2001)	0.1	58%
Metolachlor-ESA (1999-2001)	0.2	70%
Metolachlor-OXA (1999-2001)	0.1	77%

In 2005 the Wisconsin Department of Agriculture, Trade and Consumer Protection completed a survey of atrazine in 53 lakes in agricultural areas of Wisconsin using immunoassay methods. Atrazine was detected in more than 90 percent of the lakes with concentrations ranging from non-detect (0.10 ug/L) to 0.40 ug/L. Concentrations were greater where agriculture represented greater than 75 percent of the surrounding land use (Allen, 2006).

Methods

A total of 53 Minnesota lakes were sampled for pesticides. Of these 53 lakes, 41 were originally selected by the USEPA to be part of the National Lakes Assessment Program. An additional nine lakes were added by the MPCA to make it feasible to statistically assess lakes statewide. Additional lakes were added by the USEPA as reference lakes. These lakes provided a greater basis for comparing lakes statewide, and nationally (Lindon, 2008). Two of the 53 lakes, South and Pebble, were sampled twice approximately one month apart.

Figure 3 shows the lakes and their location within the MDA Pesticide Monitoring Regions (PMRs). The PMRs were developed to allow monitoring data to be compared for areas of similar agriculture, hydrology, geology, and soils. Although lake locations relative to PMRs were not part of the selection criteria, at least one lake was sampled in nine of the ten PMRs, as shown in Figure 3. No lakes from PMR 9 were sampled. This region is known as the "Driftless Area" because it was missed in the last glacial advance across Minnesota. PMR 9 generally represents a more mature geologic landscape with karst terrain where lakes are notably absent.

Sampling Protocol

Pesticide samples were collected at all of the lakes chosen for the NLAP study. A surface grab sample was taken from the pelagic (index) site of the lake. The pesticide samples were collected early in the sampling routine and taken off the bow of the boat facing upwind to minimize the possible contamination from equipment and samplers. The samples were chilled on the boat, transported on ice for the duration of the sampling trip, and refrigerated upon return. Samples had a 10-day holding time with 52 of 53 samples analyzed within the holding time. The samples were transferred by MPCA staff to the MDA laboratory for analysis.

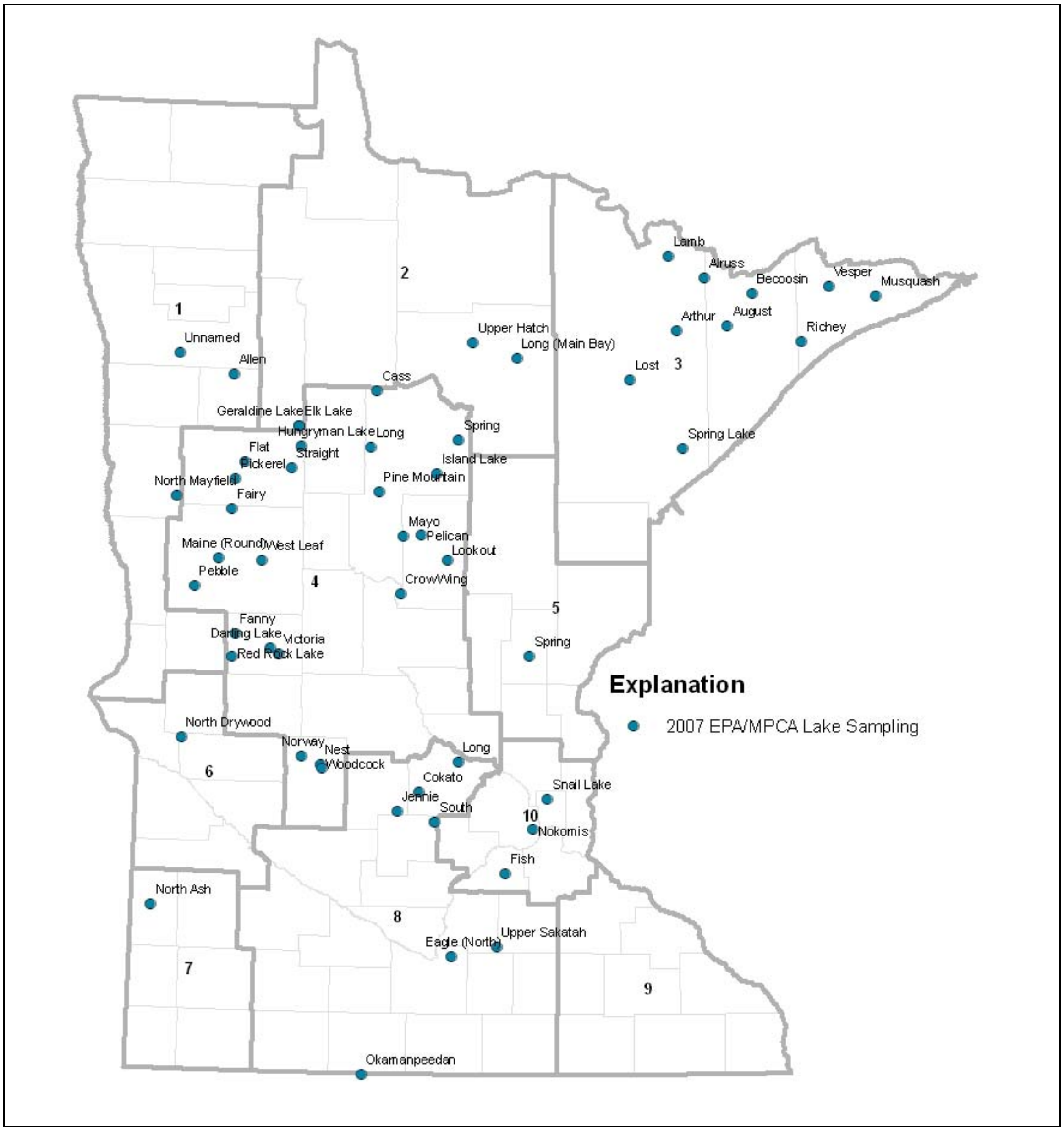

Figure 3. Lakes sampled for pesticides as part of the 2007 Minnesota portion of the National Lakes Assessment Project in cooperation with the MPCA and EPA. Map also presents the MDA Pesticide Management Regions (PMRs).

Chemical Analysis

All analyses of the pesticide samples were conducted at the MDA laboratory in St. Paul, Minnesota. Base neutral (BN) pesticide samples were prepared using automated solid phase extraction methods and analyzed with gas chromatography/mass spectrometry (GC/MS) with large volume injection. The chloroacetanilide degradate (CHL) samples were prepared using automated solid phase extraction methods and analyzed with liquid chromatography/mass spectrometry/mass spectrometry (LC/MS/MS).

Lake samples were analyzed for the standard BN target analyte pesticide list used for surface and groundwater sampling by the MDA. The BN list given in Table 2 includes many of the commonly used agricultural pesticides and their degradates. It does not include glyphosate which requires a different analytical method. In addition to the BN list, all samples were also analyzed for the chloroacetanilide degradates listed in Table 3. The method reporting limit (MRL) is the lowest concentration a given analytical method can reliably measure.

Table 2. Base neutral target analyte list.

Common Name	**Common Brand Name**	**Type**	**MRL** (ug/L)
Acetochlor	Harness, Surpass	Herbicide	0.05
Alachlor	Bronco, Lasso	Herbicide	0.05
Atrazine	Aatrex, Atrazine	Herbicide	0.05
Boscalid	Pristine	Fungicide	0.30
Chlorpyrifos	Lorsban	Insecticide	0.10
Cyanazine	Bladex	Herbicide	0.20
Deethyl atrazine	Atrazine degradate	Degradate	0.05
Deisopropylatrazine	Atrazine degradate	Degradate	0.20
Diazinon	Diazinon	Insecticide	0.12
Dimethenamid	Frontier, Outlook	Herbicide	0.05
Dimethoate	Cygon	Insecticide	0.22
EPTC	Eradicane	Herbicide	0.23
Fonofos	Dyfonate	Insecticide	0.10
Malathion	Malathion 50	Insecticide	0.09
Metolachlor	Bicep, Dual	Herbicide	0.07
Metribuzin	Boundry, Sencor	Herbicide	0.10
Metribuzin DA	Metribuzin degradate	Degradate	1.00
Metribuzin DADK	Metribuzin degradate	Degradate	1.00
Metribuzin DK	Metribuzin degradate	Degradate	1.00
Methyl Parathion	Penncap-M	Insecticide	0.12
Myclobutanil	Laredo	Fungicide	0.20
Pendimethalin	Prowl	Herbicide	0.08
Phorate	Thimet	Insecticide	0.12
Propiconazole	Tilt	Fungicide	0.20
Tebucanazole	Elite	Fungicide	0.20
Tebuprimiphos	Aztec	Fungicide	0.10
Terbufos	Counter	Insecticide	0.19
Tetraconazole	Domark	Fungicide	0.15
Trifluralin	Treflan	Herbicide	0.17

Table 3. Chloroacetanilide degradates target analyte list.

Compound	Common Brand Name	Type	MRL (ug/L)
Acetochlor ESA	Acetochlor degradate	Degradate	0.07
Acetochlor OXA	Acetochlor degradate	Degradate	0.07
Alachlor ESA	Alachlor degradate	Degradate	0.07
Alachlor OXA	Alachlor degradate	Degradate	0.07
Dimethenamid ESA	Dimethenamid degradate	Degradate	0.07
Dimethenamid OXA	Dimethenamid degradate	Degradate	0.07
Metolachlor ESA	Metolachlor degradate	Degradate	0.07
Metolachlor OXA	Metolachlor degradate	Degradate	0.07

Results

Detected compounds and frequency of detection

A total of four parent pesticide compounds were found in Minnesota lakes. Table 4 indicates that the corn herbicides acetochlor, atrazine, dimethanamid and metolachlor were all detected at least once in the 53 lakes samples.

Atrazine was detected at a much higher frequency (87 percent) and concentration as compared to the other compounds. Acetochlor, dimethanamid, and metolachlor had median concentrations that were at the non-detect level and maximum concentrations that were at or very near the method reporting limits (MRLs) identified in Table 2. The "P" in Tables 4 and 5 indicate the compounds were quantified as "present" by the laboratory but at insufficient levels to positively quantify. For statistical purposes, samples identified as "present" by the lab are assigned concentration of one half the MRL given in Table 2.

The maximum concentration of atrazine detected (0.68 ug/L) was well below the chronic atrazine water quality standard for Minnesota waters of 10 ug/L. Detections of acetochlor and metolachlor were at or below their MRL (acetochlor 0.05 ug/L and metolachlor 0.07 ug/L) and were well below their respective water quality standards of 3.6 ug/L for acetochlor and 23 ug/L for metolachlor.

With the exception of atrazine, degradates were more frequently detected than were parent compounds (Table 5). This suggests that parent compounds were likely present in more of the lakes and had degraded prior to sampling. It is also possible that some of the degradates were transported to the lakes via runoff, groundwater or atmospheric deposition. The timing of the lake sampling (July-August) was completed well after the peak pesticide application period of late spring to early summer. This may have allowed for degradation of parent compounds applied in 2007 to be transported to lakes.

When compared with the results of the ARP study presented in Table 1, the detection frequencies in this study for acetochlor (2 percent vs. 21 percent) and metolachlor (4 percent vs. 61 percent) are much lower. Atrazine detection frequency is also slightly lower (87 percent vs. 100 percent). Possible explanations for these differences include the greater sampling frequency and the timing of the sampling in the ARP study as well as the fact that the ARP study targeted lakes and reservoirs in agricultural areas. It is also possible that concentration in lakes decreased due to lower application rates or other factors since the ARP study was conducted (1995-2001).

The degradate detected at the greatest frequency was deethyl atrazine (Table 5) a breakdown product of atrazine. Other pesticide degradates were found less frequently but at higher concentrations. Currently there are no state or federal standards for pesticide degradates in surface waters; however, USEPA has available toxicity data and risk assessments for atrazine and other pesticide degradates that found water concentrations at or below reference values for the parent compound would be protective for human health (USEPA 2006a & 2006b).

Table 4. Concentration of pesticides detected in the Minnesota portion of the 2007 National Lakes Assessment Project.

	Acetochlor	Atrazine	Dimethenamid	Metolachlor
Median (ug/L)	nd*	P**	nd	nd
Maximum (ug/L)	P	0.68	P	P
Detection Frequency	2%	87%	4%	4%

*nd indicates non-detect or the median concentrations were below the method reporting limit.
** P indicates the compound was present but at a concentration below the method reporting limit (MRL) presented in table 3.

Table 5. Concentration of pesticide degradates detected in the Minnesota portion of the 2007 National Lakes Assessment Project.

	Acetochlor ESA	Acetochlor OXA	Alachlor ESA	Deisopropyl atrazine	Desethly atrazine	Metolachlor ESA	Metolachlor OXA
Median (ug/L)	nd	nd	nd	nd	P	nd	nd
Maximum (ug/L)	0.71	1.02	0.22	P	0.18	0.88	0.28
Detection Frequency	16%	18%	16%	2%	64%	27%	7%

Rivers and Lakes Compared

The median concentration of atrazine, the most commonly found agricultural pesticide in the waters of Minnesota, was compared in lakes and rivers in the 10 Pesticide Monitoring Regions (PMRs). The concentration of atrazine in the monitored rivers generally tends to be higher than the concentrations detected in lakes of the same PMR (Figure 4). PMRs 4 and 8 had the largest comparable sample data sets and in both instances indicate higher median atrazine concentrations in the rivers.

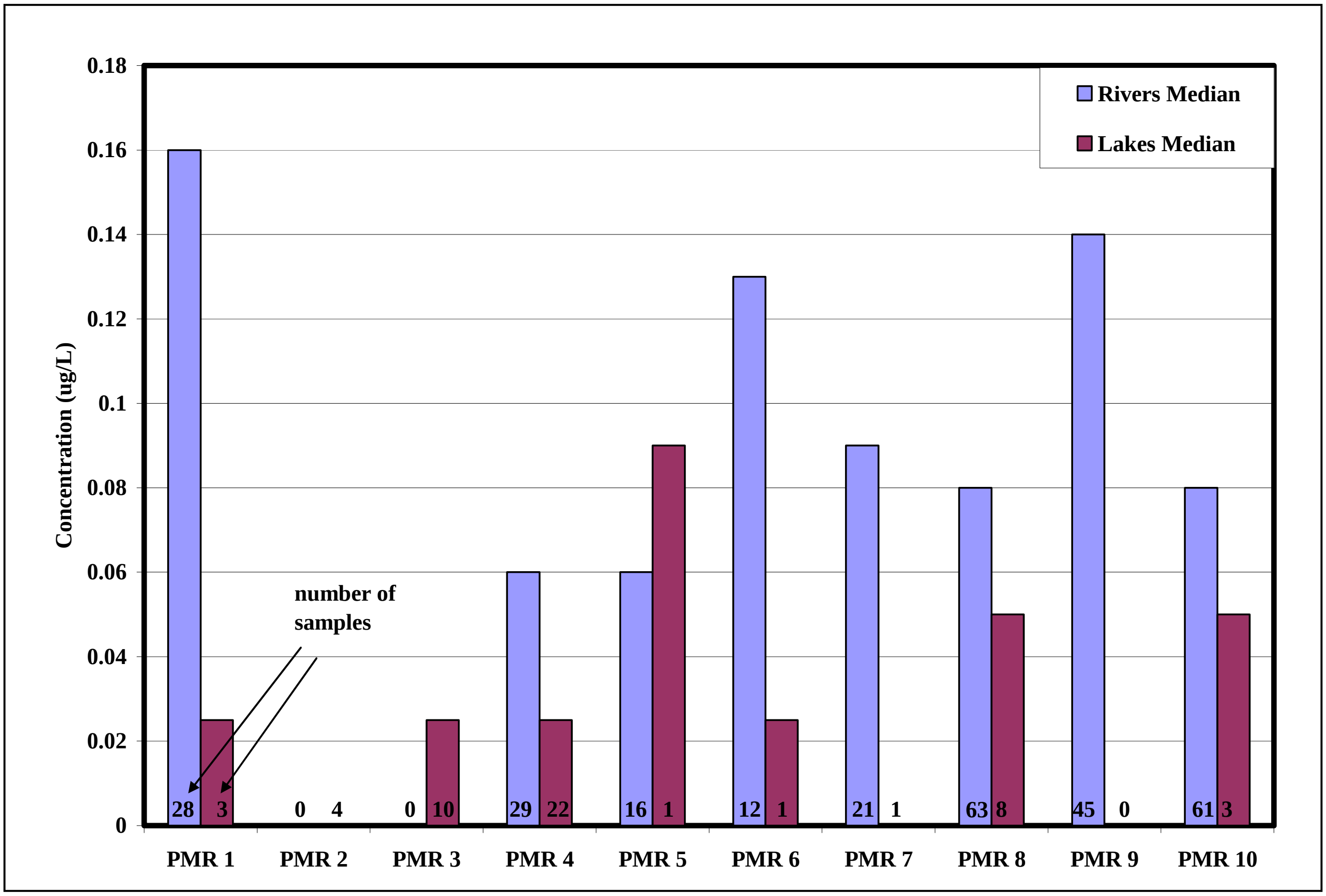

Figure 4. Median concentration of atrazine in NLAP lakes compared with median atrazine concentration in rivers sampled in 2007 by pesticide monitoring region.

Persistence in Lakes

The stability or persistence of pesticide parent compounds once they enter a lake system is not well understood. It is influenced by the conditions of the aquatic environment primarily temperature and sunlight, as well as the presence of microorganisms. Figure 5 offers an insight into the persistence question by comparing samples collected from two lakes (South and Pebble) that were sampled twice as NLAP replicate visits. The two samples collected from Pebble Lake had identical concentrations of atrazine and deethylatrazine although they were collected on July 25th and August 22nd. These results suggest apparent stability of concentration over the course of approximately one month. The two samples from South Lake also had similar results for over the course of approximately one month with the same concentration of atrazine in both samples and similar, though slightly lower concentrations of acetochlor degradates were present in the second sample. Notably absent in South Lake is deethyl atrazine in both samples and alachlor ESA in the second sample. Additional information including seasonal sampling would be required to provide further evaluation on the persistence of pesticides in lakes.

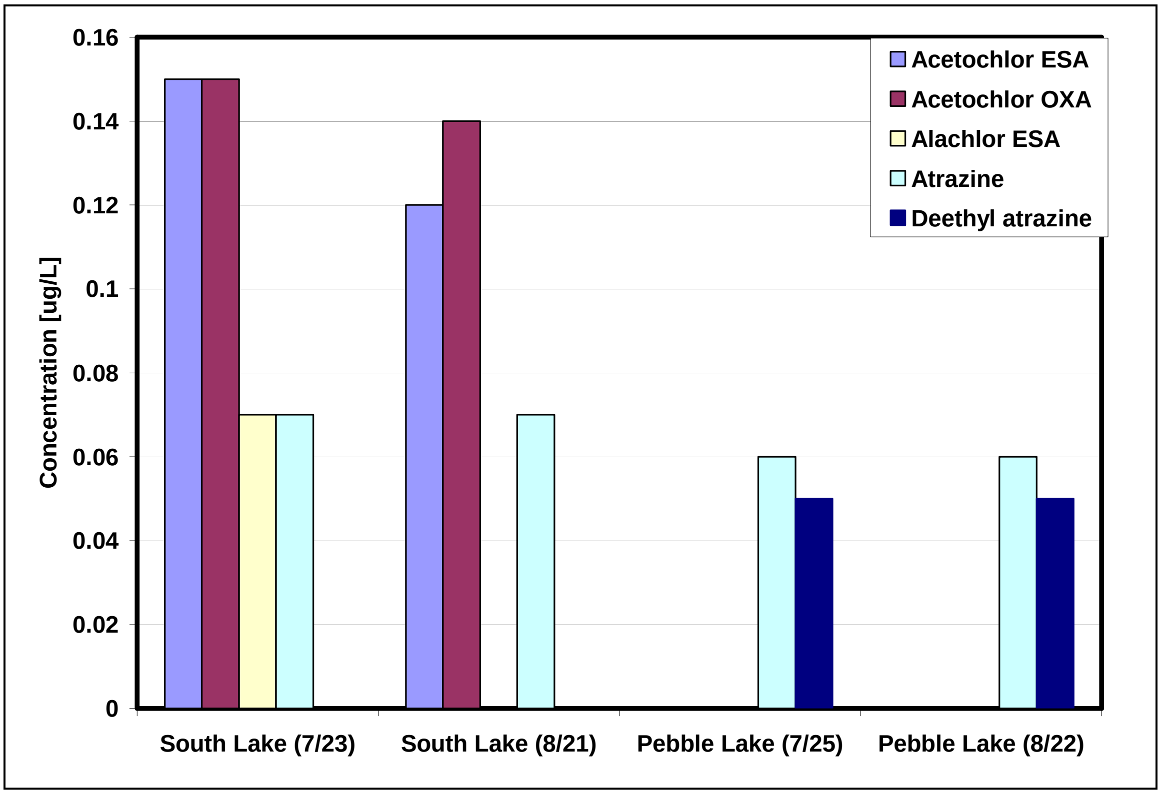

Figure 5. Pesticide concentrations of replicate sampling of South and Pebble Lakes.

Land Use

An interesting characteristic of the data collected in this study is the presence of atrazine in lakes that are located long distances (greater then 100 miles) from areas of intensive row crop agricultural activity. Previous studies (Capel, 1997, Starner, 1998 and Goolsby, 1993) have indicated that pesticides can be transported in the atmosphere long distances. Capel also suggested that there are local and regional influences on pesticide concentrations in precipitation.

In an effort to evaluate the effects of local land use, agricultural pesticide concentrations were compared between lakes with similar lakeshed land use characteristics. The lakes were placed into land use groups that are categorized by their dominate land use: urban, forest/water, or cultivated agriculture. The breakdown in average land use within these three main groups is presented in Table 6.

	Agricultural Lakesheds		Forest and Water Lakesheds		Urban Lakesheds	
	Mean	Range	Mean	Range	Mean	Range
Developed (%)	8	3-14	4	0-13	72	61-87
Cultivated Agriculture (%)	56	32-86	7	0-33	3	2-3
Pasture and Open (%)	5	0-23	1	0-8	2	1-2
Forest (%)	12	2-28	54	12-84	11	3-18
Water and Wetland (%)	19	5-33	33	14-61	12	5-18

Table 6. Land use percentage of each dominant land use groups for the lakesheds in NLAP study.

Total pesticide concentrations were summarized for the detected parent and degradate compounds and then placed into the dominant land use group of the lakeshed from which they were collected. Figure 6 presents the average total pesticide and average atrazine concentration values for each dominant land use groups.

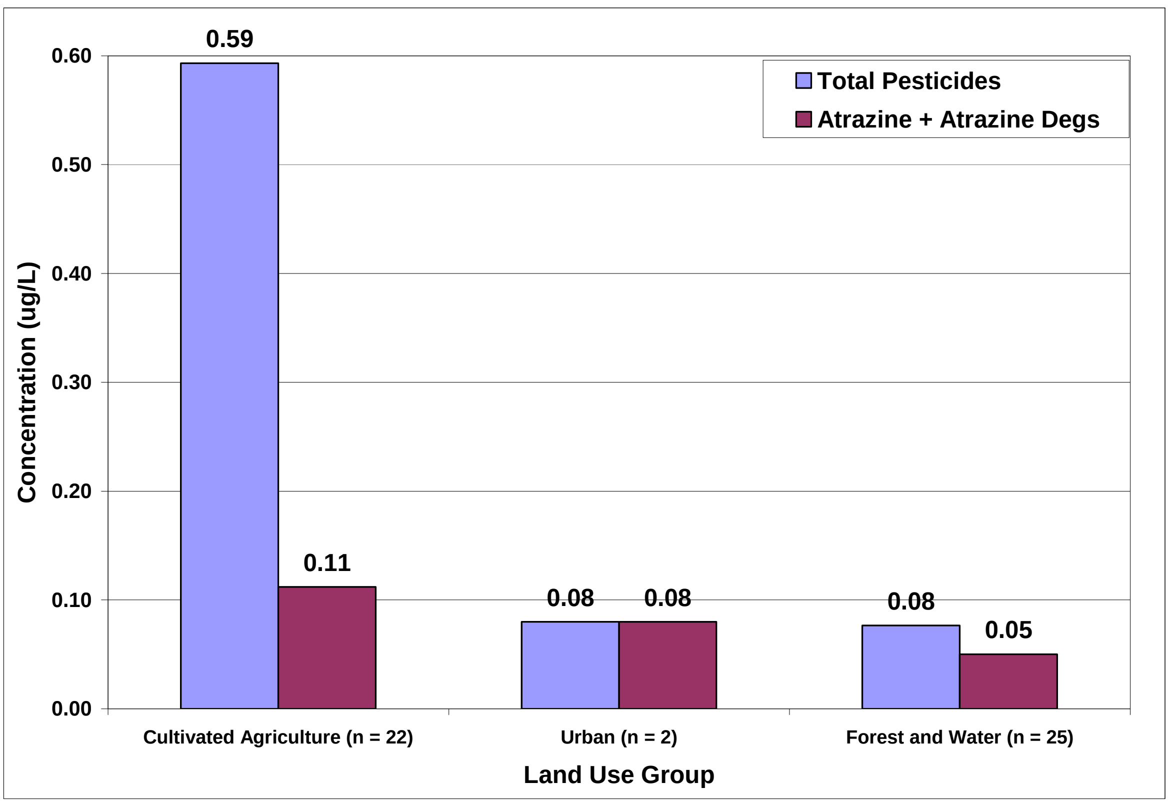

Figure 6. Average total pesticide (parent + degradate) and average atrazine concentration determined by the dominant land use in each lakeshed.

Lakes within lakesheds dominated by cultivated agriculture had substantially higher total pesticide concentrations than lakes within lakesheds dominated by urban and forest/water land use. This is generally consistent with the findings of a Wisconsin lake study that found increased concentrations of atrazine in lakes with greater than 75 percent row crop agricultural in the lakeshed (Allen, 2006).

Urban and forest/water dominated lakesheds had very similar concentrations of total pesticides. It is possible that the concentration averages presented for urban and forest/water land use groups are indicative of concentrations that might be expected as a result of atmospheric transport and deposition (Figure 6).

Conclusions

- Atrazine was detected in 87 percent of the 53 sampled lakes in Minnesota including lakes far from areas of assumed atrazine application.
- A pesticide or a pesticide degradate was detected in 91 percent of the samples collected from Minnesota lakes.
- In general, the ranges of concentrations detected as well as the frequency of detection were similar or lower when compared with other studies of lakes and reservoirs in the Midwest.
- Concentrations of all detected pesticides were well below the Minnesota aquatic life standards and other reference values used by the Minnesota Department of Agriculture.
- The concentration of atrazine was higher in samples collected from rivers then those measured in lakes located in the same Pesticide Monitoring Region (PMR).
- Lakes in lakesheds with row crop agriculture as a dominant land use had higher concentrations of total pesticides. This may be the result of direct runoff from adjacent lands or from greater atmospheric deposition due to closer proximity to areas of application.
- Atmospheric deposition is suspected as the primary method of transport in lakes where pesticides, primarily atrazine, are detected far from areas of application.

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