

US EPA ARCHIVE DOCUMENT

# TEXT SEARCHABLE DOCUMENT

## Data Evaluation Report of Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46490304

---

**Test material:** Fipronil

**IUPAC name:** 5-amino-1-(2,6-dichloro- $\alpha,\alpha,\alpha$ -trifluoro-*p*-tolyl)-4-trifluoromethylsulfinylpyrazole-3-carbonitrile

**CAS name:** 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)sulfinyl]-1*H*-pyrazole-3-carbonitrile

**Primary Reviewer:** James Hetrick, Ph.D.  
EPA

Signature:

Date:

*James A. Hetrick*

**Secondary Reviewer:** Thuy Nguyen  
EPA

Signature:

Date:

5/5/08  
*Thuy Nguyen*  
5/6/08

**EPA PC Code:** 129121

**CITATION:** Daussinm, S. 2004. Chipco Topchoice® Granular Insecticide: Fipronil Surface Water Monitoring Study at a Recreational Pond Setting in Ocala, FL. Sponsored by Bayer Crop Science, RTP, NC. Performed by Bayer Crop Science, Stillwell, KS and AgVise Laboratories, Northward, ND. MRID 46490304.

US EPA ARCHIVE DOCUMENT



2080446

# Data Evaluation Report of Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46490304

---

## EXECUTIVE SUMMARY:

The fipronil water monitoring study (MRID 46490304) provides supplemental data on the runoff potential of fipronil and its degradation products (MB46136, MB46513, and MB 46950) and its impact on fipronil residue occurrence in surface water from use of Chipco Topchoice® on a recreational pond setting in Ocala, FL. This study was submitted to fulfill a condition of registration regarding runoff concerns of fipronil residues from broadcast use of fipronil for control of fire ants. The registrant did not provide any concurrent biological monitoring of the aquatic environment to assess the impact of fipronil and its degradation products on aquatic invertebrates.

The data are deemed supplemental because there is an insufficient description of the storage stability study. To upgrade the study, the registrant should provide a complete description of the storage stability study.

The fipronil application area accounts for 3.32 acres of turf around the catchment pond in Ocala, FL. Chipco Topchoice® insecticide was broadcast applied at a rate of 0.0122 lbs ai/A on July 16, 2002. The maximum label application rate is 0.0125 lbs ai/A. Duplicate grab samples of water were taken at day 0, 1, 2, 3 days, every 10 days from day 10 to 100, every 20 days from day 120 to 180, and after each rainfall event of  $\geq 1.5$  inches within a 24 hour period. The fipronil degradation product MB46513 was detected ( $< 0.010 \mu\text{g/L}$ ) in a 20 day post-treatment sample of pond water. No other fipronil residues were detected in pond water samples. Sediment samples were not collected.

## I. MATERIALS AND METHODS

**GUIDELINE FOLLOWED:** The SETAC-Europe: Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides (March 1995; pp. 1, 34) is not applicable.

**COMPLIANCE:** This study was conducted in compliance with USEPA FIFRA Good Laboratory Practices (40 CFR Part 160), which are consistent with the OECD Principles of GLP (p. 3). Signed and dated GLP, Data Confidentiality, Quality Assurance, and Certificate of Authenticity statements were provided (pp. 2-3, 5-6).

### A. MATERIALS:

# Data Evaluation Report of Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46490304

---

The objective of this study is to assess the runoff potential of fipronil and its degradation products into aquatic environments from recreational turf use of Chipco Topchoice® Insecticide for control of fire ants.

## 1. Study Description

The runoff monitoring study was conducted on recreational turf area surrounding a small pond in Ocala (Marion County), FL (pp 43). The catchment area of the site is 25 acres. The slope of the catchment area ranges from 2 to 6%. The site was selected because the turf area the pond could be treated with fipronil as well as the site location was in Florida. The catchment area included a highway, road, trees, and pasture area for horses around the pond. The pond has a surface area of 0.8 acres.

The soil in the catchment area is classified as an Arredondo sand (loamy, siliceous, Hyperthermic Grossarenic Paleudults). Soil physicochemical properties for surface samples (0-6 inches) are shown in **Table II, pp 59**. The soil is classified as an Group A hydrologic soil. The A soil hydrology classification indicates low runoff potential due to high soil permeability.

Grab water samples were taken at four locations (Points A, B, C, D) in the pond (pp 43). A characterization of water quality is shown on Table 1 (pp. 58). Duplicate grab samples of water were taken at day 0, 1, 2, 3 days, every 10 days from day 10 to 100, every 20 days from day 120 to 180, and after each rainfall event of  $\geq 1.5$  inches within a 24 hour period. Rainfall triggered sampling was conducted on day 47, 71, 147, 151, 162, and 170. Water samples were stored frozen until chemical analysis. A rain gauge was used to collect on-site precipitation. On site precipitation accounted for 28.60 inches of rainfall or ~91% of the 30 year average of normal precipitation (Table III, pp 20).

## 2. Site Preparation and Maintenance

The test site had well maintained turf. The turf was mowed during the study. The test site was not amended with any chemicals, fertilizers, or pesticides during the study.

## 3. Pesticide Application

The fipronil application area accounts for 3.32 acres of turf around the catchment pond. Chipco Topchoice® insecticide was broadcast applied at a rate of 0.0122 lbs ai/A on July 16, 2002. The maximum label application rate is 0.0125 lbs ai/A. Applications were made using calibrated drop spreaders. A 20-foot buffer was maintained around the pond. Additionally, a 15 foot buffer was maintained around a highway drainage culvert in the treated area.

## Data Evaluation Report of Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46490304

---

### 4. Analytical

A total of 88 water samples were analyzed for fipronil residues. Water samples were stored frozen for maximum time period of 350 days (**Appendix F, pp 68**). Storage stability data is referenced in monitoring study (MRID 46733902). The storage stability data indicate fipronil, MB46513, MB45950, and MB46136 were stable during a 25 month storage period. [The reviewer notes there is an incomplete description of the storage stability study in the Texas runoff study (MRID 46733902)].

Residues of fipronil in water samples were analyzed using a LC/MS/MS method entitled Insecticides, Fipronil: Method of Analysis for Possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Water- Revisions 4 and 5 (May 21, 2002). This method has method detection limit (MDL) of 0.004  $\mu\text{g/L}$  and limit of quantification (LOQ) of 0.010  $\mu\text{g/L}$ . (Reviewer Note: The method procedure requires filtration for cloudy extracts. The extracts are filtered through a nylon filtration disk after an acetonitrile extraction of surface water.

Procedural method verification in HPCLwaters, at 10 and 100 ng/L, showed average recoveries of  $92.5 \pm 9$  for fipronil (n=3),  $92 \pm 12$  for MB46513 (n=3),  $91 \pm 11$  for MB45950 (n=3), and  $91 \pm 10$  for MB46136 (n=3) (Table V, pp 22). In pond water, average recoveries were  $95 \pm 11$  for fipronil (n=10),  $96 \pm 9$  for MB46513 (n=10),  $94 \pm 9$  for MB45950 (n=10), and  $94 \pm 9$  for MB46136 (n=10) (Table VI, pp 22).

Method verification was conducted using pond water at the 0.010  $\mu\text{g/L}$  and 0.100  $\mu\text{g/L}$ . Average recoveries were  $94\% \pm 14$  for fipronil (n=6),  $100\% \pm 12$  for MB46513 (n=6),  $98\% \pm 11$  for MB45950 (n=6), and  $106\% \pm 16$  for MB46136 (n=6) (Table IV, pp 21). Field spike fortifications were conducted in pond water at the 0.10 and 1.00  $\mu\text{g/L}$ . Recoveries ranged 101 to 114% for fipronil, 97 to 102% for MB46513, 95 to 100% for MB45950, and 96 to 108 % for MB46136 (Table VII, pp 23).

## B. REPORTED RESULTS

### 1. Fipronil Residues in Pond Water

Fipronil degradation product MB46513 was detected in a 20 day post-treatment sample. (Table VIII, pp 27-29). The concentration of MB46513 was  $< 0.010 \mu\text{g/L}$ . No other fipronil residues were detected in pond water samples.

## Data Evaluation Report of Water Monitoring Study

PMRA Submission Number {.....}

EPA MRID Number 46490304

---

### C. REVIEWER COMMENTS

1. The registrant referenced a storage stability study from a Texas runoff study (MRID 46733902). The Texas runoff study does not provide a detailed description of the storage stability study.
2. The fipronil water monitoring study (MRID 46490304) provides supplemental data on the runoff potential of fipronil and its degradation products (MB46136, MB46513, and MB 46950) and its impact on fipronil residue occurrence in surface water from use of Chipco Topchoice® on turf in Ocala, FL. This study was submitted to fulfill a condition of registration regarding runoff concerns of fipronil residues from broadcast use of fipronil for control of fire ants. The registrant did not provide any concurrent biological monitoring of the aquatic environment to assess the impact of fipronil and its degradation products on aquatic invertebrates.
3. The fipronil application was made in July. This application period does not coincide with highest precipitation months. Therefore, fipronil runoff and occurrence in surface water is expected be higher with spring (April-May) applications.
4. The registrant did not collect and analyze sediment samples for fipronil residues.



### Soil

On 15 July 2002, two soil samples were obtained from the top 6 inches of soil within the treatment area for characterization analyses. These samples were shipped to Bayer CropScience and then forwarded to Agvise Laboratories for analysis, where they were received on 17 July 2002. Three additional soil samples, collected from the top 6 inches of soil within the treatment area, were collected on 12 May 2003, and were shipped to Agvise Laboratories where they were received on 13 May 2003. These additional samples were analyzed to determine the saturated hydraulic conductivity level of the soil at the test site. The results of the soil characterization analyses are listed in Table II. Copies of the laboratory reports are included in this Appendix.

**Table II: Soil Characterization**

Parameter	Sample			
	36529-SOIL-R1 0-6"		36529-SOIL-R2 0-6"	
Sand (%)	89		89	
Silt (%)	8		8	
Clay (%)	3		3	
USDA Textural Class (hydrometer method)	Sand		Sand	
Bulk Density (disturbed) (g/cc)	1.24		1.26	
Cation Exchange Capacity (meq/ 100 g)	8.7		9.6	
% moisture at 1/3 bar	9.3		9.5	
% moisture at 15 bar	5.7		5.8	
% Organic Matter	2.5		2.7	
pH (1:1 soil:water)	7.5		7.5	
Phosphorous (ppm)	35		42	
Nitrogen (%)	0.098		0.091	
Soluble Salts (mmhos/cm)	0.11		0.12	
<b>Base Saturation:</b>	<b>%</b>	<b>ppm</b>	<b>%</b>	<b>ppm</b>
Calcium	73.7	1290	74.5	1430
Magnesium	6.7	70	6.7	77
Sodium	0.8	16	0.7	16
Potassium	1.4	49	1.4	53
Hydrogen	17.45	15	16.7	16
Parameter	Sample			
	South Steep Slope 0-6"	NE Flats 0-6"	NW Medium Slope 0-6"	
Saturated Hydraulic Conductivity (cm/hr)	31.0	32.0	33.1	





### Water

Two pond water samples for characterization were collected on 15 July 2002 and were shipped to Agvise Laboratories on 16 July 2002, where they were received the next day. The results are listed in Table I. Copies of the laboratory reports are included in this Appendix.

**Table I: Water Characterization**

Parameter	Sample	
	Character-R1	Character-R2
Sample ID		
pH	7.9	7.9
Sodium (ppm)	4	4
Calcium (ppm)	68	69
Magnesium (ppm)	2	2
Hardness mg equivalent CaCO <sub>3</sub> (ppm)	179	182
Conductivity (mmhos/cm)	0.38	0.37
Sodium Absorption Ratio	0.12	0.11
Total Dissolved Solids (ppm)	244	250
Turbidity (NTU)	9.04	7.10

### 5.3 Weather Data

Rainfall data were collected at the test site. These were supplemented by weather data collected at the University of Florida at Citra, about 8 miles away. Historical weather data, recorded from July to January of 1961 to 1999, were obtained from a University of Florida/NOAA weather station located 30 miles from the test site.

A rainfall of 0.15 inches was recorded onsite within 24 hours of application. Although July was a relatively dry month compared to the historical averages (see text below), several rainfall events occurred throughout July and August that created conditions conducive to runoff. On days 4 through 6 after treatment (July 20 to 22, 2003) a total of 1.8 inches of rain was recorded on site. On days 27 and 33 DAT, 1.0 and 1.3 inches of rain fell onsite, respectively. Over a period of two days at the end of August (45 to 46 DAT), a total 3.6 inches of rain were recorded onsite.

The monthly rainfall averages were typically close to the historical values. Two exceptions occurred in July and December of 2002, when the monthly rainfall values were lower and higher than the historical averages, respectively. The total rainfall recorded on-site (29.1 in) for the study period (July 2002 to January 2003) was similar to the historical value (31.5 in). Because the overall rainfall during the study period was typical for the test site, the variations in the monthly rainfall were not considered significant.

Both historical and study period precipitation data are summarized in Table III. Daily rainfall and temperature data are presented in Appendix D.

**Table III. Monthly Precipitation**

Month	Normal Precipitation (1961-1990)*	University of FL Precipitation Data (2002-2003)**	On-Site Precipitation (2002-2003)
July	7.09 in	4.17 in (1-31 July 2002)	2.91 in (16-31 July 2002)
August	7.99 in	6.03 in	8.20 in
September	5.60 in	4.75 in	4.65 in
October	2.33 in	0.60 in	2.16 in
November	2.04 in	2.55 in	2.33 in
December	3.19 in	8.50 in	6.30 in
January	3.27 in	0.15 in (1-31 January 2002)	2.05 in (1-12 January 2002)
<b>Total</b>	<b>31.51 in</b>	<b>26.75 in</b>	<b>28.60 in</b>

\* University of Florida/NOAA weather station located 30 miles from the test site.

\*\* Obtained from University of Florida at Citra, 8 miles from the test site.

**Table V. Procedural Recoveries of Fipronil and Its Metabolites from Fortified HPLC Water**

Analyte	Fortification Level (ng/mL)	Number of Samples	Mean Percent Recovery
Fipronil	0.010	1	89
	0.100	2	94
	<b>Mean</b>	<b>3</b>	<b>92±9</b>
MB46513	0.010	1	84
	0.100	2	96
	<b>Mean</b>	<b>3</b>	<b>92±12</b>
MB45950	0.010	1	84
	0.100	2	95
	<b>Mean</b>	<b>3</b>	<b>91±11</b>
MB46136	0.010	1	88
	0.100	2	92
	<b>Mean</b>	<b>3</b>	<b>91±10</b>

**Table VI. Procedural Recoveries of Fipronil and Its Metabolites from Fortified Control Pond Water**

Analyte	Fortification Level (ng/mL)	Number of Samples	Mean Percent Recovery
Fipronil	0.010	2	90
	0.100	8	96
	<b>Mean</b>	<b>10</b>	<b>95±11</b>
MB46513	0.010	2	88
	0.100	8	98
	<b>Mean</b>	<b>10</b>	<b>96±9</b>
MB45950	0.010	2	85
	0.100	8	96
	<b>Mean</b>	<b>10</b>	<b>94±9</b>
MB46136	0.010	2	88
	0.100	8	96
	<b>Mean</b>	<b>10</b>	<b>94±9</b>

#### 5.4.3 Analytical Results of Field Samples

A total of 88 treated pond water samples were analyzed throughout the course of the study. Residues of fipronil and its metabolites were nondetectable in 87 of the 88 samples. One sample, collected at 20 DAT, bore nonquantifiable residues of MB46513; no other analytes were detected. Residues of all analytes in three other pond water samples also collected on 20 DAT were nondetectable.

Residues were nondetectable in four samples collected prior to treatment.

## 5.4 Analytical

### 5.4.1 Method Verification

The analytical method has been validated internally and has undergone a successful independent laboratory validation. The results of these studies are presented in separate reports (References 1 and 2).

The analytical method was verified using a fortification solution of a mixture of fipronil and its metabolites. The verification set consisted of 2 control samples of pond water, 3 samples of pond water fortified at the LOQ (0.010 ng/mL), and 3 samples of pond water fortified at 10XLOQ (0.100 ng/mL). The results of the method verification were satisfactory and are shown in Table IV.

**Table IV. Recoveries of Fipronil and Its Metabolites from Method Verification Samples**

Sample Identification (36529-01-)	Fortification Level (ng/mL)	Percent Recovery			
		Fipronil	MB46513	MB45950	MB46136
Bulk-UTC-1	0	ND	ND	ND	ND
Bulk-UTC-2	0	ND	ND	ND	ND
Bulk-10 ppt-1	0.010	94	104	106	117
Bulk-10 ppt-2	0.010	97	104	102	113
Bulk-10 ppt-3	0.010	98	109	101	122
Bulk-100 ppt-1	0.100	105	103	102	103
Bulk-100 ppt-2	0.100	103	102	101	102
Bulk-100 ppt-3	0.100	66	76	77	78
<b>Mean Recovery</b>		<b>94±14 (n=6)</b>	<b>100±12 (n=6)</b>	<b>98±11 (n=6)</b>	<b>106±16 (n=6)</b>

### 5.4.2 Procedural Recoveries

Each sample set contained at least one control sample (HPLC grade water or control pond water) fortified with a mixture of fipronil, MB46513, MB45950 and MB46136. The fortification levels were 0.010 and 0.100 ng/mL. In addition, one unfortified control pond water was included with each analytical set. Fipronil-related residues were nondetectable in all unfortified control samples. The procedural recoveries are summarized in Tables V and VI. In addition, all quantitative data for the procedural recoveries are included in Appendix H.

#### 5.4.4 Analytical Results of Field Recovery Samples

Field recovery samples were prepared to establish the stability of fipronil-related residues during storage at the test site, subsequent transfer to the analytical facility, and further storage prior to analysis. The field recovery samples were prepared in the field on 15 July 2002 by fortifying duplicate 100 mL samples of pond water with a mixture of fipronil, MB46513, MB45950, and MB46136 to yield concentrations of 0.010 ng/mL and 0.100 ng/mL.

The samples were analyzed on 26 March 2003. The results are summarized in Table VII and demonstrate good accountability for the analytes after storage and transfer to the analytical facility. All quantitative data for the field recovery samples are included in Appendix H.

**Table VII. Field Recovery Results**

Sample (36529-)	Fortification Level (ng/mL)	Percent Recovery			
		Fipronil	MB46513	MB45950	MB46136
<b>Procedural Recoveries</b>					
01-Bulk-10 ppt 032503	0.010	104	113	109	118
01-Bulk-100 ppt	0.100	110	108	105	104
<b>Field Spike Recoveries</b>					
FS1-01	0.010	114	100	96	108
FS1-02	0.010	111	97	98	101
FS1-03	0.010	107	101	100	99
FS1-01	0.100	103	102	96	97
FS1-02	0.100	101	100	95	96
FS1-03	0.100	105	102	97	97
<b>Untreated Controls</b>					
01-Bulk-UTC-032503	0	ND	ND	ND	ND
FS1-01 <sup>a</sup>	0	ND	ND	ND	ND
FS1-02 <sup>a</sup>	0	ND	ND	ND	ND
FS1-03 <sup>a</sup>	0	ND	ND	ND	ND

<sup>a</sup> These controls were included with the field spike samples during the shipment and handling procedures.

#### 5.4.5 Storage Stability

Water residue samples were stored under frozen conditions for up to 350 days from collection to analysis. A complete list of critical study dates, including the storage interval for the individual samples, is provided in Appendix F.

A storage stability study investigating the stability of fipronil-related residues in frozen water stored for up to 12 months is currently in progress. Details regarding the conduct of the storage stability investigations and the results will be presented in an addendum to this report.

**Table VIII. Analytical Results of Pond Water Samples**

Sample ID (36529-01-)	Sampling Date	Sampling Event	Analysis Date	Residues (ppb) <sup>a</sup>			
				Fipronil	MB46513	MB45950	MB46136
0001	07/15/02	Pre-Treatment	10/03/02	ND	ND	ND	ND
0003	07/15/02	Pre-Treatment	06/25/03	ND	ND	ND	ND
0005	07/15/02	Pre-Treatment	10/03/02	ND	ND	ND	ND
0007	07/15/02	Pre-Treatment	06/25/03	ND	ND	ND	ND
0011	07/16/02	0 DAT	10/03/02	ND	ND	ND	ND
0013	07/16/02	0 DAT	06/25/03	ND	ND	ND	ND
0015	07/16/02	0 DAT	10/03/02	ND	ND	ND	ND
0017	07/16/02	0 DAT	06/25/03	ND	ND	ND	ND
0019	07/17/02	1 DAT	10/03/02	ND	ND	ND	ND
0021	07/17/02	1 DAT	06/25/03	ND	ND	ND	ND
0023	07/17/02	1 DAT	10/03/02	ND	ND	ND	ND
0025	07/17/02	1 DAT	06/25/03	ND	ND	ND	ND
0027	07/18/02	2 DAT	10/03/02	ND	ND	ND	ND
0029	07/18/02	2 DAT	06/25/03	ND	ND	ND	ND
0031	07/18/02	2 DAT	10/03/02	ND	ND	ND	ND
0033	07/18/02	2 DAT	06/25/03	ND	ND	ND	ND
0035	07/19/02	3 DAT	03/20/03	ND	ND	ND	ND
0037	07/19/02	3 DAT	03/20/03	ND	ND	ND	ND
0039	07/19/02	3 DAT	03/20/03	ND	ND	ND	ND
0041	07/19/02	3 DAT	03/20/03	ND	ND	ND	ND
0043	07/26/02	10 DAT	10/03/02	ND	ND	ND	ND
0045	07/26/02	10 DAT	03/20/03	ND	ND	ND	ND
0047	07/26/02	10 DAT	10/03/02	ND	ND	ND	ND
0049	07/26/02	10 DAT	03/20/03	ND	ND	ND	ND
0051	08/05/02	20 DAT	03/20/03	ND	<0.010	ND	ND
0053	08/05/02	20 DAT	03/20/03	ND	ND	ND	ND
0055	08/05/02	20 DAT	03/20/03	ND	ND	ND	ND
0057	08/05/02	20 DAT	03/20/03	ND	ND	ND	ND
0059	08/15/02	30 DAT	03/21/03	ND	ND	ND	ND
0061	08/15/02	30 DAT	03/21/03	ND	ND	ND	ND
0063	08/15/02	30 DAT	03/21/03	ND	ND	ND	ND
0065	08/15/02	30 DAT	03/21/03	ND	ND	ND	ND
0067	08/25/02	40 DAT	03/21/03	ND	ND	ND	ND
0069	08/25/02	40 DAT	03/21/03	ND	ND	ND	ND
0071	08/25/02	40 DAT	03/21/03	ND	ND	ND	ND

(continued; footnotes to follow)

US EPA ARCHIVE DOCUMENT

**Table VIII (continued). Analytical Results of Pond Water Samples**

Sample ID (36529-01-)	Sampling Date	Sampling Event	Analysis Date	Residues (ppb) <sup>a</sup>			
				Fipronil	MB46513	MB45950	MB46136
0073	08/25/02	40 DAT	03/21/03	ND	ND	ND	ND
0157	09/01/02	Rain 1 (47 DAT)	10/03/02	ND	ND	ND	ND
0161	09/01/02	Rain 1 (47 DAT)	10/03/02	ND	ND	ND	ND
0075	09/04/02	50 DAT	03/21/03	ND	ND	ND	ND
0077	09/04/02	50 DAT	03/21/03	ND	ND	ND	ND
0079	09/04/02	50 DAT	03/21/03	ND	ND	ND	ND
0081	09/04/02	50 DAT	03/21/03	ND	ND	ND	ND
0083	09/14/02	60 DAT	03/28/03	ND	ND	ND	ND
0085	09/14/02	60 DAT	03/28/03	ND	ND	ND	ND
0087	09/14/02	60 DAT	03/28/03	ND	ND	ND	ND
0089	09/14/02	60 DAT	03/28/03	ND	ND	ND	ND
0091	09/24/02	70 DAT	03/28/03	ND	ND	ND	ND
0093	09/24/02	70 DAT	03/28/03	ND	ND	ND	ND
0095	09/24/02	70 DAT	03/28/03	ND	ND	ND	ND
0097	09/24/02	70 DAT	03/28/03	ND	ND	ND	ND
0165	09/25/02	Rain 2 (71 DAT)	10/03/02	ND	ND	ND	ND
0168	09/25/02	Rain 2 (71 DAT)	09/10/03	ND	ND	ND	ND
0169	09/25/02	Rain 2 (71 DAT)	10/03/02	ND	ND	ND	ND
0172	09/25/02	Rain 2 (71 DAT)	09/10/03	ND	ND	ND	ND
0099	10/04/02	80 DAT	03/28/03	ND	ND	ND	ND
0101	10/04/02	80 DAT	03/28/03	ND	ND	ND	ND
0103	10/04/02	80 DAT	03/28/03	ND	ND	ND	ND
0105	10/04/02	80 DAT	03/28/03	ND	ND	ND	ND
0107	10/14/02	90 DAT	04/16/03	ND	ND	ND	ND
0109	10/14/02	90 DAT	04/16/03	ND	ND	ND	ND
0111	10/14/02	90 DAT	04/16/03	ND	ND	ND	ND
0113	10/14/02	90 DAT	04/16/03	ND	ND	ND	ND
0115	10/24/02	100 DAT	04/16/03	ND	ND	ND	ND
0117	10/24/02	100 DAT	04/16/03	ND	ND	ND	ND
0119	10/24/02	100 DAT	04/16/03	ND	ND	ND	ND
0121	10/24/02	100 DAT	04/16/03	ND	ND	ND	ND
0123	11/13/02	120 DAT	04/16/03	ND	ND	ND	ND
0125	11/13/02	120 DAT	04/16/03	ND	ND	ND	ND
0127	11/13/02	120 DAT	04/16/03	ND	ND	ND	ND
0129	11/13/02	120 DAT	04/16/03	ND	ND	ND	ND

(continued; footnotes to follow)

**Table VIII (continued). Analytical Results of Pond Water Samples**

Sample ID (36529-01-)	Sampling Date	Sampling Event	Analysis Date	Residues (ppb) <sup>a</sup>			
				Fipronil	MB46513	MB45950	MB46136
0131	12/03/02	140 DAT	04/23/03	ND	ND	ND	ND
0133	12/03/02	140 DAT	04/23/03	ND	ND	ND	ND
0135	12/03/02	140 DAT	04/23/03	ND	ND	ND	ND
0137	12/03/02	140 DAT	04/23/03	ND	ND	ND	ND
0174	12/10/02	Rain 3 (147 DAT)	09/24/03	ND	ND	ND	ND
0178	12/10/02	Rain 3 (147 DAT)	09/24/03	ND	ND	ND	ND
0182	12/14/02	Rain 4 (151 DAT)	09/24/03	ND	ND	ND	ND
0186	12/14/02	Rain 4 (151 DAT)	09/24/03	ND	ND	ND	ND
0139	12/23/02	160 DAT	04/23/03	ND	ND	ND	ND
0141	12/23/02	160 DAT	04/23/03	ND	ND	ND	ND
0143	12/23/02	160 DAT	04/23/03	ND	ND	ND	ND
0145	12/23/02	160 DAT	04/23/03	ND	ND	ND	ND
0194	12/25/02	Rain 5 (162 DAT)	09/10/03	ND	ND	ND	ND
0196	12/25/02	Rain 5 (162 DAT)	09/10/03	ND	ND	ND	ND
0198	01/02/03	Rain 6 (170 DAT)	09/10/03	ND	ND	ND	ND
0200	01/02/03	Rain 6 (170 DAT)	09/10/03	ND	ND	ND	ND
0202	01/02/03	Rain 6 (170 DAT)	09/10/03	ND	ND	ND	ND
0204	01/02/03	Rain 6 (170 DAT)	09/10/03	ND	ND	ND	ND
0149	01/12/03	180 DAT	05/02/03	ND	ND	ND	ND
0151	01/12/03	180 DAT	05/02/03	ND	ND	ND	ND
0153	01/12/03	180 DAT	05/02/03	ND	ND	ND	ND
0155	01/12/03	180 DAT	05/02/03	ND	ND	ND	ND

<sup>a</sup> ND = nondetectable. The method limit of detection (MDL) for all analytes = 0.004 ppb. The limits of quantification (LOQ) for all analytes = 0.010 ppb.