Wisconsin Groundwater Quality

Agricultural Chemicals in Wisconsin Groundwater

April 2008

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ARM PUB 180.qxd 03/08

AGRICULTURAL CHEMICALS IN WISCONSIN GROUNDWATER

FINAL REPORT
MARCH 2008

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This report is the result of a cooperative effort between three units of Wisconsin government. The Wisconsin Department of Agriculture, Trade and Consumer Protection (DATCP) was responsible for overall project management and laboratory analysis. The Bureau of Environmental & Occupational Health of the Wisconsin Department of Health and Family Services provided funding and supplemental water testing kits and will analyze information on water use by rural households. The Wisconsin Field Office of the National Agricultural Statistics Service developed survey procedures, collected water use data, and summarized lab results.

DATCP administers many water quality and agricultural chemical programs that are designed to protect Wisconsin's groundwater. This survey provides factual information on the chemical compounds found in water used by Wisconsin residents with private wells.

Special thanks to the residents who participated in the survey and the enumerators who collected the water samples and administered the questionnaires.


Rod Nilsestuen


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Abstract

Between January 2007 and June 2007, three hundred and ninety-eight private drinking water wells were sampled as part of a statewide survey of agricultural chemicals in Wisconsin groundwater. The purpose of the survey was to obtain a current picture of agricultural chemicals in groundwater and to compare the levels in the 2007 survey with levels found in earlier surveys conducted in 1994, 1996 and 2001. Wells were selected using a stratified random sampling procedure and were used to represent Wisconsin groundwater accessible by private wells. Samples were analyzed for 32 compounds including herbicides, herbicide metabolites, one insecticide, and nitrate-nitrogen.

Based on statistical analysis of the sample results, it was estimated that the proportion of wells in Wisconsin that contained a detectable level of a pesticide or pesticide metabolite was 33.5%. Areas of the state with a higher intensity of agriculture generally had higher frequencies of detections of pesticides and nitrate-nitrogen. The two most commonly detected pesticide compounds were the herbicide metabolites alachlor ESA and metolachlor ESA which each had a proportion estimate of 21.6 %.

The statewide estimate of the proportion of wells that contained atrazine total chlorinated residues (TCR) was 11.7%. The estimate of the proportion of wells that exceeded the 3 µg/l enforcement standard for TCR was 0.4%. Estimates of the mean detect concentrations for pesticides were generally less than 1.0 µg/l. The estimate of the proportion of wells that exceeded the 10 mg/l enforcement standard for nitrate-nitrogen was 9.0%.

Time trend analysis was performed to determine whether the proportion estimates for atrazine, TCR, nitrate-nitrogen, alachlor ESA and metolachlor ESA in private wells had changed between the 2001 survey and the 2007 survey. The results of this analysis did not show any statistically significant changes for these compounds over this time period.

Introduction

The Wisconsin Department of Agriculture, Trade and Consumer Protection conducted the Atrazine Rule Evaluation Survey in 1994 (Phase 1) and 1996 (Phase 2) (LeMasters and Baldock, 1997). These two surveys were an important part of the Department's evaluation of its regulations on the use of the herbicide atrazine. In 2000-2001, a third statewide survey was conducted to provide an update on agricultural chemicals in groundwater and to compare findings with the earlier surveys (Wisconsin Department of Agriculture, Trade and Consumer Protection, 2002).

The 2007 survey was a joint project between the Wisconsin Department of Agriculture, Trade and Consumer Protection, the National Agricultural Statistics Service (NASS) and the Department of Health and Family Services (DHFS). The specific objectives of the 2007 survey were 1) to establish the frequencies of detection and concentrations for agricultural chemicals (pesticides and nitrate-nitrogen) in rural drinking water wells in Wisconsin and 2) to determine if there have been measurable changes in pesticide compounds and nitrate-nitrogen levels in Wisconsin groundwater over time.

Each well sample was analyzed for 32 compounds including 17 pesticide parent compounds, 14 pesticide metabolites and nitrate-nitrogen. This is an expanded list compared to the 17 analytes included in the previous surveys. Of the 17 pesticide parent compounds, 16 are herbicides and one (chlorpyrifos) is an insecticide. These are the active ingredients in many commonly-used agricultural pesticide products in Wisconsin. All the metabolites are herbicide metabolites. These are related chemical compounds that are formed when the parent herbicide compounds break down in the soil and groundwater. Health standards have been established for 11 of the parent compounds and four of the metabolites.

The purpose of this report is to provide the results of the 2007 survey and to compare these results to earlier surveys. All four surveys were designed to allow for statistical comparisons.

Materials and Methods

Survey Design

The desired target population for the 1994, 1996, 2001, and 2007 surveys was Wisconsin groundwater. However, obtaining a representative sample of all Wisconsin groundwater is not easy due to its large three-dimensional extent across the state. In order to sample groundwater in an efficient manner, existing private drinking water wells were used. The actual target population for the four surveys can be best described as groundwater accessible by private wells.

Each survey used a 50 percent sample rotation scheme in which approximately half of the wells in the 1996, 2001, and 2007 surveys had been part of the previous survey and approximately half were newly selected. Wells that were tested for the first time in the 2001 survey were tested again in the 2007 survey. Wells that had been in both the 1996 and 2001 surveys were rotated out of the 2007 survey and a sample of new wells was selected. This rotation allowed for the potential to identify new areas of agricultural chemical detections within the state and for the use of statistical tests that can detect changes in pesticide levels over time.

The 2007 survey, along with the previous three surveys, used a stratified, random sampling procedure to allocate (select) samples throughout the state. The sample allocation procedure used in 2007 for the newly-selected wells utilized NASS land use strata, which are based on how intensively land in Wisconsin is cultivated for agricultural production. Each NASS stratum includes land areas falling into a specific range of intensity of cultivation. The land within each stratum is divided into "area segments" that are typically one square mile in size.

Since no comprehensive list of private wells exists, samples were allocated by randomly selecting a predetermined number of area segments within each agricultural stratum. Strata for entirely urban, non-agricultural, and water-covered areas were excluded from sampling. Since area segment boundaries are typically roads, office staff chose a starting corner in each segment and the groundwater samplers were instructed to travel clockwise within the segment until they found a well owner willing to participate in the survey. In a few sparsely-populated segments, the samplers had to contact a well owner in an adjoining segment in order to collect a sample.

All previous surveys also used a stratified, random sampling procedure to allocate samples, but the strata in these earlier surveys were the nine NASS Agricultural Statistics Districts, which are groups of adjoining counties. The number of samples collected in each of the nine districts was based on the number of acres in farms in each district. Samples were allocated by selecting a random sample list of civil sections in each district (excluding those covered by water or publicly owned). In each civil section, a random 10-acre parcel was selected and the well nearest its center was identified to represent the groundwater of the civil section.

The 2007 stratification method offered several benefits over the previously used method. First, samples were allocated proportional to agricultural intensity throughout the state. Second, the new method allowed for comparisons of water quality to agricultural intensity in addition to location within the state.

In order to compare the frequencies of detections of agricultural chemicals over time, GIS software was used to restratify the results of the 2001 survey into the NASS strata. This re-stratification allowed the 2001 survey data to be appropriately weighted so that the 2001 data could be compared to the 2007 data.

Sample Collection and Analysis

For the 2007 well water survey, 398 samples were collected from private drinking wells throughout Wisconsin. Figure 1 shows the location of the NASS strata (land use categories) used in the 2007 survey, the wells sampled in 2007, and the boundaries of the nine NASS Agricultural Statistics Districts, which were the strata in the previous three statewide surveys.

One hundred eighty-eight water samples were collected from wells that were first tested in the 2001 survey. Water samples were only obtained from wells that had not had any structural changes since the last survey. This was to ensure that water samples were collected from the same location in the aquifer as the previous survey in order to make comparisons valid.

Two hundred ten water samples were taken from newly-selected wells that replaced those rotated out of the 2001 survey. Once a new well was selected, the samplers interviewed the owner to obtain well information and inspected the plumbing system to determine if there was a water treatment device. Samples were only collected if untreated raw water could be obtained. If a groundwater sampler was not able to get an untreated sample from a well, another well was selected using the process described above.

Samples were collected through a cold water supply after running the water for approximately five minutes. Four one-liter amber glass bottles with Teflon-lined caps were filled at each site and promptly placed in an insulated box with ice. Sample collection records were completed and bottles were sealed to maintain sample integrity through delivery to the DATCP laboratory.

Each water sample was analyzed for the following compounds at the DATCP laboratory:

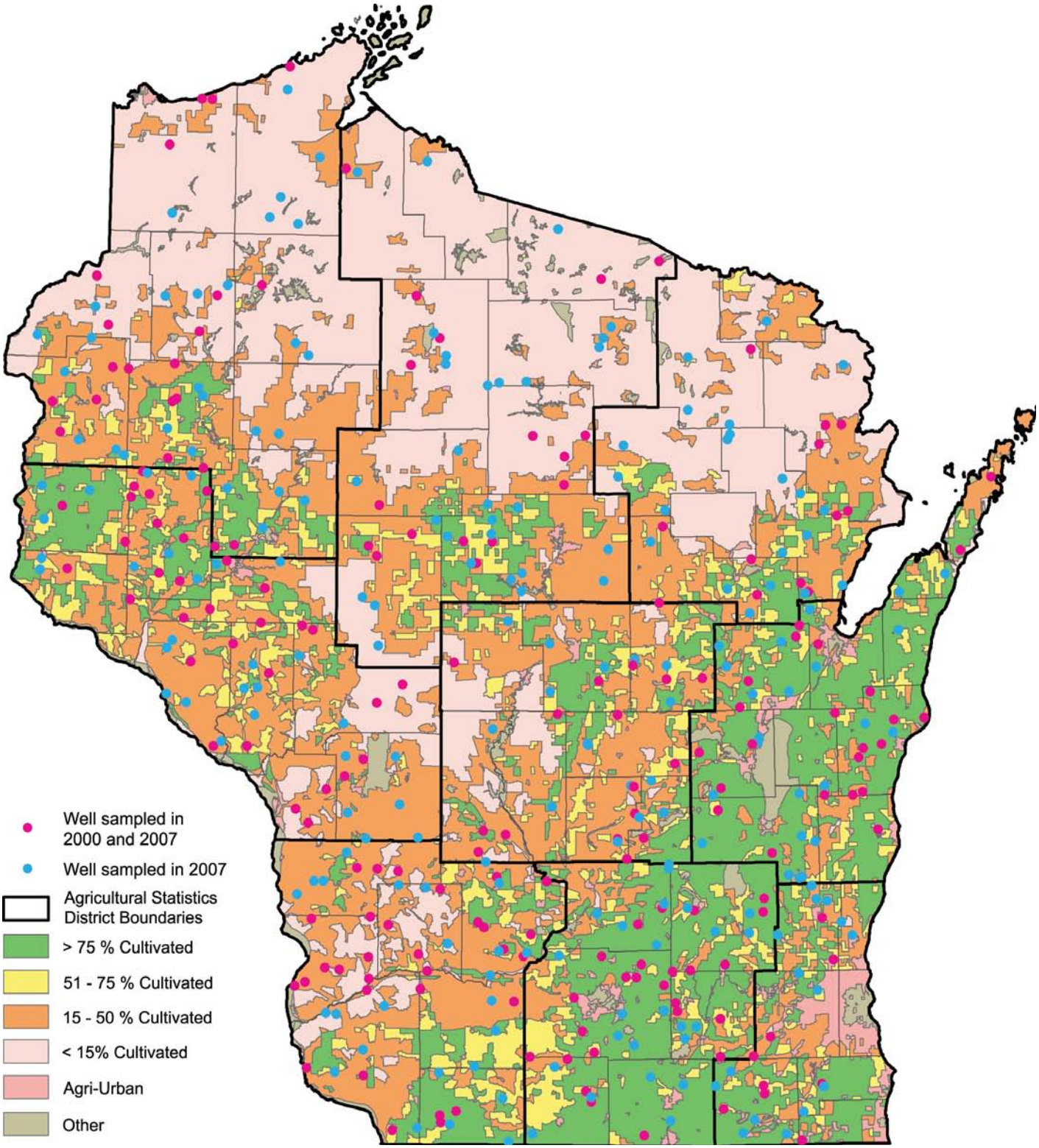
- Atrazine and its metabolites deethyl atrazine, deisopropyl atrazine and diamino atrazine (the sum of these four compounds is referred to as total chlorinated residues of atrazine or TCR)
- Alachlor, metolachlor and acetochlor and their ESA and OA metabolites
- Cyanazine
- Metribuzin
- Simazine
- Nitrate-nitrogen
- Glyphosate* and its AMPA metabolite*
- Mesotrione/mesotrione MNBA* and mesotrione AMBA*
- Dimethenamid* and its ESA* and OA* metabolites
- Prometone*
- EPTC*
- Pendimethalin*
- Chlorpyrifos*
- Bentazon*
- Clopyralid*
- 2,4-D*
- Dicamba*

* new analytes in 2007 not included in previous surveys (15 total)

For each analyte a limit of detection (LOD) and a limit of quantitation (LOQ) were established. Results below the LOD were considered to be non-detects. Results above the LOQ were quantified and presented as numerical values. Results between the LOD and LOQ were considered to be detects but were not quantified or presented as numerical values.

Figure 1

Sampling Locations and Land Use Categories for the 2007 Survey.



Results of the 2007 Survey

Pesticide and Nitrate-Nitrogen Detections

Table 1 shows the results of the 2007 survey. One hundred fifty-eight of the 398 samples contained a detectable concentration of one or more pesticides or pesticide metabolites. The most commonly detected herbicide compounds were alachlor ESA (100 detects), metolachlor ESA (106 detects), and atrazine total chlorinated residues or TCR (55 detects). Figures 2-4 show the geographic distribution of the results for these three parameters.

Two of the 55 samples that contained detectable residues of TCR exceeded the Wisconsin groundwater enforcement standard of 3 micrograms per liter (µg/l) (parts per billion). No samples exceeded the alachlor ESA enforcement standard of 20 ug/l. A standard has not been established for metolachlor ESA.

Nitrate-nitrogen was detected in 234 of the 398 samples at concentrations ranging from 0.52 milligrams per liter (mg/l) (parts per million) to 81.1 mg/l. Forty-seven of the samples exceeded the nitrate-nitrogen enforcement standard of 10 mg/l. Figure 5 is a map showing the geographic distribution of the nitrate-nitrogen results.

Table 1

Results of the 2007 Survey.

Compound	Number of detects	Limit of detection* (µg/l)	Limit of quantitation* (µg/l)	Groundwater enforcement standard (µg/l)	Groundwater samples over standard	Concentration range** (µg/l)
atrazine	22	0.046	0.15			0.15 - 1.04
deethyl atrazine	40	0.058	0.3			0.31 - 2.08
deisopropyl atrazine	9	0.07	0.3			0.33 - 0.51
diamino atrazine	28	0.12	0.5			0.53 - 1.39
TCR	55	#	#	3	2	0.16 - 3.66
alachlor	1	0.082	0.3	2		0.36
alachlor ESA	100	0.044	0.14	20		0.14 - 8.35
alachlor OA	13	0.034	0.11			0.15 - 1.33
metolachlor	0	0.067	0.25	15		
metolachlor ESA	106	0.045	0.14			0.14 - 6.54
metolachlor OA	18	0.057	0.18			0.30 - 1.37
acetochlor	0	0.03	0.1			
acetochlor ESA	16	0.064	0.2			0.23 - 2.32
acetochlor OA	3	0.038	0.12			4.36
metribuzin	0	0.03	0.1	250		
simazine	0	0.038	0.15	4		
mesotrione and MNBA	0	0.016	0.052	3		
mesotrione AMBA	0	0.018	0.06			
glyphosate	0	0.65	2.2			
glyphosate AMPA	0	0.022	0.072			
bentazon	2	0.057	0.18	300		0.18
chlorpyrifos	0	0.054	0.2			
clopyralid	1	0.4	1.3			
cyanazine	0	0.18	0.6	1		
dicamba	0	0.12	0.41	300		
dimethenamid	0	0.022	0.1			
dimethenamid ESA	1	0.057	0.19			0.205
dimethenamid OA	0	0.05	0.17			
EPTC	0	0.22	0.75	250		
pendimethalin	0	0.039	0.15			
prometone	0	0.027	0.1	90		
2,4-D	2	0.13	0.43	70		4.95
nitrate-nitrogen***	234	0.5	0.5	10	47	0.52 - 81.1

* LODs and LOQs are empirically derived statistical parameters. The LODs and LOQs noted are the lowest derived value for each target compound. Due to nominal differences in instrument sensitivity and sample size, a small number of samples have LOD and LOQ values slightly above those noted.

** quantifiable concentration

TCR is the sum of four analytes and does not have a LOD or LOQ

*** nitrate-nitrogen values are in mg/l

Figure 2

Alachlor ESA and Alachlor OA Results from the 2007 Survey.

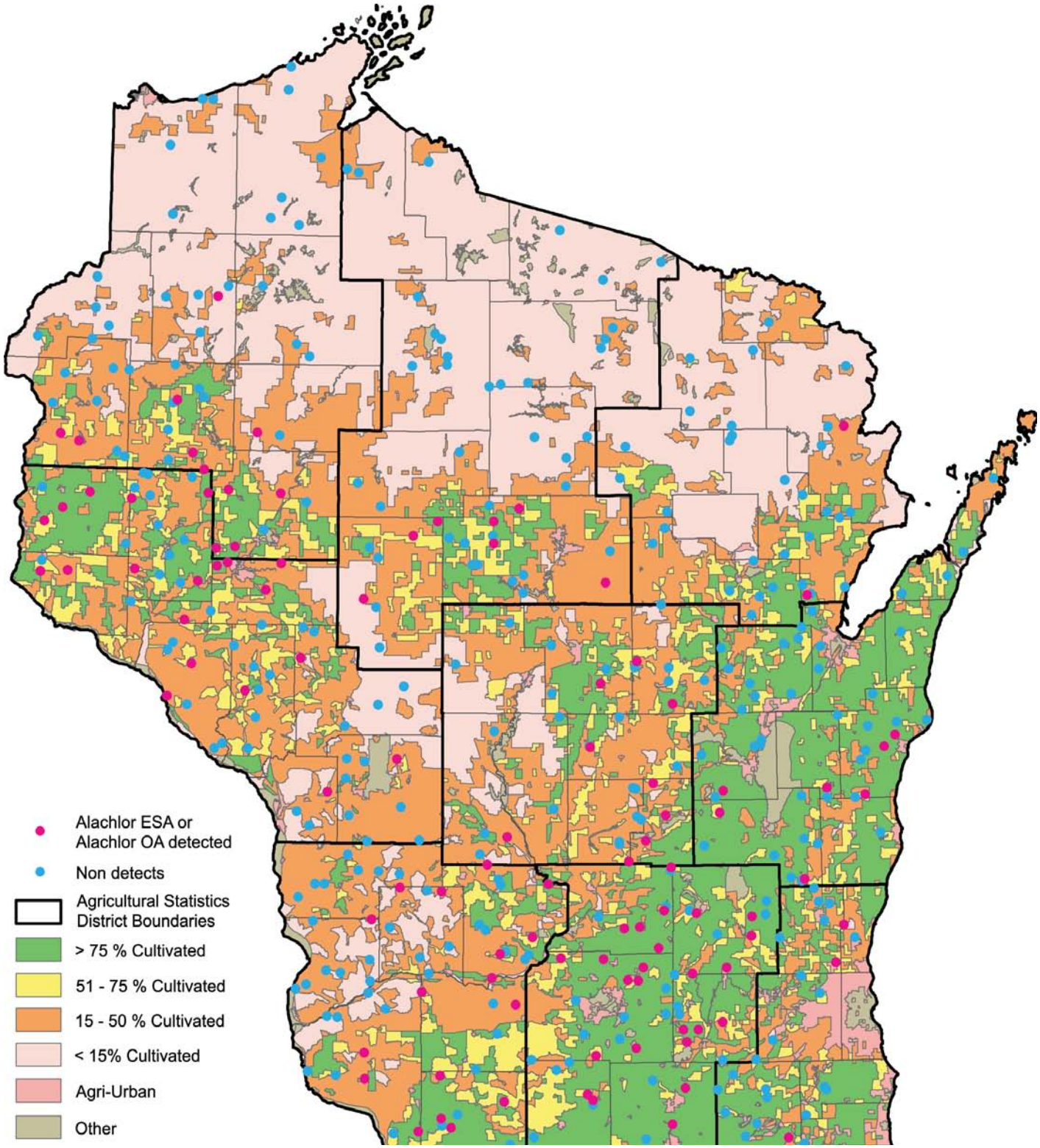


Figure 3

Metolachlor ESA and Metolachlor OA Results from the 2007 Survey.

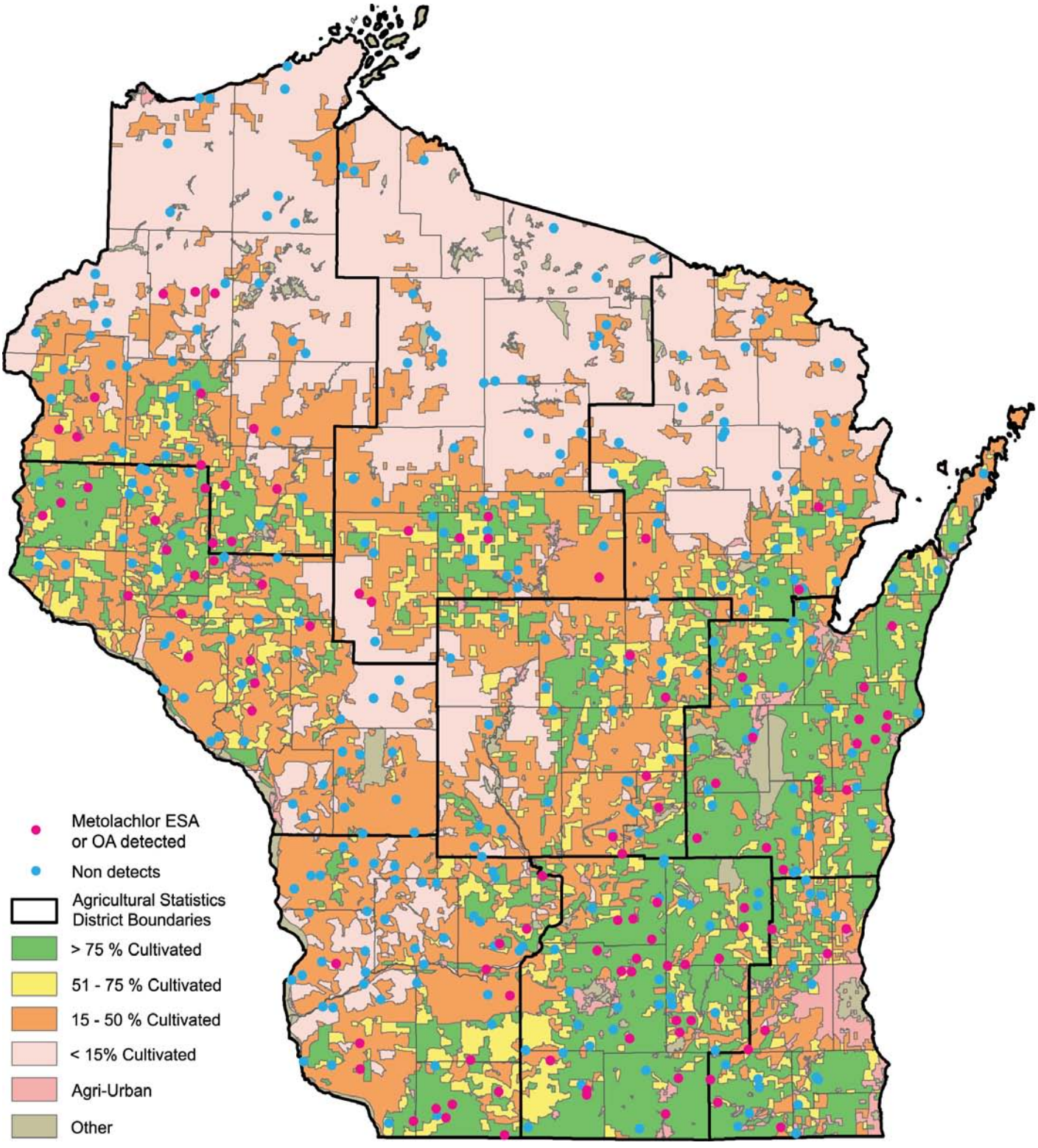


Figure 4

Atrazine TCR Results from the 2007 Survey.

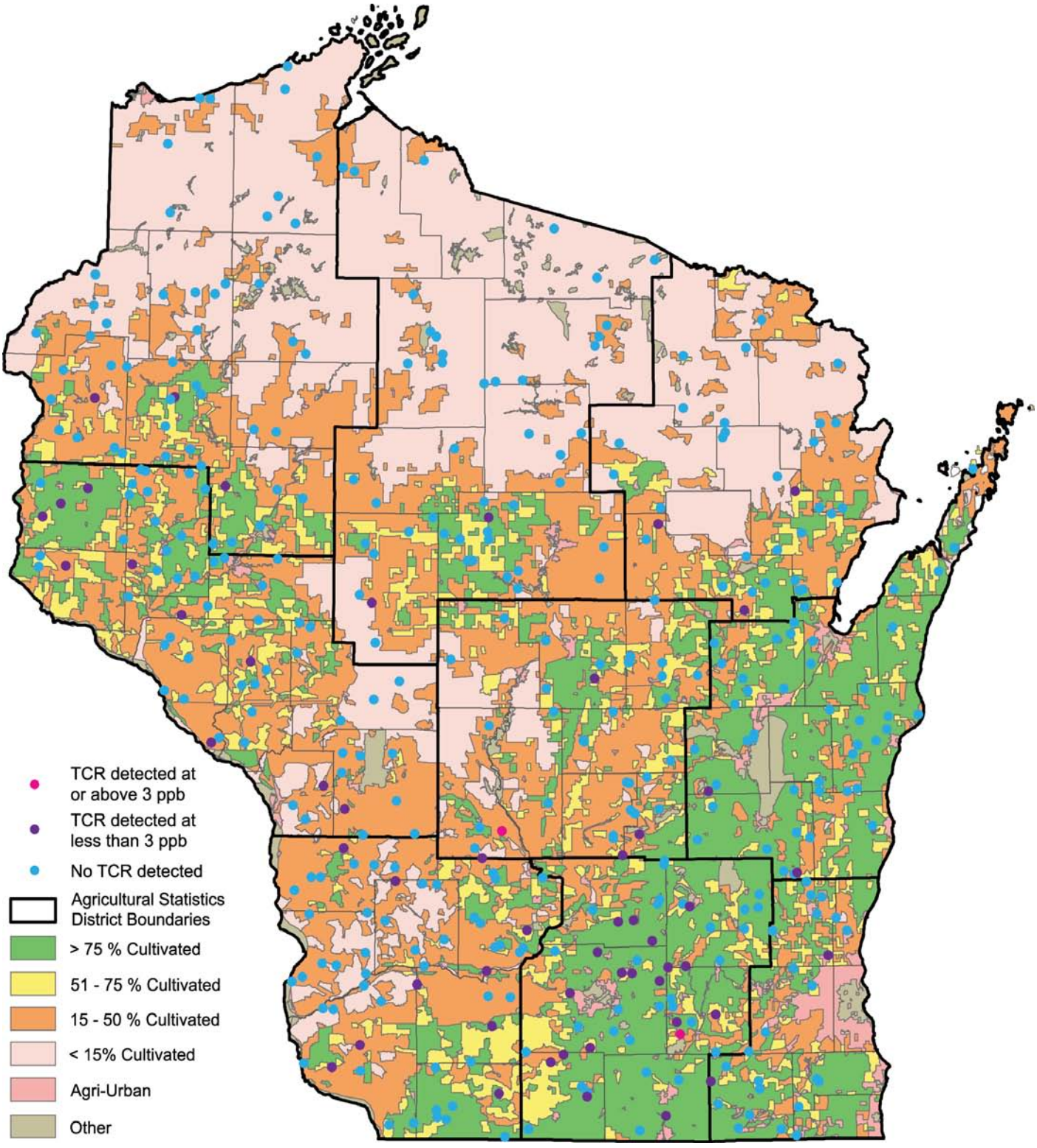
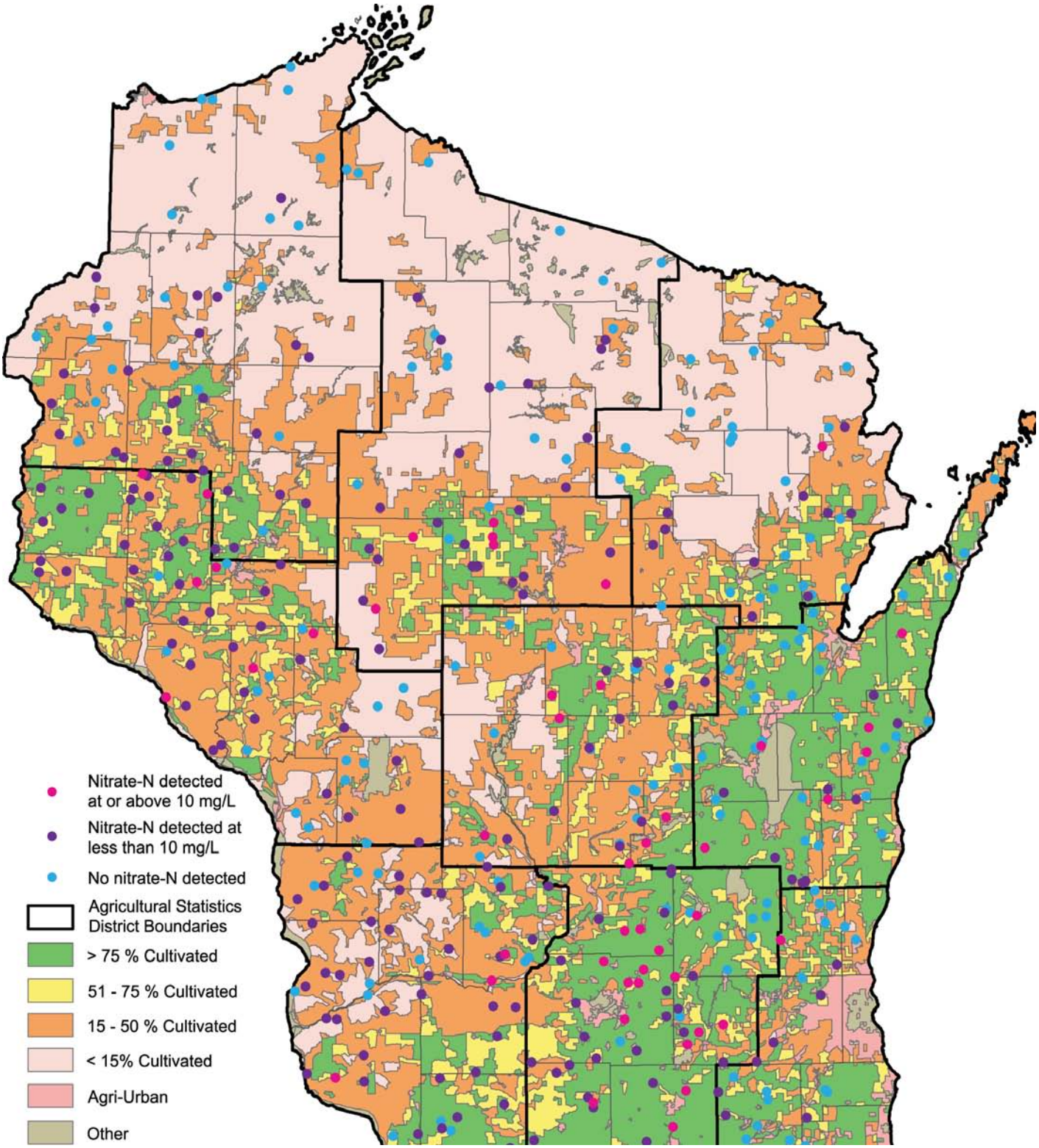


Figure 5

Nitrate-Nitrogen Results from the 2007 Survey.



Detection Frequencies in the Strata

Table 2a shows the number of detects and Table 2b shows the percentage of detects in the NASS strata for the most commonly detected compounds in the 2007 survey. Table 2c shows the number of detects in the NASS Agricultural Statistics Districts which were the strata for the three previous statewide surveys. In each table, the number of samples per stratum varies because of the stratified sampling design.

Table 2a

Number of Detects* by NASS Strata and Parameter in the 2007 Survey.

NASS Strata	Strata Description	Number of Samples	Number of Detects					
			Atrazine	TCR	Alachlor ESA	Metolachlor ESA	Nitrate-N	Nitrate-N>10 mg/l
11	>75% Cultivated	134	7	23	48	62	84	28
12	51-75% Cultivated	50	1	10	14	11	30	5
20	15-50% Cultivated	150	11	19	30	27	91	13
40	<15% Cultivated	59	3	3	7	6	28	1
31	Agri-Urban	5	0	0	1	0	1	0
Total		**398**	**22**	**55**	**100**	**106**	**234**	**47**

* quantifiable and non-quantifiable detects

Table 2b

Percentage of Detects* by NASS Strata** and Parameter in the 2007 Survey.

NASS Strata	Strata Description	Number of Samples	Percentage of Detects					
			Atrazine	TCR	Alachlor ESA	Metolachlor ESA	Nitrate-N	Nitrate-N>10 mg/l
11	>75% Cultivated	134	5.2	17	36	46	63	21
12	51-75% Cultivated	50	2.0	20	28	22	60	10
20	15-50% Cultivated	150	7.3	13	20	18	61	8.6
40	<15% Cultivated	59	5.1	5.1	12	10	47	1.7

* quantifiable and non-quantifiable detects

** the percentages for the Agri-Urban stratum are not included because of the small number of samples

Table 2c

Number of Detects* by NASS Agricultural Statistics District and Parameter in the 2007 Survey.

NASS District	Number of Samples	Number of Detects					
		Atrazine	TCR	Alachlor ESA	Metolachlor ESA	Nitrate-N	Nitrate-N >10 mg/l
NW	50	1	3	11	13	27	0
NC	46	2	2	7	7	29	6
NE	32	0	3	2	3	11	1
WC	61	7	10	20	16	46	7
CE	33	3	5	10	6	19	7
EC	48	0	2	7	16	15	6
SW	55	5	9	15	15	40	3
SC	50	4	19	25	23	40	16
SE	23	0	2	3	7	7	1
Total	**398**	**22**	**55**	**100**	**106**	**234**	**47**

* quantifiable and non-quantifiable detects

Table 2b shows that in 2007 there was generally a pattern of higher frequencies of detections in strata with higher percentages of cultivated land. Alachlor ESA and metolachlor ESA, for example, were detected in 36% and 46% of the wells, respectively, in stratum 11 which has greater than 75% cultivated land. Only 12% and 10% of wells in stratum 40 (less than 15% cultivated land) contained these two compounds. Twenty-one percent of wells in stratum 11 exceeded the 10 mg/l health standard for nitrate-nitrogen, whereas 1.7% of the wells exceeded this standard in strata 40. Table 2c shows that, as in the 2001 survey, the South Central NASS Agricultural Statistics District (Columbia, Dodge, Dane, Jefferson, Green, and Rock Counties) had the highest number of detects for most compounds.

Statewide Statistical Estimates of the Proportion of Detections

Using the results from each stratum and the methods described by Cochran (1977) and Thomson (1992), statewide estimates of the proportions of detections were calculated for eleven parameters. These estimates apply to rural Wisconsin groundwater accessible by private wells. Table 3 shows these estimates and their 95% confidence intervals. (The proportion estimates in Table 3 include the non-quantifiable detects between the LOD and LOQ.) Similar to the 2001 survey, alachlor ESA and metolachlor ESA had the highest proportion estimates for pesticide compounds. The estimate of the proportion of wells that exceeded the 10 mg/l health standard for nitrate-nitrogen is 9.0%

Table 3

Statewide Estimates of the Proportion of Detections and 95% Confidence Intervals for Eleven Parameters in the 2007 Survey.

Parameter	Statewide number of detects*	Statewide estimate of the proportion of detects (%)	95% Confidence Interval (%)
any pesticide or metabolite	158	33.5	28.6 - 38.3
TCR	55	11.7	8.5 - 14.8
TCR>3.0 µg/l	2	0.4	**
atrazine	22	5.4	2.9 - 8.0
alachlor ESA***	100	21.6	17.2 - 26.0
alachlor OA	13	2.4	1.0 - 3.9
acetochlor ESA	16	3.1	1.4 - 4.8
metolachlor ESA	106	21.6	17.7 - 25.6
metolachlor OA	18	3.6	1.8 - 5.4
nitrate-nitrogen	234	56.0	50.3 - 61.5
nitrate-nitrogen>10 mg/l	47	9.0	6.5 - 11.6

* quantifiable and non-quantifiable detects
** not enough data points to calculate a confidence interval
*** there were no detections of alachlor ESA over the 20 µg/l groundwater enforcement standard

Concentrations

We also estimated average concentrations for nine parameters. These estimates are based on detectable levels of these parameters. Non-quantifiable detects (detects between the LOD and LOQ) were assigned a value of LOQ/square root of 2 (Helsel, 2005). If wells without detections had been included, the statewide average concentration estimates would be different. Table 4 shows these estimates and their 95% confidence intervals. The estimates of mean detect concentrations for pesticides ranged from 0.20 µg/l for metolachlor OA to 1.00 µg/l for alachlor ESA.

Table 4

Estimates of the Mean Concentration of Detects and 95% Confidence Intervals for Nine Parameters in the 2007 Survey.

Parameter	Statewide number of detects	Statewide estimate of the mean detect concentration (µg/l)	95% confidence interval (µg/l)	Enforcement Standard (µg/l)
TCR	55	0.67	0.12 -1.21	3
atrazine	22	0.22	0.13 - 0.32	
alachlor ESA	100	1.00	0.53 - 1.48	20
alachlor OA	13	0.30	0.00 - 0.87	
acetochlor ESA	16	0.74	0.55 - 0.93	
acetochlor OA	3	0.57	0.00 - 10.45	
metolachlor ESA	106	0.47	0.29 - 0.65	
metolachlor OA	18	0.20	0.00 - 0.50	
nitrate-nitrogen*	234	5.64	4.83 - 6.46	10

*nitrate-nitrogen values are in mg/l

Results for the Expanded List of Analytes in 2007

Fifteen additional analytes were included in 2007 compared to previous surveys. These additional analytes (see Materials and Methods section) were included in 2007 because of increased use of the parent compounds in Wisconsin, new information suggesting potential leaching potential, or improved laboratory capability to analyze for these compounds.

Of these 15 analytes, only four were detected in the 398 wells included in the 2007 survey. Table 5 shows the results for these four compounds. Based on the small number and low concentrations of detects and the considerable increase in laboratory costs, it is unlikely that these 15 compounds will routinely be included in future surveys.

Table 5

Results for the Expanded List of Analytes in the 2007 Survey.

Compound	Number of Detects	Concentrations (µg/l)
2,4-D	2	non-quantifiable, 4.95
bentazon	2	non-quantifiable, 0.18
clopyralid	1	non-quantifiable
dimethanamid ESA	1	0.205

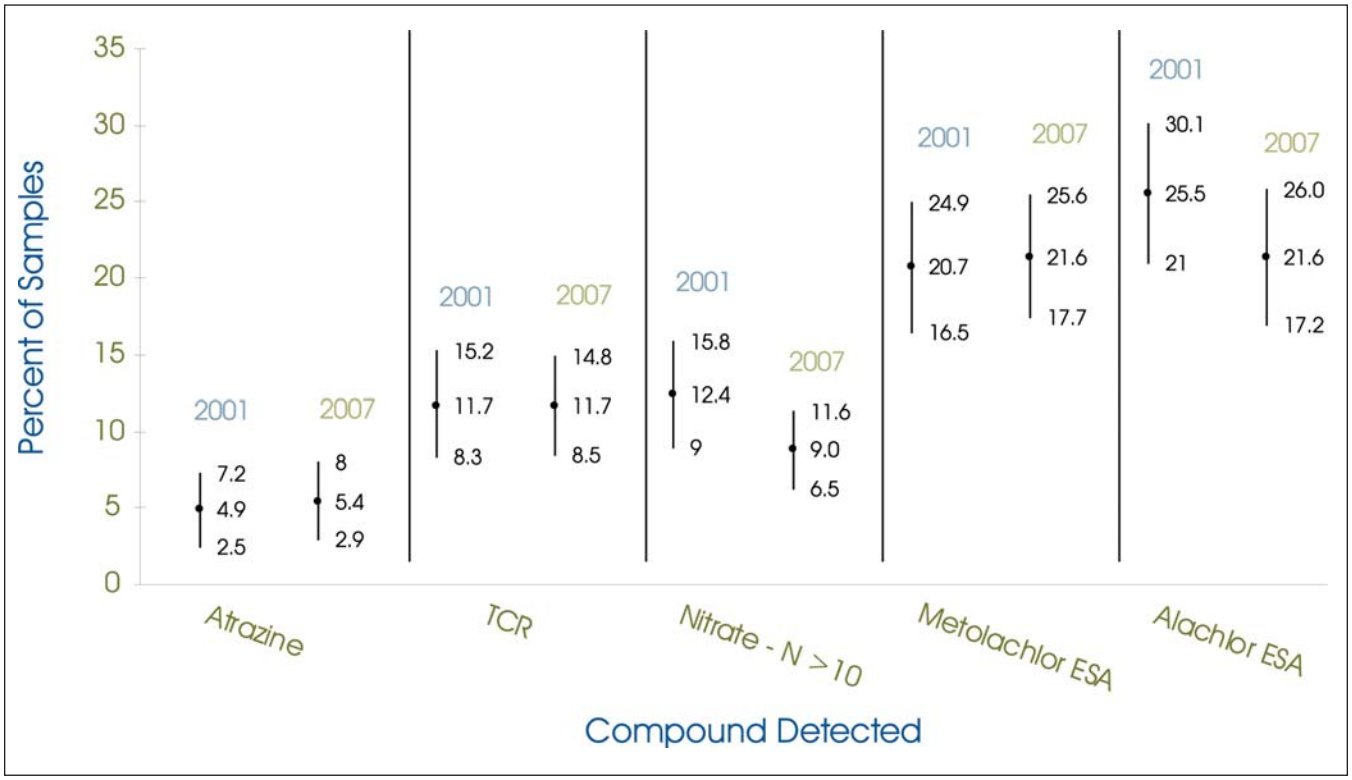
Comparing Results between Surveys

The estimates of the proportion of detects and the respective 95% confidence intervals for atrazine, TCR, and nitrate-nitrogen over 10 mg/l, metolachlor ESA, and alachlor ESA were compared to see if there were any statistically significant changes between 2001 and 2007. The results from 2001 and 2007 were chosen to allow for time trend analysis for alachlor ESA and metolachlor ESA (comparable lab methods for these compounds did not exist in 1994 and 1996).

Figure 6 shows that there were no statistically significant changes (all confidence intervals overlap) for the proportion of wells containing these five parameters. Previous analysis showed that the proportion of wells with a detection of parent atrazine had a statistically significant decline between 1994 and 2001 (WDATCP, 2002).

Figure 6

Comparison of proportion estimates and 95% Confidence Intervals for 2001 and 2007.



Relationship between Well Characteristics and Frequencies of Detection for Selected Compounds

As part of the 2007 survey, each participating household was asked to provide information about their well and various aspects of their use of the water supplied by the well on a questionnaire developed by DHFS and NASS. The purpose of the information generated by these questions was to estimate the potential exposure of well users to agricultural chemicals in well water. The findings from these questions will be presented in a separate report by DHFS.

As part of the questionnaire, DATCP included two questions on well age and well depth.

For well age, each respondent was asked if the well was under six years old, six to 20 years old, or over 20 years old. For well depth, respondents were asked if the well was less than 50 feet deep, 50-150 feet deep, or over 150 feet deep. This information was used to evaluate the relationships between well characteristics (age and depth) and detection frequencies for selected agricultural chemicals. Not all respondents were able to provide the age and depth of their well and those who did generally answered based on their memory.

Table 6a shows the percentage of detections by well age. The majority of wells in the survey fell in the greater than 20 year old range. Noticeable trends are higher percentages of detections of alachlor ESA and nitrate-nitrogen with increasing well age. The older wells also had a higher percentage of nitrate-nitrogen over the 10 mg/l health standard.

Table 6b shows the percentage of detections by well depth. The majority of wells in the survey were in the 50-150 feet depth range. The shallower wells had a higher percentage of detections of nitrate-nitrogen and a higher percentage of wells with nitrate-nitrogen over the 10 mg/l health standard.

Table 6a

Percentage of Detection for Selected Compounds, by Well Age.

Well Age	Number of Samples*	Percentage of Detects					
		Atrazine	TCR	Alachlor ESA	Metolachlor ESA	Nitrate-N	Nitrate-N>10 mg/l
<6	31	6	16	19	29	42	6
6-20	87	7	17	20	21	48	9
>20	222	5	11	28	28	64	11

* 340 respondents knew the age of their well

Table 6b

Percentage of Detection for Selected Compounds, by Well Depth.

Well Depth	Number of Samples*	Percentage of Detects					
		Atrazine	TCR	Alachlor ESA	Metolachlor ESA	Nitrate-N	Nitrate-N>10 mg/l
<50	42	10	17	24	26	71	24
50-150	149	3	11	30	31	64	8
>150	89	8	24	22	17	44	7

* 280 respondents knew the depth of their well

Summary

- The statewide estimates of the proportion of wells containing atrazine, atrazine TCR, nitrate-nitrogen over 10 mg/l, metolachlor ESA and alachlor ESA did not show statistically-significant changes between 2001 and 2007.
- The estimate for the proportion of wells that exceeded the 10 mg/l enforcement standard for nitrate-nitrogen was 9.0%.
- The statewide estimate of the proportion of wells that contained a detectable level of a pesticide or pesticide metabolite was 33.5%.
- Alachlor ESA and metolachlor ESA were the most commonly detected herbicide compounds with identical proportion estimates of 21.6%.
- The statewide estimate of the proportion of wells that contained atrazine TCR was 11.7%.
- The estimate for the proportion of wells that exceeded the 3 ug/l enforcement standard for atrazine TCR was 0.4%.

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Notes