Data Evaluation Report of Drinking Water Monitoring Study

PMRA Submission Number {.....} EPA MRID Number 45526101

Water Chlorination Study

Data Requirement: PMRA Data Code: N/A
EPA DP Barcode: N/A
OECD Data Point:N/A
EPA Guideline: N/A

Test material:
Common name: Methomyl
Chemical name
IUPAC: *S*-methyl (*EZ*)-*N*-(methylcarbamoyloxy)thioacetimidate
CAS name: methyl *N*-[[(methylamino)carbonyl]oxy]ethanimidothioate

Primary Reviewer: James Hetrick, Ph.D
EPA

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EPA PC Code: 129121

CITATION: Lee, Robert. 2001. Surface Water Monitoring for Residues of Fipronil in Primary Use Areas in the United States. Sponsored by Aventis CropScience, Research Triangle Park, NC. Study Identification No. 98V15506.

EXECUTIVE SUMMARY:

The monitoring study provides supplemental data on the occurrence of fipronil residues in raw surface source drinking water impacted from in-furrow corn uses of fipronil. The study is deficient in assessing the occurrence of fipronil residues in finished drinking water due to the lack of chemical methods for separation and detection of chlorinated fipronil residues. The data are classified as supplemental because there are insufficient storage stability data to support a 16 month sample storage time. Storage stability data were provided to support only a 12 month sample storage time.

Twelve community water systems (CWSs) were selected from the Acetochlor Registration Partnership (ARP) Monitoring Program. These CWSs were selected because there is corn production and fipronil use information for the CWS watersheds. Raw and treated water samples were taken to coincide with the application window of fipronil use on corn (at planting), biweekly samples for 140 days after planting (April to August), and then every third month from September to April. Samples were taken from1998 to 2000. Fipronil and its degradation products (MB46513, MB45950, and MB46136) occurrence was infrequent in the monitoring. Most water samples had fipronil residue concentrations in raw water of ≤ 4 ng/L. The maximum confirmed daily concentration of fipronil

residues was 17.1 ng/L for fipronil, 57.9 ng/L for MB46513, and 55.4 for MB46136 on April 26, 1999 at the Milford, KS site.

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:

MONITORING DESIGN

Study Objective- Evaluate the impact of fipronil corn use products (including REGENT™ 80 WG, REGENT™ 1.5G, and REGENT™ 4SC) on fipronil residue concentrations in raw and finished drinking at selected community water systems (CWSs).

Site Selection Process- Twelve CWS were selected from CWSs in the Acetochlor Registration Partnership (ARP) Monitoring Program because there is corn production as well as fipronil use is projected in their watershed based on 1998 sales data. The sites locations and watershed characteristics are shown in Table 1.

Table 1: Summary of Selected ARP Monitoring Sites in the Fipronil Drinking Water Monitoring Study

Site #	Location	Watershed Size (acres)	County (acres)	Total Crop Land (%)	County Percent in Corn (%)	Water Body Type	Water Treatment
15506-01	Milford, KS	15,963,347	28,434,666	56.1	9.6	Reservoir	Convent, GAC, PAC
15506-02	Paris, IL	11,733	402,873	2.9	38.7	Reservoir	Convent, GAC
15506-03	Decatur, IL	602,057	2,805,517	21.1	41.1	Reservoir	Convent,GAC
15506-04	Pana, IL	4,545	957,274	0.5	32.5	Reservoir	Convent, PAC
15506-05	Kankakee, IL	2,952,111	7,070,194	41.8	37.4	River	Convent, PAC
15506-06	Hudson, IL	41,942	750,843	5.6	44.4	Reservoir	Convent, GAC, PAC

	Bloomington, IN						
15506-07	Litchfield, IL	62,219	915,007	7.6	30.1	Reservoir	Convent, PAC
15506-08	Elgin, IL	953,176	3,160,271	30.2	20.3	River	Convent, GAC
15506-09	Speedway.IN	119,080	1,067,002	11.2	26.8	River	Convent, PAC
15506-10	Milford, IA	14,866	262,026	5.7	32.6	Lake	Convent
15506-11	Archbold, OH	208.,202	1,413,188	14.7	23.9	River	Convent, PAC
15506-12	Delta, OH	22,000	260.,116	33.6	8.5	River	Convent, PAC
15506-13*	Des Moines, IA	2,304,810	6,278,078	36.7	39.4	River	Convent, PAC
15506-14	Streator, IL	NA	686,277	87.07	43.99	River	Convent

Table 2 illustrates the water quality of raw water samples at the selected sites. Water pH was slightly-alkaline to alkaline (7.4 to 8.8).

Table 2: Summary of Water Quality Parameters for Selected Sampling Sites

Site #	Location	pH	Hardness (mg CaCO3 /L)	TDSS (mg/L)	Turbidity (NTU)
15506-01	Milford, KS	7.4	199	354	56.6
15506-02	Paris, IL	7.9	223	248	60.1
15506-03	Decatur, IL	8.1	255	328	46
15506-04	Pana, IL	7.7	108	150	43.6
15506-05	Kankakee, IL	8.0	291	338	8.37
15506-06	Hudson, IL Bloomington, IN	8.2	265	330	1
15506-07	Litchfield, IL	7.6	123	190	107
15506-08	Elgin, IL	8.0	308	406	18.8
15506-09	Speedway.IN	7.7	160	210	236
15506-10	Milford, IA	8.3	221	264	1.06
15506-11	Archbold, OH	8.2	242	340	10.6
15506-12	Delta, OH	8.8	253	390	3.69
***15506-13**	**Des Moines, IA**	**8.3**	**305**	**444**	**34.5**
15506-14	Streator, IL	8.4	275	592	6.99

* Water characterization data for pretreatment water was determined in another study (Aventis CropScience Study Number 98B14452, Ref 4).

SAMPLING AND ANALYTICAL METHODS

Sampling Method and Handling- Paired samples of raw and finished drinking water were taken at each sampling time. The primary focus of sampling methods was on the raw water sample because of poor analytical recovery of fipronil residues from chlorinated waters. There were several sampling sites impacted from upstream anthropogenic processes. [1] Water samples were taken to coincide with the application window of fipronil use on corn (at planting), biweekly samples for 140 days after planting (April to August), and then every third month from September to April. Samples were taken from1998 to 2000.

Water sampling practices were conducted according to the standard operating procedures by the ARP. Several disruptions occurred in the sampling schedule at numerous sites. Two sampling sites (De Moines, IA and Streaton, IL) were added to substitute for the Litchfield, IL and Speedway, IN monitoring sites. The new sampling sites were selected because of low fipronil use in the watershed of the De Moines, IA and Streaton, IL monitoring sites. Additionally, the Milford, KS site had a change in sampling personnel during the study. At the Milford, KS site, 4 targeted monitoring events in the 1998 were missed because sampling personnel took 125 ml samples in polyethylene bottles rather than 1 liter samples in glass bottles. The sampling protocol was changed in 1999 and 2000 to collect 125 ml samples in polyethylene bottles. Samples were shipped on ice via overnight Federal Express to the Aventis CropScience laboratories. Samples were stored in a refrigerator prior to analysis. In 1998, samples were analyzed within the first twelve months. Samples in 1999 and 2000 were stored for a maximum of 16 months prior to analysis.

[1] Upstream anthropogenic processes influenced the quality of water quality at several sites including chlorination at the Milford, KS, occasional PAC at the Decatur, IL, presence of cationic polymer at the Hudson, IL, and additions of CuSO4, KMnO4, and activated carbon at the Archbold, OH and Delta, OH sites.

Analytical Methods-

Analytical methods were designed to determine concentrations of fipronil, MB 45950, MB 46513, and MB 46136. Analytes were determined using a revision of the original analytical method.[2] A revised method was established to address compensate for surface water turbity effects on analyte separation[3]. The liquid chromatograph method was modified to use two 2.0 x 50 mm LC columns instead of 2.0 x 100 mm LC column. The method limit or quantification is 10 ng/L (ppt) and the limit of detection is 4 ng/L (ppt). Sample recoveries from fortified surface water are as follows: 87±8 for fipronil, 84± 7 for MB46513, 82±11 for MB45950, and 84±7 for MB46136. Sample recoveries from fortified laboratory water are as follows: 99±11 for fipronil, 98± 11 for MB46513, 96±13 for MB45950, and 102±19 for MB46136.

Storage Stability

Storage stability studies were conducted in tap water, untreated control surface water, and HPLC grade water fortified with fipronil analytes at 5 ppt. (**Reviewer Note**: ***There is a contradiction on the fortified concentration of analytes. It is stated the fortified concentration is 5 mg/L (5 ppt). The reviewer notes that 5 ppt is equilvalent to 5 ng/L instead 5 mg/L.***) Details of the storage stability studies in tap water were not presented because of unacceptable recovery of fipronil residues. Fortified samples of the untreated control water and HPLC grade water were analyzed on day 0, 7, 30, 90, 120, 270, and 365 of refrigerated storage. Because the analytical method was modified to account for matrix effects in raw surface water, samples were reanalyzed during the course of the study. To evaluate the impact of prolonged storage, storage stability samples were analyzed after a maximum of 16 months storage.

Additionally, a storage stability study was conducted to assess fipronil residue stability during sample storage conditions. Raw water samples from the raw intake water and reservoir of Milford, KS site and the raw water intake of Delta, OH were fortified with fipronil residues and then shipped and analyzed using the proprosed sample handling and analysis SOP .

II. RESULTS AND DISCUSSION:

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[2] "Insecticides, Fipronil: Method of Analysis for possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Water-Revision 4" Issued March 16, 1999.

[3] Seymour, R.J. 2001. Investigation of Analytical Signal Suppression Matrix Effect and HPLC Separation Conditions in Analysis of Fipronil and its Metabolites MB46513, MB45950, and MB46136 in Surface Waters by Electrospray HPLC/MS/MS. Aventis Crop Science Study Number 001V5502; AGREDOC No. B003343; June 26th , 2001.

A. Recovery studies in tap water showed low analytical recovery of "fipronil-related residues" (fipronil and its degradation products). The registrant stated the low recovery is due formation chlorinated products in the presence of high concentrations of free chlorine. Method performance for recovery of fipronil residues in raw surface water were verified. Sample recoveries from fortified surface water are as follows: 87±8 for fipronil, 84± 7 for MB46513, 82±11 for MB45950, and 84±7 for MB46136. Sample recoveries from fortified laboratory water are as follows: 99±11 for fipronil, 98± 11 for MB46513, 96±13 for MB45950, and 102±19 for MB46136 (**Tables II and III**).

B. Storage stability studies showed high procedural corrected recoveries for fipronil and its degradation products (**Table IV; Figures 1 to 4**). Recoveries in raw surface water after 12 months of storage ranged from 85 to 98% (of applied) for fipronil, 91 to 95 % for MB46513, 82 to 90% for MB45950, and 80 to 90% for MB46136.

C. Fipronil residue recoveries in field spiked samples of Milford, KS chlorinated water were low for MB46513, MB45950, and MB46136 (**Table V**). Field spike recoveries in chlorinated water ranged from 5.3 to 27.5% (of applied) for fipronil degradation products. Procedural corrected recoveries of fipronil residues in field spikes for sources waters from Milford, KS and Delta, OH ranged from 87.5 to 102.4%.

D. The registrant stated there were no confirmed detections of fipronil residues in raw source water at the fourteen test sites during a 3 year sampling period (**Table VI**). Confirmatory analysis was conducted on duplicate water samples with a detection of fipronil residues. There was a detection of fipronil residues in the April 26, 1999 sample at the Milford, KS site. However, the residues were not detected in duplicate water samples. (The registrant stated this sample was the last sample taken by the Milford, KS personnel. The sample was send to Aventis without a chain of custody form.) The concentrations for fipronil residues ranged from 12.7 to 17.1 ng/L for fipronil, 52.5 to 57.9 ng/L for MB46513, and 48.4 to 55.4 for MB46136. Additionally, unconfirmed fipronil residues detection were found for fipronil (11.4 ng/L) and MB46136 (4.5 ng/l) for 6/23/99 samples at Bloomington,IL: MB46513 (20 ng/L) and MB46136 (11.3 ng/l) for 5/13/98 samples at Litchfield, IL; and MB46136 (18.8 ng/l) for 7/8/98 samples at Elgin, IL.

E. Because chlorinated degradation products of fipronil were not determined in the chemical analysis, the water monitoring study is focused solely on raw source water.

III. STUDY DEFICIENCIES:

The study is deficient in assessing the occurrence of fipronil residues in finished drinking water due to the lack of chemical methods for separation and detection of chlorinated fipronil residues. Additionally, the reviewer notes most of the water treatment plants in the monitoring study employ activated carbon in their treatment scheme. Activated carbon is expected to be effective in the removal of fipronil residues.

IV. REVIEWER'S COMMENTS:

A. Storage stabilities studies were conducted for 12 months; however, samples were reanalyzed at 16 months post-treatment. Storage stability studies should be designed to bracket the storage time for the samples. The data are classified as supplemental because there are insufficient storage stability data to support a 16 month sample storage time. Storage stability data were provided to support only a 12 month sample storage time.

B. The monitoring study provides supplemental data on the occurrence of fipronil residues in raw surface source drinking water. The maximum confirmed daily concentration of fipronil residues was 17.1 ng/L for fipronil, 57.9 ng/L for MB46513, and 55.4 for MB46136.

C. There is a contradiction on the fortified concentration of analytes in the storage stability studies. It is stated the fortified concentration is 5 mg/L (5 ppt). The reviewer notes that 5 ppt is equilvalent to 5 ng/L instead 5 mg/L.

D. The registrant did not analyze water samples for the alkaline hydrolysis degradation product RPA 105048 (desulfiniylfipronil amide). Because the monitoring sites had slightly alkaline to alkaline pH, RPA 105048 is expected to be an important fipronil degradation product.

Table II: Recovery of Fipronil and Its Metabolites from Fortified Control HPLC Grade Water

		% Recovery[a]			
Analysis Date[b]	Spike Level (ppt)	Fipronil	MB46513	MB45950	MB46136
3/30/1999	10	106.9	108.8	109.4	113.1
3/31/1999	10	88.5	100.1	93.5	78.8
4/1/1999	10	95.0	97.5	82.2	90.0
4/2/1999	10	86.0	89.2	85.0	97.6
9/13/1999	10	88.7	79.2	86.5	120.9
9/30/1999	10	107.1	98.3	97.3	127.5
9/30/1999	10	96.2	89.2	101.7	113.4
10/1/1999	10	101.9	118.6	103.9	126.7
10/1/1999	10	92.1	117.0	89.9	127.1
10/8/1999	10	90.8	99.6	99.0	95.3
10/11/1999	10	113.1	101.5	105.9	120.0
10/11/1999	10	113.1	101.5	105.9	120.0
10/12/1999	10	111.5	97.3	103.0	115.7
10/12/1999	10	105.8	97.6	93.7	114.9
10/15/1999	10	87.3	88.5	95.2	111.6
10/18/1999	10	87.3	88.1	82.3	81.0
10/20/1999	10	94.1	97.5	91.2	83.0
10/21/1999	10	92.9	97.4	100.5	88.7
11/9/1999	10	81.3	90.1	97.7	109.8
12/7/1999	10	107.4	98.6	106.0	97.9
12/10/1999	10	111.1	103.7	123.4	116.3
2/7/2000	10	116.2	102.0	74.5	101.8
3/29/1999	50	102.9	101.5	103.1	98.3
3/30/1999	50	98.0	96.5	94.7	97.2
3/31/1999	50	122.5	111.7	104.8	107.8
4/1/1999	50	105.5	98.8	99.3	102.0
4/6/1999	50	74.9	73.4	72.1	73.1
6/27/2000	50	100.8	101.0	96.8	95.5
7/6/2000	50	103.5	105.4	101.3	94.9
7/10/2000	50	113.2	108.4	101.7	99.5
7/19/2000	50	101.8	100.4	101.2	93.8
8/14/2000	50	101.3	94.6	98.6	94.5
8/30/2000	50	95.6	96.0	94.8	90.2
1/4/2001	50	97.4	98.1	91.7	84.2
3/12/2001	50	74.3	60.3	44.4	51.4
3/26/1999	100	103.3	99.7	98.2	103.3

Table II: Recovery of Fipronil and Its Metabolites from Fortified Control HPLC Grade Water

Analysis Date[b]	Spike Level (ppt)	% Recovery[a]			
		Fipronil	MB46513	MB45950	MB46136
3/29/1999	100	100.2	112.9	109.0	106.8
3/30/1999	100	108.8	111.2	100.4	100.3
3/30/1999	100	73.3	71.8	71.7	71.7
3/31/1999	100	102.1	104.5	100.7	105.2
4/1/1999	100	92.6	97.9	97.0	168.9
4/2/1999	100	90.6	102.3	99.5	100.6
4/7/1999	100	104.6	105.5	102.7	105.4
9/13/1999	100	110.1	105.7	104.5	111.7
11/12/1999	100	95.4	99.1	98.4	88.4
Average[c] ± SD		98.8 ± 11.2	98.2 ± 11.1	95.9 ± 12.6	102.1 ± 18.8

a. Samples were spiked and analyzed on the analysis date shown

b. Recoveries were corrected for apparent residues in the associated controls.

c. n = 45 (determinations)

Table III: Recovery of Fipronil and Its Metabolites from Fortified Raw Water

Sample Number 15506-	Analysis Date	Spike Level (ppt)	%Recovery			
			Fipronil	MB46513	MB45950	MB46136
MK-Lake	6/27/2000	50	84.3	89.7	91.5	91.9
684	6/27/2000	50	89.3	92.2	94.8	92.3
740	6/27/2000	50	88.0	92.1	95.9	94.0
797	7/6/2000	50	74.6	88.0	92.5	93.0
852	7/6/2000	50	92.1	89.2	93.6	91.1
909	7/6/2000	50	79.9	88.2	91.1	93.8
1021	7/10/2000	50	100.2	93.3	87.6	91.9
1076	7/10/2000	50	95.7	96.8	94.0	95.4
1244	7/10/2000	50	94.0	89.4	90.5	89.8
1852	7/19/2000	50	98.5	94.8	96.4	86.1
1854	7/19/2000	50	97.3	94.0	92.4	86.1
DMUTC051198	7/19/2000	50	96.3	93.0	88.5	88.9
3063	7/19/2000	50	95.8	92.4	90.4	86.5
MK-Lake	8/14/2000	50	98.7	82.5	87.1	88.0
684	8/14/2000	50	101.1	77.5	90.7	84.6
740	8/14/2000	50	72.0	85.6	96.4	85.5
797	8/14/2000	50	91.2	79.3	91.7	84.6
852	8/14/2000	50	90.1	83.1	87.0	81.0
909	8/14/2000	50	86.8	89.5	89.8	82.1
1021	8/30/2000	50	90.5	85.9	86.0	84.4
1244	8/30/2000	50	80.3	80.4	80.4	80.0
1852	8/30/2000	50	87.4	89.0	84.2	84.0
1854	8/30/2000	50	97.6	87.4	87.2	81.4
MDUTC051198	8/30/2000	50	93.6	83.3	82.5	93.1
3603	8/30/2000	50	93.7	89.8	88.5	90.8
Mk-Lake c	1/4/2001	50	88.0	89.2	88.9	82.8
684 c	1/4/2001	50	91.4	89.5	90.0	84.8
740 c	1/4/2001	50	94.2	90.0	89.2	80.2
797 c	1/4/2001	50	96.9	91.6	91.6	89.9
852 c	1/4/2001	50	90.2	91.1	90.0	84.2
051198 c	1/4/2001	50	88.9	83.7	85.0	85.9
1021 c	1/4/2001	50	88.3	87.1	90.0	86.0
1244 c	1/4/2001	50	93.7	86.7	90.6	85.7
1852 c	1/4/2001	50	96.8	88.3	89.2	84.6
1854 c	1/4/2001	50	90.2	83.3	83.8	81.2
3603 c	1/4/2001	50	93.0	88.6	89.3	82.6
909 c	1/4/2001	50	93.8	87.6	92.4	87.0
797	3/12/2001	50	90.2	89.0	80.6	84.8
684	3/12/2001	50	85.6	84.7	77.9	85.8
1854	3/12/2001	50	80.9	80.0	74.4	78.9
740	3/12/2001	50	78.9	83.2	75.0	88.4
1021	3/12/2001	50	86.2	82.3	73.2	83.0

Table III: Recovery of Fipronil and Its Metabolites from Fortified Raw Water

Sample Number 15506-	Analysis Date	Spike Level (ppt)	%Recovery			
			Fipronil	MB46513	MB45950	MB46136
1244	3/12/2001	50	85.5	84.6	73.9	82.2
852	3/12/2001	50	86.1	84.2	73.2	85.6
909	3/12/2001	50	81.4	82.3	76.1	80.3
1852	3/12/2001	50	86.0	85.9	74.6	79.4
Mk-Lake	3/12/2001	50	85.5	85.0	77.0	82.1
1852	3/12/2001	50	85.8	80.5	76.3	85.2
3603	3/12/2001	50	81.8	79.6	74.0	82.1
Mk-Lake	6/7/2001	50	83.2	77.5	68.4	77.3
684	6/7/2001	50	76.7	72.9	64.2	74.8
740	6/7/2001	50	74.4	70.9	63.4	70.1
797	6/7/2001	50	78.2	73.1	65.1	72.9
852	6/7/2001	50	83.5	74.0	67.5	72.5
909	6/7/2001	50	75.7	71.4	64.7	72.0
1021	6/7/2001	50	75.3	71.4	66.3	75.6
1244	6/7/2001	50	82.3	76.6	70.0	79.6
1852	6/7/2001	50	77.8	70.8	63.0	71.7
1854	6/7/2001	50	78.7	75.9	68.8	77.2
3603	6/7/2001	50	77.5	72.9	64.9	72.5
051198	6/7/2001	50	70.7	65.2	57.9	67.4
3603	7/27/2001	50	90.3	84.0	83.2	90.9
Average ± SD[a]			87.3 ± 7.6	84.2 ± 7.1	82.2 ± 10.5	83.7 ± 6.5

a. n = 62 (determinations)

Table IV: Stability of Fipronil-Related Residues Fortified in Water at 500 ppt and Stored Refrigerated for Up to 12 Months

Sample Description	Sample Number	Analyte	Storage Interval						
			0 Time	1 Week	1 Month	3 Months	6 Months[a]	9 Months	12 Month
			Procedural Recoveries (%)						
HPC Grade Water	-	Fipronil	n/a[b]	98	84	97	93	111	105
		MB46513		102	83	97	100	110	103
		MB45950		100	82	98	96	107	98
		MB46136		94	75	91	99	104	96
Raw Water from Milford, IA Trial No. 15506-10	15506-1244	Fipronil	n/a	n/a	n/a	n/a	92	90	99
		MB46513					89	91	96
		MB45950					90	95	97
		MB46136					90	97	92
Raw Water from Kankakee, IL Trial No. 15506-05	15506-852	Fipronil	n/a	n/a	n/a	n/a	89	89	101
		MB46513					90	95	95
		MB45950					91	94	92
		MB46136					90	92	92
Raw Water from Milford, KS Trial No. 15506-01	15506-628 15506-629	Fipronil	n/a	n/a	n/a	n/a	74	94	92
		MB46513					84	100	91
		MB45950					85	101	89
		MB46136					86	102	89

Table IV: Stability of Fipronil-Related Residues Fortified in Water at 500 ppt and Stored Refrigerated for Up to 12 Months

Sample Description	Sample Number	Analyte	Storage Interval						
			0 Time	1 Week	1 Month	3 Months	6 Months[a]	9 Months	12 Month
			Stored Recoveries (%)[c]						
Raw Water from Milfold, IA Trial No. 15506-10	15506-1245	Fipronil	93	83 **[85]**	84 **[100]**	89 **[92]**	90 **[98]**	88 **[98]**	97 **[98]**
		MB46513	97	93 **[91]**	85 **[102]**	92 **[95]**	87 **[98]**	90 **[99]**	91 **[95]**
		MB45950	95	92 **[92]**	84 **[102]**	93 **[95]**	89 **[99]**	92 **[97]**	90 **[93]**
		MB46136	114	92 **[98]**	80 **[107]**	91 **[100]**	87 **[97]**	92 **[95]**	90 **[98]**
Raw Water from Kankakee, IL Trial No. 15506-05	15506-852	Fipronil	75	75 **[77]**	74 **[88]**	69 **[71]**	87 **[98]**	86 **[97]**	86 **[85]**
		MB46513	93	91 **[89]**	86 **[104]**	86 **[89]**	89 **[99]**	85 **[89]**	86 **[91]**
		MB45950	93	90 **[90]**	84 **[102]**	83 **[85]**	87 **[96]**	85 **[90]**	85 **[92]**
		MB46136	96	88 **[94]**	89 **[119]**	91 **[100]**	87 **[97]**	84 **[91]**	88 **[96]**
Raw Water from Milford, KS Trial No. 15506-01	15506-628 15506-629[d]	Fipronil	78	74 **[76]**	72 **[86]**	63 **[65]**	72 **[97]**	86 **[91]**	90 **[98]**
		MB46513	94	91 **[89]**	82 **[99]**	82 **[85]**	77 **[92]**	86 **[86]**	86 **[95]**
		MB45950	94	91 **[91]**	81 **[99]**	79 **[81]**	73 **[86]**	80 **[79]**	82 **[92]**
		MB46136	97	90 **[96]**	82 **[109]**	86 **[95]**	69 **[80]**	73 **[72]**	80 **[90]**

a. For the 6, 9, and 12 month storage intervals, samples were analyzed using method revision 20002, which specifies the use of two 2 x 50 mm LC columns.

b. n/a = not analyzed.

c. Stored recoveries corrected for procedural recoveries are bolded and shown parenthetically. The following formula was used: corrected stored recov. = stored recov./procedural recov. x 100. For the 1 week, 1 month, and 3 month storage intervals, procedural recoveries from spiked HPLC water were used for calculations. For the 6, 9, and 12 month storage intervals, procedural recoveries from spiked raw water collected from the associated trial site were used.

d. Replicate samples from bulk sample 15506-629 were analyzed at the 3-12 months storage intervals.

Figure 1: Storage Stability of Fipronil Residues for 12 Months in Raw Water from Selected Water Treatment Facilities

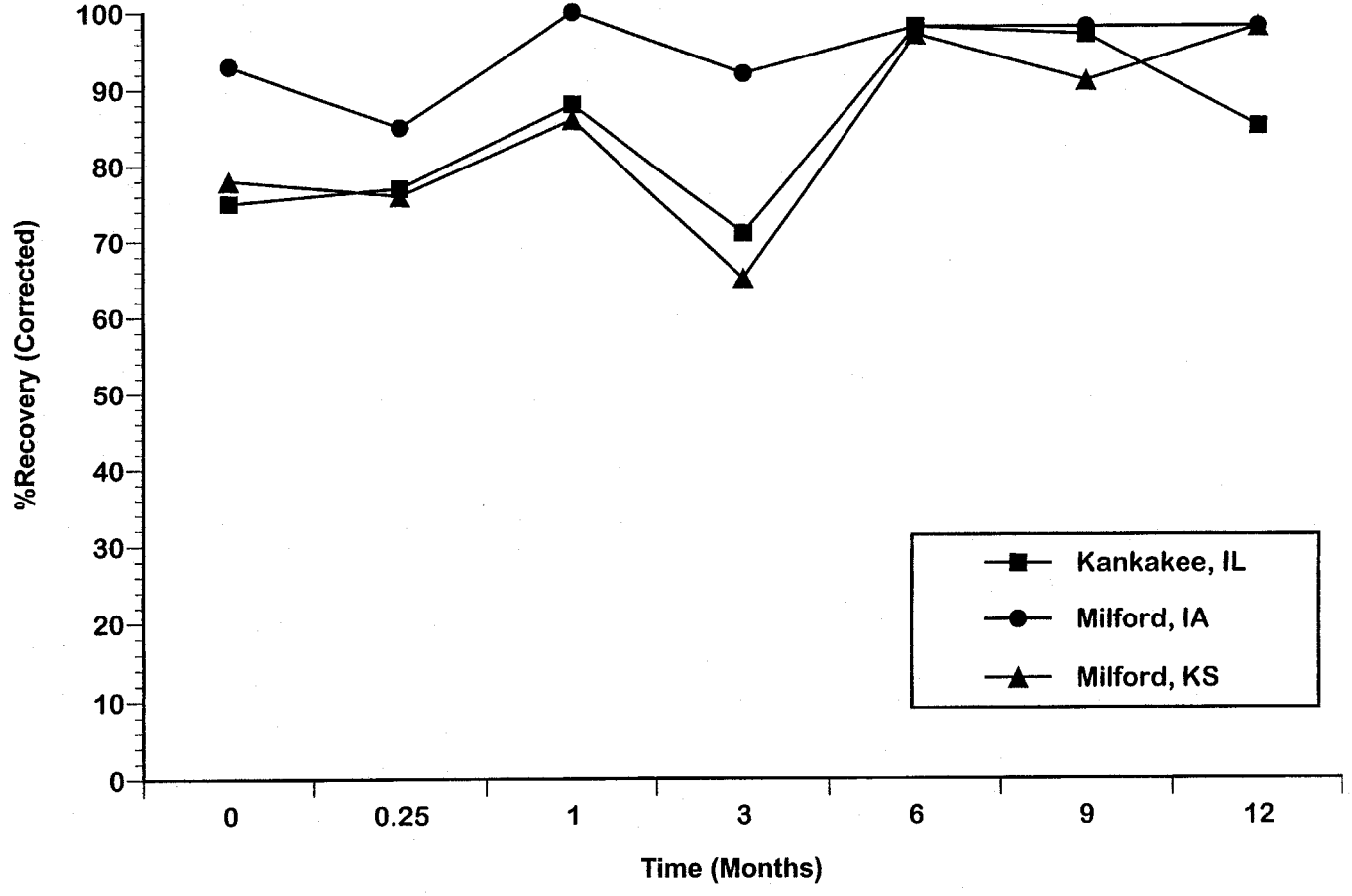

Figure 2: Storage Stability of MB46136 Residues for 12 Months in Raw Water from Selected Water Treatment Facilities

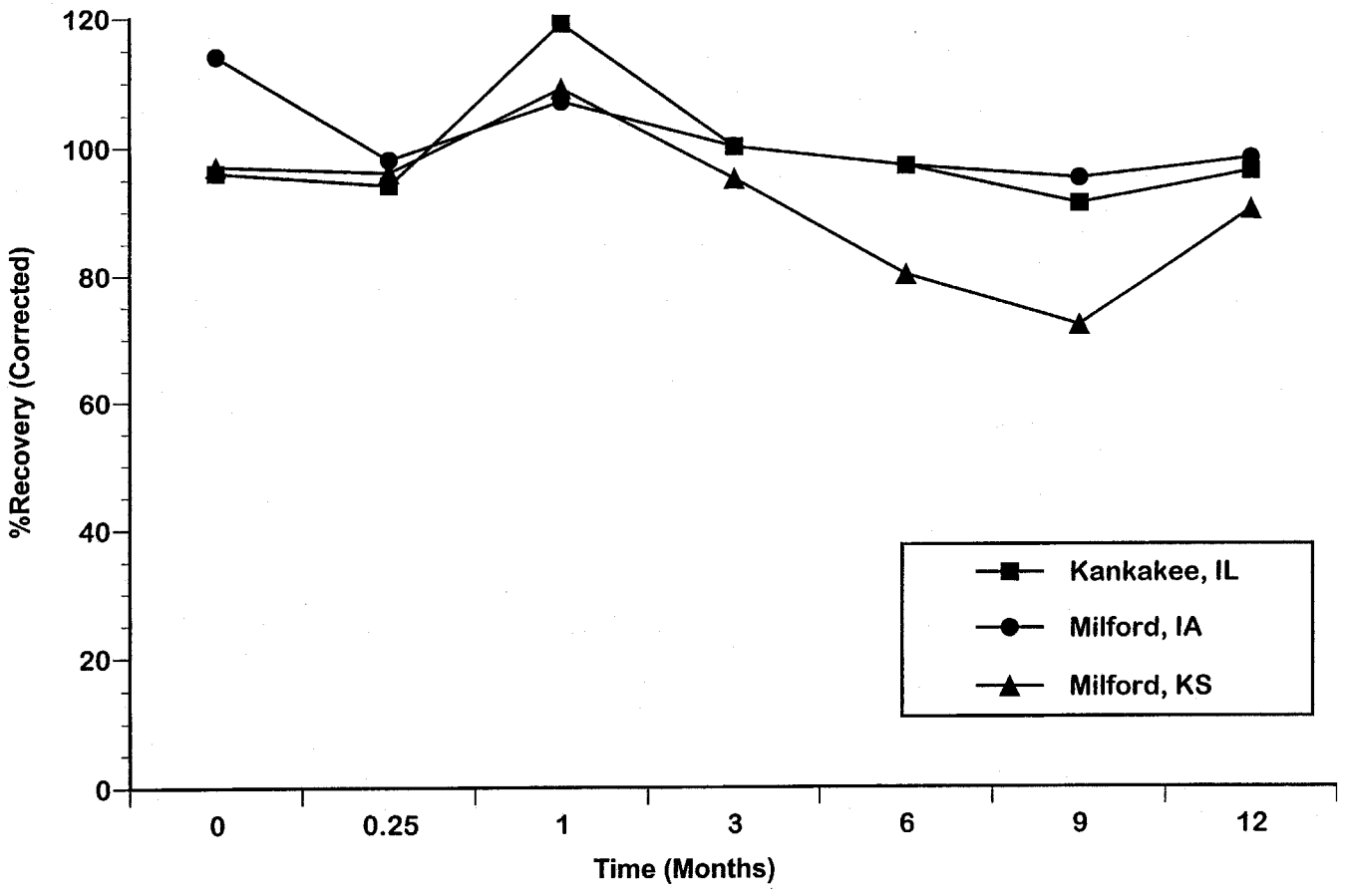

Figure 3: Storage Stability of MB45950 Residues for 12 Months in Raw Water from Selected Water Treatment Facilities

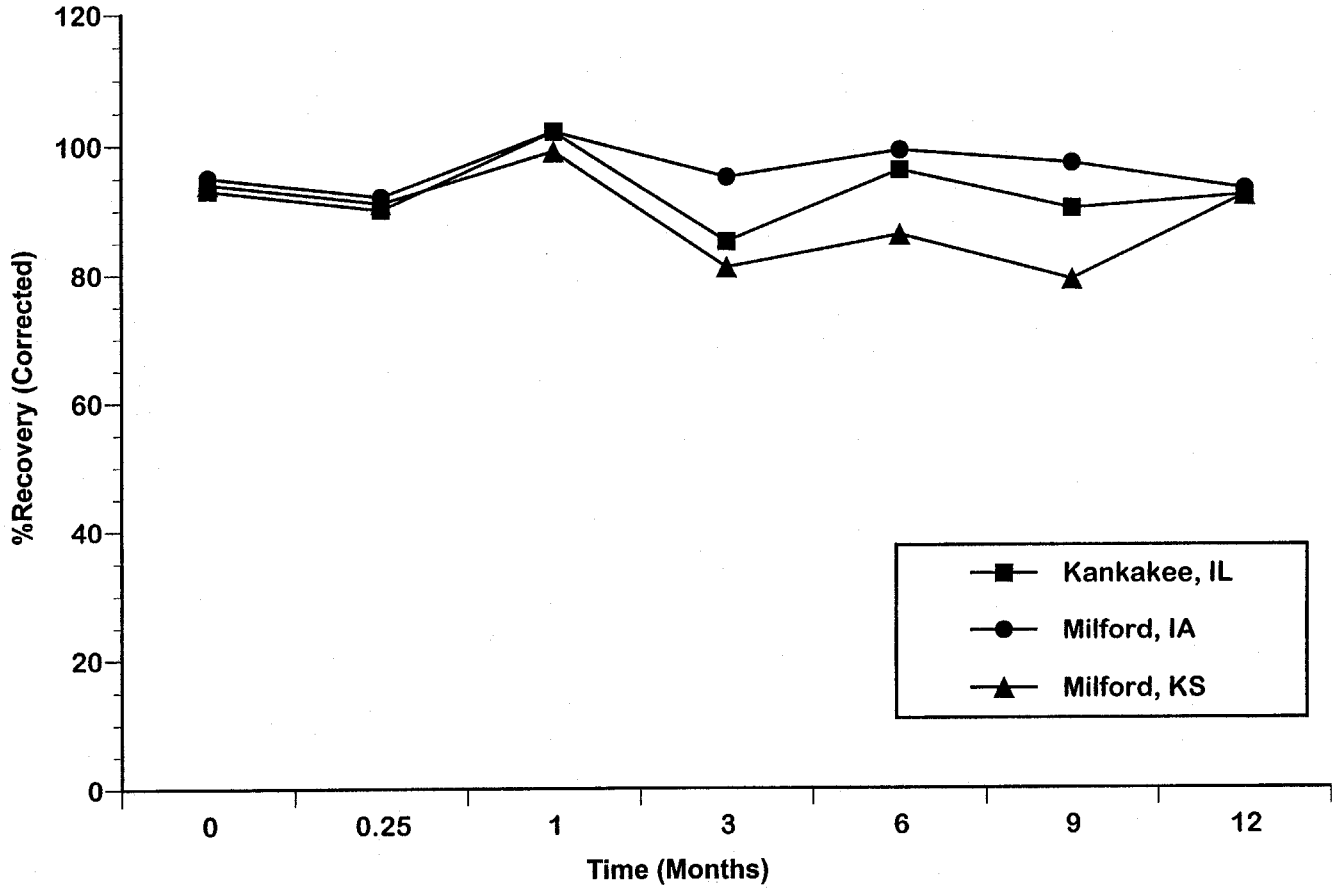

Figure 4: Storage Stability of MB46513 Residues for 12 Months in Raw Water from Selected Water Treatmet Facilities

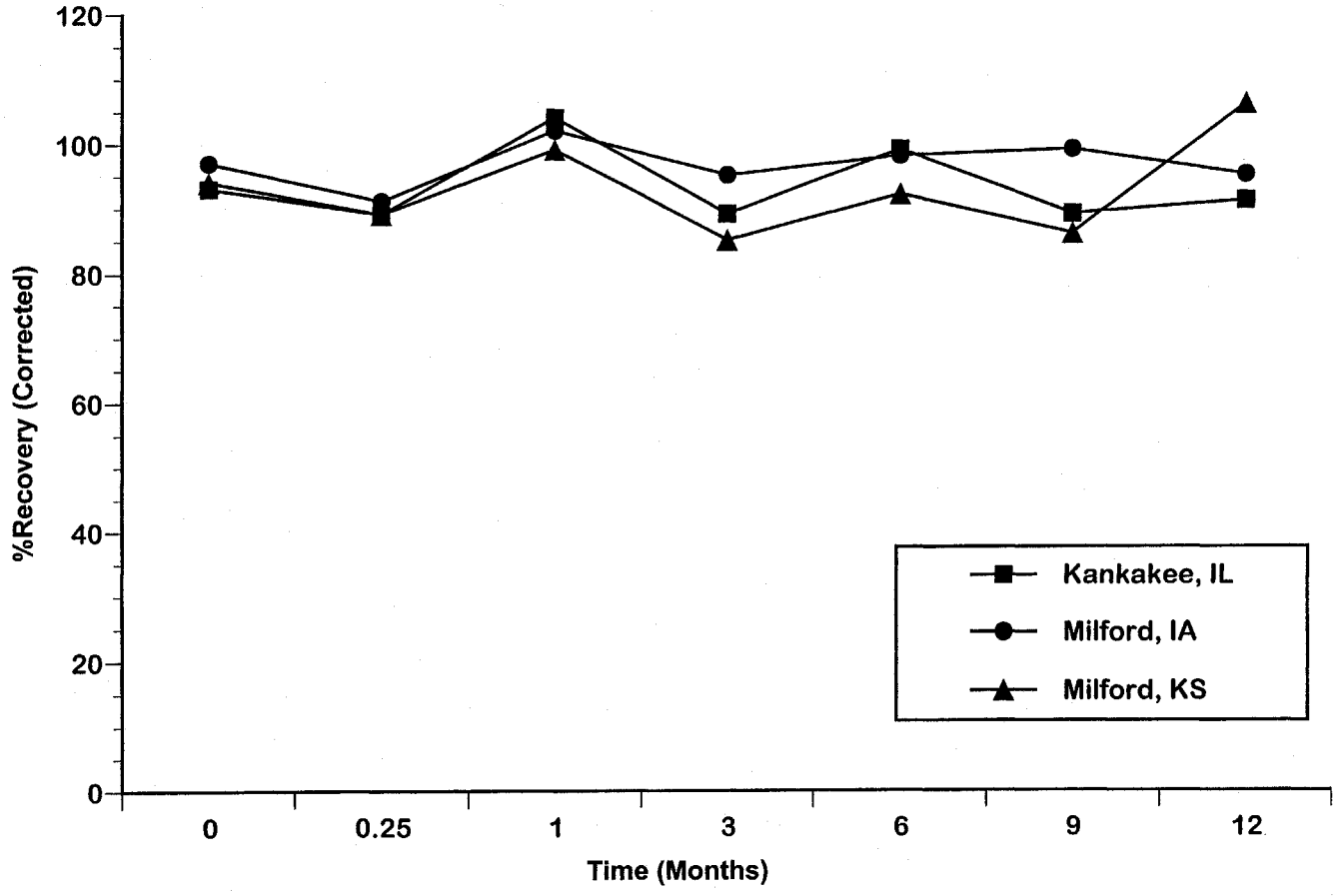

Table V: Recoveries from Field Spike Samples Prepared at Trials 15506-01 and -12

Analytes	Procedural Recoveries (%)			Field Spike Recoveries (%)[a]	
	HPLC Grade Water	Raw Water[b]	Average Recovery from Raw Water	Uncorrected	Corrected[c]
Trial 15506-01 (Milford, KS) - Sampled from Inside Plant[d]					
Fipronil	90.7, 91.7	87.6, 82.5	85.1	nd, nd	-
MB46513	77.2, 79.1	75.7, 77.3	76.5	20.7, 21.0	27.1, 27.5
MB45950	63.3, 63.1	66.6, 69.4	68.0	3.3, 2.9	4.9, 4.3
MB46136	61.3, 60.9	68.5, 72.0	70.3	3.7, 4.0	5.3, 5.7
Trial 15501-01 (Milford, KS) - Sampled Directly From Source					
Fipronil	89.1	79.7, 69.6	74.8	76.6, 76.1	102.4, 101.7
MB46513	89.9	74.8, 69.5	72.2	68.2, 76.3	94.5, 105.8
MB45950	78.1	68.2, 63.3	65.8	65.4, 57.5	99.5, 87.5
MB46136	88.7	77.4, 74.7	76.1	63.4, 72.3	83.4, 95.1
Trial 15506-12 (Delta, OH)					
Fipronil	106.8	71.7, 70.3	71.0	68.7, 69.1	96.8, 97.3
MB46513	106.4	91.8, 88.5	90.2	91.8, 88.5	101.8, 98.2
MB49550	107.8	95.4, 91.8	93.6	93.0, 92.3	99.4, 98.6
MB46136	100.8	91.2, 88.6	89.9	84.7, 85.9	94.2, 95.6

a. Field spike samples were fortified at 100 ppt of each analyte

b. Uncorrected

c. Field spike recoveries were corrected for the average procedural recovery from two fortified raw water samples collected at the corresponding trial site.

d. Samples where taken inside treatment facility downstream of the chlorinator.

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
15506-01	628	4/9/98	3/26/99	nd	nd	nd	nd
(Milford, KS)	632	4/13/98	3/26/99	nd	nd	nd	nd
	636	4/27/98	3/26/99	nd	nd	nd	nd
	640	5/11/98	3/30/99	nd	nd	nd	nd
	644	5/26/98	3/30/99	nd	nd	nd	nd
	648	6/11/98	3/30/99	nd	nd	nd	nd
	652	6/22/98	3/31/99	nd	nd	nd	nd
	656	7/7/98	3/31/99	nd	nd	nd	nd
	660	7/20/98	3/31/99	nd	nd	nd	nd
	664	8/3/98	4/1/99	nd	nd	nd	nd
	668	8/17/98	4/1/99	nd	nd	nd	nd
	672	11/9/98	4/1/99	nd	nd	nd	nd
	2752	1/11/99	9/13/99	nd	nd	nd	nd
			5/23/00	nd	nd	nd	nd
	2756	3/15/99	9/13/99	nd	nd	nd	nd
			5/23/00	nd	nd	nd	nd
	2760	4/5/99	9/30/99	<LOQ (7.5)	nd	nd	nd
			10/19/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2761[d]	4/5/99	10/20/99	nd	nd	nd	nd
	2764	4/26/99	9/30/99	17.1	52.5	<LOQ (8.6)	48.4
			10/19/99	15.8	57.9	<LOQ (8.7)	55.4
			11/9/99	12.7	55.4	<LOQ (9.4)	50.3
	2765[d]	4/26/99	10/20/99	nd	nd	nd	nd
			11/9/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2766[e]	4/26/99	11/12/99	nd	nd	nd	nd
	2767	4/26/99	11/12/99	nd	nd	nd	nd
	2776	5/27/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2780	6/8/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2784	6/21/99	10/8/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2788	7/6/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2792	7/19/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2796	8/2/99	10/15/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
			6/12/00	nd	nd	nd	nd
	2800	8/16/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2804	11/7/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2848	1/17/00	2/7/00	nd	nd	nd	nd
			6/27/00	nd	nd	nd	nd
	2852	3/21/00	6/27/00	nd	nd	nd	nd
	2856	4/3/00	6/27/00	nd	nd	nd	nd
	2860	4/17/00	6/27/00	nd	nd	nd	nd
	2864	5/2/00	6/27/00	nd	nd	nd	nd
	2868	5/16/00	6/27/00	nd	nd	nd	nd
	2872	5/30/00	6/27/00	nd	nd	nd	nd
	2876	6/13/00	6/27/00	nd	nd	nd	nd
	2880	6/26/00	8/18/00	nd	nd	nd	nd
	2884	7/10/00	8/18/00	nd	nd	nd	nd
	2888	7/24/00	8/18/00	nd	nd	nd	nd
	2892	8/7/00	8/18/00	nd	nd	nd	nd
	2896	8/21/00	1/4/01	nd	nd	nd	nd
	2900	11/6/00	1/4/01	nd	nd	nd	nd
	4064	1/24/01	3/12/01	nd	nd	nd	nd
	4068	3/19/01	6/7/01	nd	nd	nd	nd
15506-02	684	4/9/98	3/26/99	nd	nd	nd	nd
(Paris, IL)	688	4/15/98	3/26/99	nd	nd	nd	nd
	692	4/29/98	3/26/99	nd	nd	nd	nd
	696	5/13/98	3/30/99	nd	nd	nd	nd
	700	5/28/98	3/30/99	nd	nd	nd	nd
			4/6/99	nd	nd	nd	nd
	704	6/10/98	3/30/99	nd	nd	nd	nd
	708	6/25/98	3/31/99	nd	nd	nd	nd
	712	7/13/98	3/31/99	nd	nd	nd	nd
	716	7/22/98	3/31/99	nd	nd	nd	nd
	720	8/5/98	4/1/99	nd	nd	nd	nd
			4/6/99	nd	nd	nd	nd
	724	8/19/98	4/1/99	nd	nd	nd	nd
	1912	1/14/99	9/13/99	nd	nd	nd	nd
			5/23/00	nd	nd	nd	nd
	1916	3/17/99	9/13/99	nd	nd	nd	nd
			5/23/00	nd	nd	nd	nd
	1920	3/31/99	9/30/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
			5/24/00	nd	nd	nd	nd
	1924	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	1928	4/28/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1932	5/12/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1936	5/26/99	10/8/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1940	6/10/99	10/8/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1944	6/23/99	10/8/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1948	7/8/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	1952	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	1956	8/4/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	1960	8/23/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	1964	11/10/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3016	1/19/00	2/7/00	nd	nd	nd	nd
			6/27/00	nd	nd	nd	nd
	3020	3/23/00	6/27/00	nd	nd	nd	nd
	3024	4/5/00	6/27/00	nd	nd	nd	nd
	3028	4/19/00	6/27/00	nd	nd	nd	nd
	3032	5/3/00	6/27/00	nd	nd	nd	nd
	3036	5/18/00	6/27/00	nd	nd	nd	nd
	3040	5/31/00	6/27/00	nd	nd	nd	nd
	3044	6/14/00	6/27/00	nd	nd	nd	nd
	3048	6/28/00	8/18/00	nd	nd	nd	nd
	3052	7/12/00	8/18/00	nd	nd	nd	nd
	3056	7/26/00	8/18/00	nd	nd	nd	nd
	3060	8/9/00	8/18/00	nd	nd	nd	nd
	3068	6/28/00	1/4/01	nd	nd	nd	nd
	4132	1/17/01	3/12/01	nd	nd	nd	nd
	4136	3/21/01	6/7/01	nd	nd	nd	nd
	4140	4/4/01	6/7/01	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
15506-03	740	4/8/98	3/26/99	nd	nd	nd	nd
(Decatur, IL)	744	4/15/98	3/26/99	nd	nd	nd	nd
	748	4/29/98	3/26/99	nd	nd	nd	nd
	752	5/13/98	3/30/99	nd	nd	nd	nd
	756	5/27/98	3/30/99	nd	nd	nd	nd
	760	6/10/98	3/30/99	nd	nd	nd	nd
	764	6/24/98	3/31/99	nd	nd	nd	nd
	768	7/8/98	3/31/99	nd	nd	nd	nd
	772	7/22/98	3/31/99	10	nd	nd	59
			4/6/99	nd	nd	nd	nd
	773[d]	7/22/98	4/7/99	nd	nd	nd	nd
	776	8/6/98	4/1/99	nd	nd	nd	nd
	780	8/20/98	4/1/99	nd	nd	nd	nd
	784	11/17/98	4/1/99	nd	nd	nd	nd
	2136	1/13/99	9/13/99	nd	nd	nd	nd
			5/23/00	nd	nd	nd	nd
	2140	3/17/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2148	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2152	4/29/99	10/20/99	nd	nd	nd	nd
	2156	5/12/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2160	5/26/99	10/8/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2164	6/10/99	10/8/99	nd	nd	nd	nd
			10/19/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2165[d]	6/10/99	10/20/99	nd	nd	nd	nd
	2168	6/23/99	10/8/99	nd	nd	nd	nd
			10/19/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2169[d]	6/23/99	10/20/99	nd	nd	nd	nd
	2172	7/7/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2176	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2180	8/4/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	2184	8/18/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2188	11/10/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3184	1/19/00	2/7/00	nd	nd	nd	nd
			6/27/00	nd	nd	nd	nd
	3188	3/22/00	6/27/00	nd	nd	nd	nd
	3192	4/5/00	6/27/00	nd	nd	nd	nd
	3196	4/19/00	6/27/00	nd	nd	nd	nd
	3200	5/3/00	6/27/00	nd	nd	nd	nd
	3204	5/17/00	6/27/00	nd	nd	nd	nd
	3208	5/31/00	6/27/00	nd	nd	nd	nd
	3212	6/14/00	6/27/00	nd	nd	nd	nd
	3216	6/28/00	8/18/00	nd	nd	nd	nd
	3220	7/12/00	8/18/00	nd	nd	nd	nd
	3224	7/26/00	8/18/00	nd	nd	nd	nd
	3228	8/9/00	8/18/00	nd	nd	nd	nd
	3232	8/23/00	8/18/00	nd	nd	nd	nd
	3236	11/8/00	1/4/01	nd	nd	nd	nd
	4096	1/17/01	3/12/01	nd	nd	nd	nd
	4100	3/21/01	6/7/01	nd	nd	nd	nd
	4104	4/4/01	6/7/01	nd	nd	nd	nd
15506-04	796	4/8/98	3/29/99	nd	nd	nd	nd
(Pana, IL)	800	4/15/98	3/29/99	nd	nd	nd	nd
	804	4/29/98	3/29/99	nd	nd	nd	nd
	808	5/13/98	3/30/99	nd	nd	nd	nd
	812	5/27/98	3/30/99	nd	nd	nd	nd
	816	6/10/98	3/30/99	nd	nd	nd	nd
	820	6/24/98	3/31/99	nd	nd	nd	nd
	824	7/8/98	3/31/99	nd	nd	nd	nd
	828	7/22/98	3/31/99	nd	nd	nd	nd
	832	8/5/98	4/1/99	nd	nd	nd	nd
	836	8/19/98	4/1/99	nd	nd	nd	nd
	840	11/9/98	4/1/99	nd	nd	nd	nd
	1856	1/20/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	1860	3/17/99	9/13/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	1864	4/8/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	1868	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	1872	4/28/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1876	5/13/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	1880	5/26/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	1884	6/9/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	1888	6/23/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	1892	7/7/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	1896	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	1900	8/4/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	1904	8/18/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	1908	11/10/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2960	1/19/00	2/7/00	nd	nd	nd	nd
			7/5/00	nd	nd	nd	nd
	2964	3/22/00	7/5/00	nd	nd	nd	nd
	2968	4/5/00	7/5/00	nd	nd	nd	nd
	2972	4/19/00	7/5/00	nd	nd	nd	nd
	2976	5/3/00	7/5/00	nd	nd	nd	nd
	2980	5/17/00	7/5/00	nd	nd	nd	nd
	2984	5/31/00	7/5/00	nd	nd	nd	nd
	2988	6/14/00	7/5/00	nd	nd	nd	nd
	2992	6/28/00	8/18/00	nd	nd	nd	nd
	2996	7/12/00	8/18/00	nd	nd	nd	nd
	3000	7/26/00	8/18/00	nd	nd	nd	nd
	3004	8/9/00	8/18/00	nd	nd	nd	nd
	3008	8/23/00	1/4/01	nd	nd	nd	nd
	3012	11/8/00	1/4/01	nd	nd	nd	nd
	4144	1/17/01	3/12/01	nd	nd	nd	nd
	4148	3/21/01	6/7/01	nd	nd	nd	nd
	4152	4/4/01	6/7/01	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
15506-05	852	4/9/98	3/29/99	nd	nd	nd	nd
(Kankakee, IL)	856	4/15/98	3/29/99	nd	nd	nd	nd
	860	4/29/98	3/29/99	nd	nd	nd	nd
	864	5/13/98	3/30/99	nd	nd	nd	nd
	868	5/27/98	3/30/99	nd	nd	nd	nd
	872	6/10/98	3/30/99	nd	nd	nd	nd
	876	6/24/98	3/31/99	nd	nd	nd	nd
	880	7/8/98	3/31/99	nd	nd	nd	nd
	884	7/22/98	3/31/99	nd	nd	nd	nd
	888	8/10/98	4/1/99	nd	nd	nd	nd
	892	8/19/98	4/1/99	nd	nd	nd	nd
	896	11/9/98	4/1/99	nd	nd	nd	nd
	2080	1/13/99	9/13/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2084	3/17/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2088	3/31/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2092	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2096	4/28/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2100	5/12/99	10/1/99	nd	nd	nd	nd
			10/19/99	nd	nd	nd	nd
			6/1/99	nd	nd	nd	nd
	2101[d]	5/12/99	10/20/99	nd	nd	nd	nd
	2104	5/26/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2108	6/9/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2112	6/23/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2116	7/7/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2120	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2124	8/4/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2128	8/18/99	10/15/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	2132	11/10/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3128	1/19/00	2/7/00	nd	nd	nd	nd
			7/5/00	nd	nd	nd	nd
	3132	3/22/00	7/5/00	nd	nd	nd	nd
	3136	4/5/00	7/5/00	nd	nd	nd	nd
	3140	4/19/00	7/5/00	nd	nd	nd	nd
	3144	5/3/00	7/5/00	nd	nd	nd	nd
	3148	5/17/00	7/5/00	nd	nd	nd	nd
	3152	5/31/00	7/5/00	nd	nd	nd	nd
	3156	6/14/00	7/5/00	nd	nd	nd	nd
	3160	6/28/00	8/18/00	nd	nd	nd	nd
	3164	7/12/00	8/18/00	nd	nd	nd	nd
	3168	7/26/00	8/18/00	nd	nd	nd	nd
	3172	8/9/00	8/18/00	nd	nd	nd	nd
	3176	8/23/00	1/4/01	nd	nd	nd	nd
	3180	11/8/00	1/4/01	nd	nd	nd	nd
	4108	1/17/01	3/12/01	nd	nd	nd	nd
	4112	3/21/01	6/7/01	nd	nd	nd	nd
	4116	4/4/01	6/7/01	nd	nd	nd	nd
15506-06	908	4/7/98	3/29/99	nd	nd	nd	nd
(Bloomington, IL)	912	4/15/98	3/29/99	nd	nd	nd	nd
			4/6/99	nd	nd	nd	nd
	916	4/29/98	3/29/99	nd	nd	nd	nd
			4/6/99	nd	nd	nd	nd
	920	5/13/98	3/30/99	nd	nd	nd	nd
	924	5/27/98	3/30/99	nd	nd	nd	nd
	928	6/10/98	3/30/99	nd	nd	nd	nd
	932	6/24/98	3/31/99	nd	nd	nd	nd
	936	7/8/98	3/31/99	nd	nd	nd	nd
	940	7/22/98	3/31/99	nd	nd	nd	nd
	944	8/5/98	4/1/99	nd	nd	nd	nd
	948	8/19/98	4/1/99	nd	nd	nd	nd
	952	11/9/98	4/1/99	nd	nd	nd	nd
	2640	1/13/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2644	3/18/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2652	4/14/99	9/30/99	nd	nd	nd	nd
	2656	4/28/99	10/1/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b] Fipronil	MB46513	MB45950	MB46136
			6/1/00	nd	nd	nd	nd
	2660	5/12/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2664	5/26/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2668	6/9/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2672	6/23/99	10/8/99	11.4	nd	nd	<LOQ (4.5)
			11/9/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2673[d]	6/23/99	11/9/99	nd	nd	nd	nd
	2674	6/23/99	11/22/99	nd	nd	nd	nd
	2675	6/23/99	11/12/99	nd	nd	nd	nd
	2676	7/7/99	10/12/99	nd	nd	nd	nd
			6/18/00	nd	nd	nd	nd
	2680	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2684	8/4/99	10/21/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2688	8/18/99	10/21/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2692	11/10/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3240	1/19/00	2/7/00	nd	nd	nd	nd
			7/5/00	nd	nd	nd	nd
	3244	3/22/00	7/5/00	nd	nd	nd	nd
	3248	4/5/00	7/5/00	nd	nd	nd	nd
	3252	4/19/00	7/5/00	nd	nd	nd	nd
	3256	5/3/00	7/5/00	nd	nd	nd	nd
	3260	5/31/00	7/5/00	nd	nd	nd	nd
	3264	5/17/00	7/5/00	nd	nd	nd	nd
	3268	6/14/00	7/5/00	nd	nd	nd	nd
	3272	6/28/00	8/18/00	nd	nd	nd	nd
	3276	7/12/00	8/18/00	nd	nd	nd	nd
	3280	7/26/00	8/18/00	nd	nd	nd	nd
	3284	8/9/00	8/18/00	nd	nd	nd	nd
	3288	8/23/00	1/4/01	nd	nd	nd	nd
	3292	11/8/00	1/4/01	nd	nd	nd	nd
	4076	1/17/01	3/12/01	nd	nd	nd	nd
	4080	3/21/01	6/7/01	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b] Fipronil	MB46513	MB45950	MB46136
	4084	4/4/01	6/7/01	nd	nd	nd	nd
15506-07	964	4/8/98	3/29/99	nd	nd	nd	nd
(Litchfield, IL)	968	4/15/98	3/29/99	nd	nd	nd	nd
	972	4/29/98	3/29/99	nd	nd	nd	nd
	976	5/13/98	3/30/99	nd	20.0	nd	11.3
	977[d]	5/13/98	4/7/99	nd	nd	nd	nd
	980	5/27/98	3/30/99	nd	nd	nd	nd
	984	6/10/98	3/30/99	nd	nd	nd	nd
	988	6/30/98	3/31/99	nd	nd	nd	nd
	992	7/8/98	3/31/99	nd	nd	nd	nd
	996	7/22/98	3/31/99	nd	nd	nd	nd
	1000	8/5/98	4/1/99	nd	nd	nd	nd
	1004	8/19/98	4/2/99	nd	nd	nd	nd
	1008	11/9/98	4/2/99	nd	nd	nd	nd
	2192	1/13/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2196	3/17/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2200	3/31/99	9/30/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2204	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
15506-08	1020	4/9/98	3/29/99	nd	nd	nd	nd
(Elgin, IL)	1024	4/15/98	3/29/99	nd	nd	nd	nd
	1028	4/29/98	3/29/99	nd	nd	nd	nd
	1032	5/13/98	3/30/99	nd	nd	nd	nd
	1036	5/27/98	3/30/99	nd	nd	nd	nd
	1040	6/11/98	3/30/99	nd	nd	nd	nd
	1044	6/24/98	3/31/99	nd	nd	nd	nd
	1048	7/8/98	3/31/99	nd	nd	nd	18.8
			4/6/99	nd	nd	nd	nd
	1052	7/22/98	3/31/99	nd	nd	nd	nd
	1056	8/5/98	4/2/99	nd	nd	nd	nd
	1060	8/19/98	4/2/99	nd	nd	nd	nd
	1064	11/9/98	4/2/99	nd	nd	nd	nd
	2024	1/13/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2028	3/18/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2032	3/31/99	9/30/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
			5/24/00	nd	nd	nd	nd
	2036	4/14/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2040	4/28/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2044	5/12/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2048	5/26/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2052	6/9/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2056	6/23/99	10/8/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2060	7/7/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2064	7/21/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2068	8/4/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2072	8/18/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2076	11/10/99	12/10/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3072	1/19/00	7/10/00	nd	nd	nd	nd
	3076	3/22/00	7/10/00	nd	nd	nd	nd
	3080	4/5/00	7/10/00	nd	nd	nd	nd
	3084	4/19/00	7/10/00	nd	nd	nd	nd
	3088	5/3/00	7/10/00	nd	nd	nd	nd
	3092	5/17/00	7/10/00	nd	nd	nd	nd
			7/12/00	nd	nd	nd	nd
	3096	5/31/00	7/10/00	nd	nd	nd	nd
	3100	6/14/00	7/10/00	nd	nd	nd	nd
	3104	6/28/00	8/30/00	nd	nd	nd	nd
	3108	7/12/00	8/30/00	nd	nd	nd	nd
	3112	7/26/00	8/30/00	nd	nd	nd	nd
	3116	8/9/00	8/30/00	nd	nd	nd	nd
	3120	8/23/00	8/30/00	nd	nd	nd	nd
	3124	11/8/00	1/4/01	nd	nd	nd	nd
	4120	1/17/01	3/12/01	nd	nd	nd	nd
	4124	3/21/01	6/7/01	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	4128	4/4/01	6/7/01	nd	nd	nd	nd
15506-09	1076	4/9/98	3/29/99	nd	nd	nd	nd
(Speedway, IN)	1080	4/14/98	3/29/99	nd	nd	nd	nd
	1084	4/28/98	3/29/99	nd	nd	nd	nd
	1088	5/12/98	3/30/99	nd	nd	nd	nd
	1092	5/26/98	3/30/99	nd	nd	nd	nd
	1096	6/9/98	3/30/99	nd	nd	nd	nd
	1100	6/23/98	3/31/99	nd	nd	nd	nd
			4/6/99	nd	nd	nd	nd
	1104	7/7/98	4/6/99	nd	nd	nd	nd
	1108	7/21/98	3/31/99	nd	nd	nd	nd
	1112	8/4/98	4/2/99	nd	nd	nd	nd
	1116	8/18/98	4/2/99	nd	nd	nd	nd
	1120	11/9/98	4/2/99	nd	nd	nd	nd
	2248	1/12/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2252	3/16/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2256	4/1/99	9/30/99	nd	nd	nd	nd
	2260	4/13/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2264	4/27/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2268	5/11/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2272	5/25/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2276	6/8/99	10/20/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2280	6/22/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2284	7/6/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2288	7/20/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2292	8/3/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2296	8/17/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2300	11/9/99	12/10/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
			6/13/00	nd	nd	nd	nd
	2904	1/19/00	7/10/00	nd	nd	nd	nd
	2908	3/21/00	7/10/00	nd	nd	nd	nd
	2912	4/4/00	7/10/00	nd	nd	nd	nd
	2916	4/18/00	7/10/00	nd	nd	nd	nd
	2920	5/2/00	7/10/00	nd	nd	nd	nd
15506-10	1244	4/10/98	3/30/99	nd	nd	nd	nd
(Milford, IA)	1248	4/22/98	3/30/99	nd	nd	nd	nd
	1252	5/6/98	3/30/99	nd	nd	nd	nd
	1256	5/20/98	3/30/99	nd	nd	nd	nd
	1260	6/3/98	3/30/99	nd	nd	nd	nd
	1264	6/17/98	4/6/99	nd	nd	nd	nd
	1268	7/1/98	4/6/99	nd	nd	nd	nd
	1272	7/15/98	4/6/99	nd	nd	nd	nd
	2472	1/20/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2476	3/24/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2480	4/7/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2484	4/21/99	9/13/99	nd	nd	nd	nd
			5/22/00	nd	nd	nd	nd
	2488	5/5/99	9/13/99	nd	nd	nd	nd
	2492	5/19/99	9/13/99	nd	nd	nd	nd
	2496	6/2/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2500	6/16/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2504	6/30/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2508	7/14/99	12/7/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	2512	7/28/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2516	8/11/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2520	8/25/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2524	11/17/99	12/10/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	3408	1/26/00	7/10/00	nd	nd	nd	nd
	3412	3/29/00	7/10/00	nd	nd	nd	nd
	3416	4/12/00	7/10/00	nd	nd	nd	nd
	3420	4/26/00	7/10/00	nd	nd	nd	nd
	3424	5/10/00	7/10/00	nd	nd	nd	nd
	3428	5/24/00	7/10/00	nd	nd	nd	nd
	3432	6/7/00	7/10/00	nd	nd	nd	nd
	3436	6/21/00	7/10/00	nd	nd	nd	nd
	3440	7/5/00	8/30/00	nd	nd	nd	nd
	3444	7/19/00	8/30/00	nd	nd	nd	nd
	3448	8/2/00	8/30/00	nd	nd	nd	nd
	3452	8/16/00	8/30/00	nd	nd	nd	nd
	3456	8/30/00	1/4/01	nd	nd	nd	nd
	3460	11/15/00	1/4/01	nd	nd	nd	nd
	4016	1/24/01	3/12/01	nd	nd	nd	nd
	4024	4/11/01	6/7/01	nd	nd	nd	nd
15506-11	1304	5/4/98	3/30/99	nd	nd	nd	nd
(Archbold, OH)	1308	5/26/98	3/30/99	nd	nd	nd	nd
	1312	6/1/98	4/6/99	nd	nd	nd	nd
	1316	6/15/98	4/6/99	nd	nd	nd	nd
	1320	6/29/98	4/6/99	nd	nd	nd	nd
	1324	7/13/98	4/2/99	nd	nd	nd	nd
	1328	8/4/98	4/2/99	nd	nd	nd	nd
	1332	8/10/98	4/2/99	nd	nd	nd	nd
	1336	8/24/98	4/2/99	nd	nd	nd	nd
	1340	11/16/98	4/2/99	nd	nd	nd	nd
	2304	1/19/99	9/13/99	nd	nd	nd	nd
	2308	3/22/99	9/13/99	nd	nd	nd	nd
	2312	4/5/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2316	4/19/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2320	5/3/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2324	5/17/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2328	6/1/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2332	6/14/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	2336	6/28/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2340	7/12/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2344	7/26/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2348	8/9/99	10/18/99	nd	nd	nd	nd
	.		6/12/00	nd	nd	nd	nd
	2352	8/23/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2356	11/15/99	12/10/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3296	1/24/00	7/19/00	nd	nd	nd	nd
	3300	3/27/00	7/19/00	nd	nd	nd	nd
	3304	4/10/00	7/19/00	nd	nd	nd	nd
	3308	4/24/00	7/19/00	nd	nd	nd	nd
	3312	5/8/00	7/19/00	nd	nd	nd	nd
	3316	5/22/00	7/19/00	nd	nd	nd	nd
	3320	6/5/00	7/19/00	nd	nd	nd	nd
	3324	6/19/00	7/19/00	nd	nd	nd	nd
	3328	7/5/00	8/30/00	nd	nd	nd	nd
	3332	7/17/00	8/30/00	nd	nd	nd	nd
	3336	7/31/00	8/30/00	nd	nd	nd	nd
	3340	8/14/00	8/30/00	nd	nd	nd	nd
	3344	8/28/00	8/30/00	nd	nd	nd	nd
	3348	11/13/00	1/4/01	nd	nd	nd	nd
	4000	1/22/01	3/12/01	nd	nd	nd	nd
	4004	3/26/01	6/7/01	nd	nd	nd	nd
	4008	4/9/01	6/7/01	nd	nd	nd	nd
	4012	4/23/01	6/7/01	nd	nd	nd	nd
15506-12	1360	5/6/98	3/30/99	nd	nd	nd	nd
(Delta, OH)	1364	5/20/98	3/30/99	nd	nd	nd	nd
	1368	6/3/98	4/6/99	nd	nd	nd	nd
	1372	6/17/98	4/6/99	nd	nd	nd	nd
	1376	7/1/98	4/6/99	nd	nd	nd	nd
	1380	7/15/98	4/2/99	nd	nd	nd	nd
	1384	7/29/98	4/2/99	nd	nd	nd	nd
	1388	8/12/98	4/2/99	nd	nd	nd	nd
	1392	8/26/98	4/2/99	nd	nd	nd	nd
	1396	11/23/98	4/2/99	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	2360	1/21/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2364	3/24/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2368	4/7/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2372	4/21/99	9/30/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2376	5/5/99	10/1/99	nd	nd	nd	nd
			5/24/00	nd	nd	nd	nd
	2380	5/19/99	10/1/99	nd	nd	nd	nd
			6/1/00	nd	nd	nd	nd
	2384	6/2/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2388	6/16/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2392	6/30/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2396	7/14/99	10/12/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2400	7/28/99	10/12/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2404	8/11/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2408	8/25/99	10/18/99	nd	13.9	nd	nd
			11/9/199	nd	14.0	nd	nd
	2409[d]	8/25/99	11/9/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2410	8/25/99	11/12/99	nd	nd	nd	nd
	2411	8/25/99	11/12/99	nd	nd	nd	nd
	2412	11/17/99	12/10/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3356	1/26/00	7/19/00	nd	nd	nd	nd
	3360	4/12/00	7/19/00	nd	nd	nd	nd
	3364	4/26/00	7/19/00	nd	nd	nd	nd
	3368	5/10/00	7/19/00	nd	nd	nd	nd
	3372	5/24/00	7/19/00	nd	nd	nd	nd
	3376	6/7/00	7/19/00	nd	nd	nd	nd
	3380	6/21/00	7/19/00	nd	nd	nd	nd
	3384	7/5/00	8/30/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	3388	7/19/00	8/30/00	nd	nd	nd	nd
	3392	8/2/00	8/30/00	nd	nd	nd	nd
	3396	8/16/00	8/30/00	nd	nd	nd	nd
	3400	8/30/00	1/4/01	nd	nd	nd	nd
	3404	11/15/00	1/4/01	nd	nd	nd	nd
	4048	1/24/01	3/12/01	nd	nd	nd	nd
	4056	4/11/01	6/7/01	nd	nd	nd	nd
	4060	4/25/01	6/7/01	nd	nd	nd	nd
15506-13	2808	5/14/99	10/11/99	nd	nd	nd	nd
(Des Moines, IA)			6/5/00	nd	nd	nd	nd
	2812	5/19/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2816	6/2/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2820	6/16/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2824	6/30/99	10/11/99	nd	nd	nd	nd
			6/5/00	nd	nd	nd	nd
	2828	7/14/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2832	7/28/99	10/12/99	nd	nd	nd	nd
			6/8/00	nd	nd	nd	nd
	2836	8/11/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2840	8/25/99	10/18/99	nd	nd	nd	nd
			6/12/00	nd	nd	nd	nd
	2844	11/17/99	12/10/99	nd	nd	nd	nd
			6/13/00	nd	nd	nd	nd
	3464	1/26/00	7/19/00	nd	nd	nd	nd
	3468	3/29/00	7/19/00	nd	nd	nd	nd
	3472	4/12/00	7/19/00	nd	nd	nd	nd
	3476	4/26/00	7/19/00	nd	nd	nd	nd
	3480	5/10/00	7/19/00	nd	nd	nd	nd
	3484	5/24/00	7/19/00	nd	nd	nd	nd
	3488	6/7/00	7/19/00	nd	nd	nd	nd
	3492	6/7/00	7/19/00	nd	nd	nd	nd
	3496	7/5/00	8/30/00	nd	nd	nd	nd
	3500	7/19/00	8/30/00	nd	nd	nd	nd
	3504	8/2/00	8/30/00	nd	nd	nd	nd
	3508	8/16/00	8/30/00	nd	nd	nd	nd

Table VI: Summary of Residue Results for Raw Water Samples Analyzed for Fipronil-Related Residues

Trial	Sample No. 15506-	Date Sampled[c]	Date Analyzed	Residues (ppt)[a,b]			
				Fipronil	MB46513	MB45950	MB46136
	3512	8/30/00	1/4/01	nd	nd	nd	nd
	3516	11/15/00	1/4/01	nd	nd	nd	nd
	4028	1/24/01	3/12/01	nd	nd	nd	nd
	4036	4/11/01	6/7/01	nd	nd	nd	nd
	4040	4/25/01	6/7/01	nd	nd	nd	nd
	4044	5/9/01	6/7/01	nd	nd	nd	nd
15506-14	3520	3/20/00	7/19/00	nd	nd	nd	nd
Streator, IL	3524	4/3/00	7/19/00	nd	nd	nd	nd
	3528	4/17/00	7/19/00	nd	nd	nd	nd
	3532	5/1/00	7/19/00	nd	nd	nd	nd
	3536	5/15/00	7/19/00	nd	nd	nd	nd
	3540	5/30/00	7/19/00	nd	nd	nd	nd
	3544	6/12/00	7/19/00	nd	nd	nd	nd
	3548	6/26/00	7/19/00	nd	nd	nd	nd
	3552	7/10/00	8/30/00	nd	nd	nd	nd
	3556	7/24/00	8/30/00	nd	nd	nd	nd
	3560	8/7/00	8/30/00	nd	nd	nd	nd
	3564	8/21/00	8/30/00	nd	nd	nd	nd
	3568	11/6/00	1/4/01	nd	nd	nd	nd
	4088	1/16/01	3/12/01	nd	nd	nd	nd

a. For each analyte, the MDL = 4 ppt and the LOQ = 10 ppt. ND = nondetectable.

b. Isolated samples bore detectable residues upon initial analyses. With the exception of one sample from Trial 15506-01 (raw water collected on 4/26/99), no residues were found when these raw water samples were re-analyzed. In addition, no residues were detected in any of the duplicate raw water samples analyzed that were collected at the same sampling intervals. Therefore, all residues detected in this study are considered laboratory contamination.

c. For each study year, typically biweekly sampling occurred between April and August; thereafter, samples were collected once in each November, and in January and March of the following calendar year (see Section 2.2.3). At each sampling interval, duplicate raw water samples were collected at each test site. Unless otherwise noted, only one replicate raw water sample was analyzed for each sampling interval.

d. Samples are the replicate raw water sample from a single sampling event.

e. Bold sample numbers indicate the results are from finished water samples.