Data Evaluation Report of Surface Water Monitoring Study

PMRA Submission Number {.....} EPA MRID Number 46477003

Test material: Fipronil
IUPAC name: 5-amino-1-(2,6-dichloro-α,α,α-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: James C. Hetrick
Date: 5/5/08

Secondary Reviewer: Thuy Nguyen
EPA
Signature:
Date: 5/6/08

EPA PC Code: 129121

CITATION: Jackson, Scott and Huns Nejad. 2005. Monitoring Fipronil Residues in Surface and Runoff Water at Twenty Locations Throughout the Midwest Corn Belt. Unpublished study performed and sponsored by BASF, Research Triangle Park, NC. 2005/5000001.

EXECUTIVE SUMMARY: The study provides acceptable data on the edge-of-field runoff for fipronil residues for field corn in the Mid-Western United States. The study provides a probabilistic site selection process to represent a 1 year distribution of edge-of-field runoff of fipronil residues in the Mid-Western U.S. corn belt. These data indicate that the maximum edge-of-field concentration of fipronil residues range from 0 to 3,600 ng/L for fipronil, <10 to 13 ng/L for MB 46513, <10 to 165 ng/L for MB 46950, and <10 to 61 ng/L for MB 46136. Several sites had farm ponds for sample collection. The maximum concentration in pond water was 159 ng/L for fipronil and 24 ng/L for MB46513. MB 45950 and MB 46136 were not detected in the farm pond water. As expected, runoff of fipronil residues were correlated to precipitation. However, an analysis of the time series of fipronil concentrations on short time intervals (0 to 12 hours after runoff event) indicate a sinus curve of fipronil concentration independent of rainfall amounts. These fluctuations in concentrations appear to correspond to changes in volume and velocity of runoff waters.

Data Evaluation Report of Surface Water Monitoring Study

PMRA Submission Number {.....} EPA MRID Number 46477003

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:

B. MONITORING DESIGN:

Site Selection Process- Twenty monitoring sites were selected to represent runoff sites for fipronil use on field corn in the Mid-Western United States. Selection of runoff sites were based on county level GIS assessment of geographic location, 1980-1990 NRCS rainfall intensity (USLE R-factor), and kriged Regent® total sales data relative to potential sampling points on receiving waters. Selected monitoring sites had an appropriate hydrology to allow sampling of runoff points into surface water or a water channel flowing into surface water. The equation used for deriving a site vulnerability is as follows:

$$\text{Vulnerability Ranking} = (\text{use lb/mile}^2)*0.01 \int_{n}^{1} (916 + 331* \log \text{USLE R factor (in/hr)}$$

Site were selected to be statistically representative of regional populations of fields. Representative sites were selected according to allow appropriate local hydrology for monitoring edge-of-field runoff and surface water.

Field Treatment/Application- Selected field sites were amended with a single application of in-furrow fipronil (formulated as Regent®) at 4.16 ounces/A on field corn during planting in April or May. The fields in the monitoring study remained in corn production during the 2004 season. All monitoring sites required a 60 foot runoff buffer from the edge-of-field and monitoring site to be in accordance with the Regent® label. Farmers were allowed to make other maintenance pesticide applications.

Summary of Locations - Site locations were found in IL, KY, NE, IA, MN, MO, IN, MI, and OH. Information on each monitoring site is shown in Table 1.

Table 1: Site Location

State	Site ID	Sampling Locations	Sampling Method	Total Acreage in Watershed
IL	IL3	edge- of-field	Grab/Auto	~ 58
	IL5	edge-of- field	Grab/Siphon	~ 20
	IL6	edge-of- field	Grab/Siphon	~ 37
	IL4	edge-of- field	Grab/Siphon	~ 20
KY	KY4	edge-of-field	Grab/Siphon	~ 14
NE	NE11	edge-of-field	Grab/Siphon	~ 45
	NE5	edge-of-field	Grab/Siphon	~ 44
	NE1	edge-of-field	Siphon/Auto	~ 50
IA	IA9	edge-of-field	Grab/Siphon	~ 24
	IA10	edge-of-field	Grab/Siphon	~ 23
MN	MN1	edge-of-field	Grab/Siphon	~ 48
MO	MO4	Pond	Grab	~ 60
IN	IN7	Ditch	Grab/Siphon/Auto	~ 55
	IN8	Ditch	Grab/Siphon	~ 66
	IN10	Pond	Grab/Siphon	~ 21
	IN12	edge-of-field	Grab/Siphon	~15
	IN13	edge-of-field	Grab/Siphon	~27
MI	MI3	edge-of-field	Grab/Siphon/Auto	~85
OH	OH3	edge-of-field	Grab/Siphon	~31
	OH4	edge-of-field	Grab/Siphon	~30

SAMPLING AND ANALYTICAL METHODS:

Sampling Methods-

Three sampling methods were used in the study including grab sample, siphon sample, and automatic sample collection. Siphon collection employed a simple siphon bottle with an inlet port for surface water and exhaust port for displaced air. Automatic sampling was conducted using a fraction collector connected to a peristaltic pump. Water samples were taken at triplicate, sequential time intervals of 2, 5, 10, 30, 45 minutes; and 1, 2, and 4 hours.

Analytical Methods-

Analytical methods were designed to determine concentrations of fipronil, MB 45950, MB 46513, and MB 46136. Water samples collected at each site were kept cooled and then shipped via overnight courier to the BASF laboratory. At the laboratory, the samples were frozen and stored (< -5°C) prior to analysis. Samples were initially analyzed using an analytical method by Bayer[1]. The method limit or quantification is 10 ng/L (10 ppt) and the limit of detection is 2 ng/L (2 ppt). Frozen samples were thawed, shaken vigorously and filtered. Acetonitrile was added to each sample prior to chemical analysis. Samples were analyzed using LC/MS/MS in negative ion mode.

BASF modified the Bayer analytical method to account for turbidity of surface waters. Water samples were shaken vigorously prior to sampling. A 5 ml aliquot was removed and then filtered through a 4.5 µ telflon filter. An aliquot of the filtered extract was spiked with acetonitrile and then analyzed using HPLC-MS/MS. LC-MS/MS analysis was based on the following transitions m/z 434.9-330.0 for fipronil, m/z 386.9-351 for MB46513, m/z 418.9 - 383.1 for MB 45950, and m/z 450.9 - 415 for MB46136. Because the Bayer analytical method was modified during study, several samples were not be analyzed using the new BASF method due to destructive sample extraction method. Sample recoveries from fortified surface water at 10 ng/L are as follows: 99±15 % for fipronil, 86± 9 % for MB46513, 82±7 % for MB45950, and 60±4 % for MB46136. Procedural recoveries in HPLC water range from 91 to 104 % for fipronil, 84 to 97 % for MB46513, 71 to 86% for MB45950, and 63 to 82% for MB46136.

Calculation of Compound Edge of Field Loss

Equations used for estimating mass of fipronil loss and % of runoff are as follows:

µg Lost = runoff volume (inches)/12 (inches)* 1233531 L/ac ft* concentration (µg/L)

[1] "Insecticides, Fipronil: Method of Analysis for possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Water-Revision 4" Issued May 21, 2002.

% edge of field loss = µg/ac applied/µg lost* 100 (This equation should be reversed.)

II. RESULTS AND DISCUSSION:

A. Rainfall totals in the month of April were generally below historical rainfall totals for 1971-2000 rainfall data from the Parameter-elevation Regressions on Independent Slopes Model (PRISM grids) (Figure 26). In contrast, rainfall totals in the month of May were above historical rainfall totals. Therefore, monitoring sites planted in April were exposed to lower runoff conditions compared to sites planted in May.

B. Peak runoff concentrations for fipronil residues are shown in Table 2. The maximum edge of field concentration in runoff water was 3600 ng/L for fipronil, 216 ng/L for MB46513, 165 ng/L for MB 45950, and 218 ng/L for MB 46136. The maximum fipronil loss accounted for 0.761% of applied fipronil. Complete data set summaries are shown in Tables 5 to 24.

Table 2: Summary of Maximum Fipronil Residue Runoff Concentration and Total Edge of Field Runoff Percentage

State	Site ID	Fipronil	MB46513	MB 45950	MB 46136	Total edge-of field runoff
		ng/L				% of applied
IL	IL3	<10	<10	<10	<10	0.0
	IL5	<10	<10	<10	<10	0.0
	IL6	65	<10	12	49	0.028
	IL4	1038	<10	<10	12	0.068
KY	KY4	648	14	13	53	0.34
NE	NE11	38	<10	<10	<10	0.013
	NE5	3600	167	165	218	0.761
	NE1	665	21	<10	38	0.152
IA	IA9	226	<10	<10	43	0.35
	IA10	226	< 10	<10	49	0.133
MN	MN1	0	<10	<10	<10	0.0
MO	MO4	159	11	21	61	0.095

IN	IN7	203	29	<10	17	0.027
	IN8	0	<10	<10	24	0.0

	IN10	410	26	<10	39	0.205
	IN12	93	<10	<10	83	0.046
	IN13	308	11	<10	21	0.162
MI	MI3	305	<10	<10	25	0.068
OH	OH3	171	<10	<10	18	0.0199
	OH4	2188	216	17	101	0.127

C. There was no consistent management practice among monitoring sites that appear to increase runoff of fipronil (Table 25). Management practices among the monitoring sites generally consisted of side dressed N fertilization or cultivation for weed control.

D. Fipronil loss generally corresponded to rainfall patterns with highest loss occurring with maximum rainfall amounts (Figures 27 and 43; Figure 46). However, an analysis of time series of fipronil concentrations on short time intervals (0 to 12 hours after runoff event) indicate a sinus curve of fipronil concentration independent of rainfall amounts (Figures 48, 49 and 50). These fluctuations in concentrations appear to correspond to changes in volume and velocity of runoff waters.

F. Several of the monitoring sites (IL3, MO4, IN10) had farm ponds as a collection point for monitoring fipronil residue concentrations. in the ponds. The maximum concentration in pond water was 159 ng/L for fipronil and 24 ng/L for MB46513. MB 45950 and MB 46136 were not detected in the farm pond water.

III. STUDY DEFICIENCIES:

A. The general monitoring study design is excellent. The reviewer notes that a single year of monitoring is not be sufficient to represent variability in rainfall occurrence.

IV. REVIEWER'S COMMENTS:

A. The reviewer complements the registrant's attempt to provide probabilistic context to the edge-of-field monitoring data.

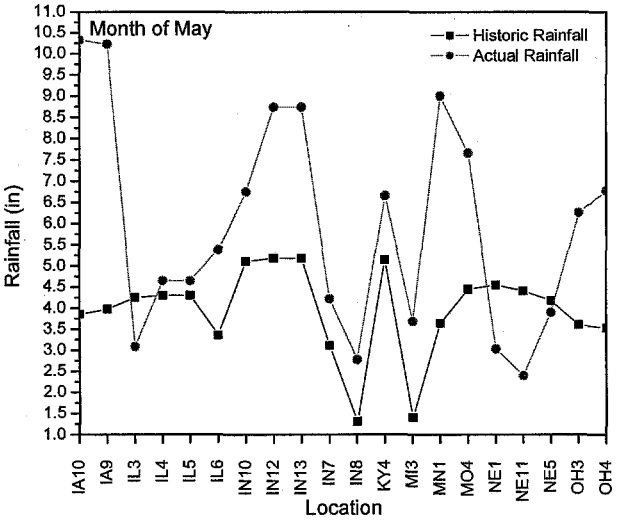

Figure 26. Summary of rainfall during the month of May at the twenty sites.

Sample designation used in the summary tables are summarized below.

1. G1, G2 - grab sample rep 1 and rep 2
2. A, B, C - siphon samples 1st, 2nd, and 3rd (height 1, 2 and 3)
3. B1-B24 - autosampler samples 1 through a maximum of 24
4. P1 - sampling point 1
5. E01 - runoff event 1

Table 5. Fipronil edge of field loss summary for the Iowa 9 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IA9 P1E01G2	73	<10	<10	<10	73	1.65	0.073	0.021	0.021	12,381.6	1.5	24
IA9 P1E02G2	198	<10	<10	15	198	1.1	0.198	0.038	0.038	22,388.6	1.5	24
IA9 P1E03A	183	<10	<10	17			0.183	0.135		79,572.0	1.5	24
IA9 P1E03B	178	<10	<10	18	195	4.23	0.178	0.131	0.144	77,397.9	1.5	24
IA9 P1E03C	224	<10	<10	21			0.224	0.165		97,399.6	1.5	24
IA9 P1E04A	480	<10	<10	33	529	0.88	0.48	0.074	0.081	43,420.3	1.5	24
IA9 P1E04B	578	<10	<10	35			0.578	0.089		52,285.3	1.5	24
IA9 P1E07A	226	<10	<10	43	214.5	0.83	0.226	0.033	0.031	19,282.2	1.5	24
IA9 P1E07B	203	<10	<10	35			0.203	0.029		17,319.8	1.5	24
IA9 P1E08G1	123	<10	13	29	123.00	1.3	0.123	0.028	0.028	16,436.8	1.5	24
IA9 P1E09G1	0	<10	<10	<10	6.5	2.52	0	0.000	0.0065	0.0	1.5	24
IA9 P1E09A	13	<10	<10	<10			0.013	0.006		3,367.5	1.5	24

Soil Texture = silty clay loam
Mean edge of field loss = 0.053%
Total edge of field loss = 0.35%

0.013 - 0.578

Table 6. Fipronil edge of field loss summary for the Iowa 10 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IA10 P1E01G2	130	<10	<10	<10			0.13	0.028		16,303.2		
IA10 P1E01A	225	<10	<10	<10	193.67	1.22	0.225	0.048	0.041	28,217.0	3	23
IA10 P1E01B	226	<10	<10	<10			0.226	0.048		28,342.4		
IA10 P1E02G2	87	<10	<10	<10	53.50	3.88	0.087	0.059	0.036	34,699.2	3	23
IA10 P1E02A	20	<10	<10	<10			0.02	0.014		7,976.8		
IA10 P1E03G2	59	<10	<10	<10	61.50	2.58	0.059	0.027	0.028	15,647.3	3	23
IA10 P1E03C	64	<10	<10	<10			0.064	0.029		16,973.4		
IA10 P1E04G1	29	<10	<10	<10			0.029	0.029		16,991.9		
IA10 P1E04A	29	<10	<10	<10	19.75	5.7	0.029	0.029	0.020	16,991.9	3	23
IA10 P1E04B	0	<10	<10	<10			0	0.000		0.0		
IA10 P1E04C	21	<10	<10	49			0.021	0.021		12,304.5		
IA10 P1E05G1	45	<10	<10	<10			0.045	0.015		8,881.4		
IA10 P1E05B	29	<10	<10	<10	24.67	1.92	0.029	0.010	0.008	5,723.6	3	23
IA10 P1E05C	0	<10	<10	<10			0	0.000		0.0		

Soil Texture = silt loam
Mean edge of field loss = 0.027%
Total edge of field loss = 0.133%

0.021 - 0.226 µg/L

Table 7. Fipronil edge of field loss summary for the Illinois 3 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
p- IL3P1E01G1	0	<10	<10	<10	0.00	2.16	0	0.000	0.000	0.0	6	58

p-pond
Soil Texture = silt loam
Mean edge of field loss = 0.0%
Total edge of field loss = 0.0%

Table 8. Fipronil edge of field loss summary for the Illinois 4 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IL4P1E01A	57	<10	<10	<10			0.057	0.014		8,203.0		
IL4P1E01B	34	<10	<10	<10	34	1.4	0.034	0.008	0.008	4,893.0	2.5	11
IL4P1E01C	11	<10	<10	<10			0.011	0.003		1,583.0		
IL4P2E01A	0	<10	<10	<10			0	0.000		0.0		
IL4P2E01B	15	<10	<10	<10	5	1.4	0.015	0.004	0.001	2,158.7	2.5	7
IL4P2E01C	0	<10	<10	<10			0	0.000		0.0		
IL4P3E01A	565	<10	<10	<10	801.5	1.4	0.565	0.138	0.195	81,310.3	2.5	3
IL4P3E01B	1038	<10	<10	12			1.038	0.253		149,380.7		
								Field Event Average loss	0.068			
IL4P1E02A	0	<10	<10	<10	0	0.32	0	0.000	0.000	0.0	2.5	11
IL4P1E02B	0	<10	<10	<10			0	0.000		0.0		
IL4P3E03A	0	<10	<10	<10	0	1.15	0	0.000	0.000	0.0	2.5	3

Soil Texture = silt loam
Mean edge of field loss = 0.023%
Total edge of field loss = 0.068%

.011 - to - 1.038

Table 9. Fipronil edge of field loss summary for the Illinois 5 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IL5P1E01A	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B	0	<10	<10	<10	0.00	1.4	0	0.000	0.0	0.0	6	12.5
IL5P1E01C	0	<10	<10	<10			0	0.000		0.0		
IL5P2E01A	0	<10	<10	<10			0	0.000		0.0		
IL5P2E01B	0	<10	<10	<10	0.00	0.1	0	0.000	0.0	0.0	6	7
IL5P2E01C	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B1	0	<10	<10	<10	0.00	1.4	0	0.000	0.00	0.0	6	12.5
IL5P1E01B2	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B3	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B4	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B5	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B6	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B7	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B8	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B9	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B10	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B11	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B12	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B13	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B14	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B15	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B16	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B17	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B18	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B19	0	<10	<10	<10			0	0.000		0.0		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IL5P1E01B20	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B21	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B22	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B23	0	<10	<10	<10			0	0.000		0.0		
IL5P1E01B24	0	<10	<10	<10			0	0.000		0.0		
IL5P1E02A	0	<10	<10	<10		0.1	0	0.000		0.0		
IL5P1E02B	0	<10	<10	<10	0.00	0.1	0	0.000	0.0	0.0	6	12.5
IL5P1E02C	0	<10	<10	<10		0.1	0	0.000		0.0		

Soil Texture = silt loam
Mean edge of field loss = 0.0%
Total edge of field loss = 0.0%

Table 10. Fipronil edge of field loss summary for the Illinois 6 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IL6P1E02G2	26	<10	<10	<10	45.50	0.48	0.026	0.002	0.004	1,282.9	1.5	36
IL6P1E02A	65	<10	12	49			0.065	0.005		3,207.2		
IL6P1E03A	41	<10	<10	20			0.041	0.026		15,509.6		
IL6P1E03B	33	<10	<10	26	37.67	3.68	0.033	0.021	0.024	12,483.3	1.5	36
IL6P1E03C	39	<10	<10	27			0.039	0.025		14,753.0		

Soil Texture = silt loam
Mean edge of field loss = 0.014%
Total edge of field loss = 0.028%

1126+1.065

Table 11. Fipronil edge of field loss summary for the Indiana 7 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN7P1E00G2	0	<10	<10	<10	0.00	0	0	0.000	0.000	0.0	1	55
IN7P1E01G2	0	<10	<10	<10	0.00	0.53	0	0.000	0.000	0.0	1	55
IN7P1E02G2	203	29	<10	17	203.00	0.75	0.203	0.027	0.027	15,650.4	1	55
IN7P1E04G2	0	<10	<10	<10	0.00	2.18	0	0.000	0.000	0.0	1	55
IN7P1E07A	0	<10	<10	<10	0.00	2.44	0	0.000	0.000	0.0	1	55
IN7P1E08G1	0	<10	<10	<10	0.00	1.86	0	0.000	0.000	0.0	1	55
IN7P1E09G1	0	<10	<10	<10	0.00	0.69	0	0.000	0.000	0.0	1	55
IN7P1E09A	0	<10	<10	<10	0.00	0.69	0	0.000	0.000	0.0	1	55
IN7P1E10G1	0	<10	<10	<10	0.00	1.4	0	0.000	0.000	0.0	1	55
IN7P1E10A	0	<10	<10	<10	0.00		0	0.000		0.0	1	55

Soil Texture = silt loam
Mean edge of field loss = 0.005%
Total edge of field loss = 0.027%

ave 0.203

Table 12. Fipronil edge of field loss summary for the Indiana 8 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN8P2E02A	0	<10	<10	<10	0.00	2.18	0	0.000		0.0	3	61
IN8P1E02A	0	<10	<10	<10	0.00	2.18	0	0.000		0.0	3	64
IN8P1E02B	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B1	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B2	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B3	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B4	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B5	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B6	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B7	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B8	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B10	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B11	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B12	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B13	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B14	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B15	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B16	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B17	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B18	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B19	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B20	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B21	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B22	0	<10	<10	<10		2.18	0			0.0		
IN8P1E02B23	0	<10	<10	<10		2.18	0			0.0		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN8P1E02B24	0	<10	<10	<10		2.18	0			0.0		
IN8P2E03A	0	<10	<10	12	0.00	0.4	0	0.000		0.0	3	61
IN8P1E04A	0	<10	<10	<10		2.47	0	0.000		0.0	3	64
IN8P1E04B1	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B2	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B3	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B4	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B5	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B6	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B7	0	<10	<10	<10	0.0	2.47	0	0.000		0.0	3	64
IN8P1E04B8	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B9	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B10	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B11	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B12	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B13	0	<10	<10	<10			0	0.000		0.0		
IN8P1E04B14	0	<10	<10	<10			0	0.000		0.0		
IN8P2E04A	0	<10	<10	<10	0.0	2.47	0	0.000		0.0	3	61
IN8P1E04A	0	<10	<10	<10	0.0	2.47	0	0.000		0.0	3	64
IN8P2E05A	0	<10	<10	<10	0.0	1.86	0	0.000		0.0	3	61
IN8P1E05A	0	<10	<10	<10	0.0	1.86	0	0.000		0.0	3	64
IN8P2E06A	0	<10	<10	<10	0.0	0.69	0	0.000		0.0	3	61
IN8P1E06A	0	<10	<10	<10	0.0	0.69	0	0.000		0.0	3	64
IN8P2E07A	0	<10	<10	24	0.0	2	0	0.000		0.0	3	61
IN8P1E07A	0	<10	<10	13	0.0	2	0	0.000		0.0	3	64
IN8P1E07B	0	<10	<10	<10	0.0	2	0	0.000		0.0	3	64

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN8P1E07B1	0	<10	<10	<10	0.0	2	0	0.000		0.0	3	64
IN8P1E07B2	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B3	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B4	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B5	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B6	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B7	0	<10	<10	<10	0.00	2	0	0.000		0.0	3	64
IN8P1E07B8	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B9	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B10	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B11	0	<10	<10	<10			0	0.000		0.0		
IN8P1E07B12	0	<10	<10	<10			0	0.000		0.0		

Soil Texture = sand
Mean edge of field loss = 0.0%
Total edge of field loss = 0.0%

Table 13. Fipronil edge of field loss summary for the Indiana 10 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN10P2E01G2	41	<10	<10	<10	41	0.92	0.041	0.007	0.007	3,877.4	4	9
IN10P3E01G2	116	<10	<10	<10	116	0.92	0.116	0.019	0.019	10,970.2	4	21
							Field Averaged Event loss		0.013			
IN10P2E02G2	137	14	<10	<10	137	3.01	0.137	0.072	0.072	42,389.3	4	9
IN10P3E02G2	410	18	<10	39	410	3.01	0.41	0.215	0.215	126,858.4	4	21
							Field Averaged Event loss		0.18			
IN10P2E03G1	0	<10	<10	<10			0	0.000		0.0		
IN10P2E03A	40	<10	<10	<10	23.3	3.38	0.04	0.024	0.014	13,897.8	4	9
IN10P2E03B	30	<10	<10	<10			0.03	0.018		10,423.3		
IN10P3E03A	28	<10	<10	<10	21.67	3.38	0.028	0.016	0.015	9,728.5		
IN10P3E03B	24	<10	<10	<10		3.38	0.024	0.014		8,338.7	4	21
IN10P3E03G1	13	26	<10	<10	13	3.38	0.013	0.008	0.008	4,516.8		
							Field Averaged Event loss		0.012			
pIN10P1E03G1	0	24	<10	<10	0	3.38	0	0.000	0	0.0	4	6

p - pond
Soil Texture = silt loam
Mean edge of field loss = 0.068%
Total edge of field loss = 0.205%

Table 14. Fipronil edge of field loss summary for the Indiana 12 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN12P1E06A	30	<10	<10	16			0.03	0.011		6,506.9		
IN12P1E06B	93	<10	<10	83	55.33	2.11	0.093	0.034	0.017	20,171.3	8.5	15
IN12P1E06C	43	<10	<10	18			0.043	0.016		9,326.5		
IN12P1E07A	60	<10	<10	<10	30.00	0.73	0.06	0.008	0.004	4,502.4	8.5	15
IN12P1E07B	0	<10	<10	<10			0	0.000		0.0		
IN12P1E08A	29	<10	<10	<10			0.029	0.006		3,279.1		
IN12P1E08B	23	<10	<10	<10	25.67	1.1	0.023	0.004	0.005	2,600.7	8.5	15
IN12P1E08C	25	<10	<10	<10			0.025	0.005		2,826.8		
IN12P1E09A	30	<10	<10	<10			0.03	0.008		4,656.6		
IN12P1E09B	13	<10	<10	<10	26.00	1.51	0.013	0.003	0.007	2,017.9	8.5	15
IN12P1E09C	35	<10	<10	<10			0.035	0.009		5,432.7		
IN12P1E10A	42	<10	<10	<10			0.042	0.006		3,324.4		
IN12P1E10B	42	<10	<10	<10	37.67	0.77	0.042	0.006	0.005	3,324.4	8.5	15
IN12P1E10C	29	<10	<10	<10			0.029	0.004		2,295.4		
IN12P1E11A	16	<10	<10	17			0.016	0.002		1,167.7		
IN12P1E11B	20	<10	<10	31	16.33	0.71	0.02	0.002	0.002	1,459.7	8.5	15
IN12P1E11C	13	<10	<10	20			0.013	0.002		948.8		
IN12P1E12A	22	<10	<10	<10			0.022	0.003		1,854.4		
IN12P1E12B	11	<10	<10	<10	16.00	0.82	0.011	0.002	0.002	927.2	8.5	15
IN12P1E12C	15	<10	<10	<10			0.015	0.002		1,264.4		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN12P1E13G1	16	<10	<10	<10			0.016	0.008		4,934.1		
IN12P1E13A	0	<10	<10	<10	7.50	3	0	0.000	0.004	0.0	8.5	15
IN12P1E13B	0	<10	<10	<10			0	0.000		0.0		
IN12P1E13C	14	<10	<10	<10			0.014	0.007		4,317.4		

Soil Texture = silt loam
Mean edge of field loss = 0.006%
Total edge of field loss = 0.046%

Table 15. Fipronil edge of field loss summary for the Indiana 13 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
IN13P2E02C	308	11	<10	<10	308.00	0.75	0.308	0.040	0.040	23,745.5	3.5	13
IN13P1E05A	0	<10	<10	<10	0.00	0.89	0	0.000	0.0	0.0	3.5	13
IN13P1E06A	154	<10	<10	11	154.00	2.11	0.154	0.057	0.057	33,402.0	3.5	13
IN13P1E07A	44	<10	<10	21	44.00	1.1	0.044	0.008	0.008	4,975.2	3.5	13
IN13P1E08A	95	<10	<10	<10	95.00	1.51	0.095	0.025	0.025	14,745.8	3.5	13
IN13P2E08A	49	<10	<10	<10	49.00		0.049	0.013	0.013	7,605.7		
							Field Event Average loss		0.019			
IN13P2E09A	122	<10	<10	12	122.00	0.77	0.122	0.016	0.016	9,656.5	3.5	13
IN13P1E10A	34	<10	<10	<10	34.00	0.82	0.034	0.005	0.005	2,865.9	3.5	13
IN13P1E11A	31	<10	<10	<10	31.50	3	0.031	0.016	0.017	9,559.9	3.5	13
IN13P1E11B	32	<10	<10	<10			0.032	0.017		9,868.3		
IN13P2E11A	29	<10	<10	<10	32.00	3	0.029	0.015	0.017	8,943.1	3.5	13
IN13P2E11B	35	<10	<10	<10			0.035	0.018		10,793.4	3.5	13
							Field Event Average loss		0.017			

Soil Texture = silt loam
Mean edge of field loss = 0.020%
Total edge of field loss = 0.162%

Table 16. Fipronil edge of field loss summary for the Kentucky 4 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
KY4P1E01G2	648	<10	<10	10	648.00	2.52	0.648	0.284	0.284	167,859.0	4	4.5
KY4P1E02G1	52	14	<10	<10	52.00	0.55	0.052	0.005	0.005	2,939.9	4	4.5
KY4P1E03G1	89	<10	13	43	89.00	0.9	0.089	0.014	0.014	8,233.8	4	4.5
KY4P2E03G1	72	<10	11	32	72.00	0.9	0.072	0.011	0.011	6,661.1	4	4
KY4P3E03G1	53	<10	<10	35	53.00	0.9	0.053	0.008	0.008	4,903.3	4	2
							Field Event Average loss		0.011			
KY4P1E04G1	112	<10	12	53	112.00	2.1	0.112	0.041	0.041	24,177.2	4	4.5
KY4P2E04G1	111	10	11	52	111.00	2.1	0.111	0.041	0.041	23,961.3	4	4
KY4P3E04G1	126	<10	<10	29	126.00	2.1	0.126	0.046	0.046	27,199.4	4	2
							Field Event Average loss		0.043			

Soil Texture = silt loam
Mean edge of field loss = 0.086%
Total edge of field loss = 0.34%

Table 17. Fipronil edge of field loss summary for the Michigan 3 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
MI3P2E01G2	305	<10	<10	11	305.00	1.85	0.305	0.098	0.098	58,001.7	1.5	60
MI3P1E01B1	101	<10	<10	<10			0.101	0.033		19,207.1		
MI3P1E01B2	49	<10	<10	<10			0.049	0.016		9,318.3		
MI3P1E01B3	97	<10	<10	11			0.097	0.031		18,446.4		
MI3P1E01B4	48	<10	<10	<10			0.048	0.015		9,128.1		
MI3P1E01B5	27	<10	<10	<10	67.44	1.85	0.027	0.009	0.022	5,134.6	1.5	26
MI3P1E01B6	151	<10	<10	<10			0.151	0.049		28,715.6		
MI3P1E01B7	59	<10	<10	<10			0.059	0.019		11,220.0		
MI3P1E01B8	42	<10	<10	<10			0.042	0.014		7,987.1		
MI3P1E01B9	33	<10	<10	<10			0.033	0.011		6,275.6		
							Field Event Average loss		0.060			
MI3P2E02A	114	<10	<10	11	164.93	1.18	0.114	0.023	0.016	13,827.9	1.5	60
MI3P2E02B	39	<10	<10	<10			0.039	0.008		4,730.6		
MI3P1E02B1	0	<10	<10	15			0	0.000		0.0		
MI3P1E02B2	0	<10	<10	14			0	0.000		0.0		
MI3P1E02B3	31	<10	<10	<10			0.031	0.006		3,760.2		
MI3P1E02B4	44	<10	<10	13	24.86	1.18	0.044	0.009	0.005	5,337.1	1.5	26
MI3P1E02B5	21	<10	<10	<10			0.021	0.004		2,547.2		
MI3P1E02B6	47	<10	<10	11			0.047	0.010		5,701.0		
MI3P1E02B7	31	<10	<10	<10			0.031	0.006		3,760.2		
							Field Event Average loss		0.011			

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
MI3P2E03A	61	<10	<10	25		0.7	0.061	0.007		4,389.3		
MI3P2E03B	53	<10	<10	<10	72.33	0.7	0.053	0.006	0.009	3,813.7	1.5	60
MI3P2E03C	103	<10	<10	11		0.7	0.103	0.013		7,411.5		
MI3P2E04A	17	<10	<10	<10		0.5	0.017	0.001	0.001	873.8	1.5	60
MI3P2E05A	26	<10	<10	<10	13.00	1	0.026	0.005	0.025	2,672.7	1.5	60
MI3P2E05B	0	<10	<10	<10		1	0	0.000		0.0		

Soil Texture = sandy loam
Mean edge of field loss = 0.0136%
Total edge of field loss = 0.068%

Table 18. Fipronil edge of field loss summary for the Minnesota 1 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
MN1P1E01G1	0	<10	<10	<10	0.00	2	0	0.000	0.0	0.0	6	26

Soil Texture = silt loam
Mean edge of field loss = 0.0%
Total edge of field loss = 0.0%

Table 19. Fipronil edge of field loss summary for the Missouri 4 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
p-MO4P1E01G2	0	<10	<10	<10	0.00	0.69	0	0.000	0.000	0.0	6	60
p MO4P1E02G2	12	<10	<10	<10	12.00	0.83	0.012	0.002	0.002	1,023.8	6	60
p MO4P1E03G2	0	<10	<10	<10	0.00	1.44	0	0.000	0.000	0.0	6	60
p MO4P1E04G2	159	<10	<10	<10	159.00	0.85	0.159	0.024	0.024	13,892.6	6	60
p MO4P1E08G1	158	11	21	61	158.00	1.71	0.158	0.047	0.047	27,773.0	6	60
p MO4P1E09G1	130	<10	13	56	130.00	0.93	0.13	0.021	0.021	12,427.8	6	60
p MO4P1E10G1	12	<10	14	12	12.00	0.67	0.012	0.001	0.001	826.5	6	60

p- pond outflow
Soil Texture = silt loam
Mean edge of field loss = 0.014%
Total edge of field loss = 0.095%

Table 20. Fipronil edge of field loss summary for the Nebraska 1 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE1P1E01G1	244	<10	<10	25	152.50	3.04	0.244	0.129	0.081	76,248.7	1	60
NE1P1E01G2	61	<10	<10	<10			0.061	0.032		19,062.2		
NE1P1E01A	665	<10	<10	32	269.33	3.04	0.665	0.352	0.143	207,808.9	1	20
NE1P1E01B	98	<10	<10	<10			0.098	0.052		30,624.5		
NE1P1E01C	45	<10	<10	<10			0.045	0.024		14,062.3		
NE1P1E01B1	42	<10	<10	<10	19.58	3.04	0.042	0.022	0.0103	13,124.8	1	20
NE1P1E01B2	45	<10	<10	<10		3.04	0.045	0.024		14,062.3		
NE1P1E01B3	31	<10	<10	<10		3.04	0.031	0.016		9,687.3		
NE1P1E01B4	26	<10	<10	<10		3.04	0.026	0.014		8,124.9		
NE1P1E01B5	21	<10	<10	<10		3.04	0.021	0.011		6,562.4		
NE1P1E01B6	23	<10	<10	<10		3.04	0.023	0.012		7,187.4		
NE1P1E01B7	0	<10	<10	<10		3.04	0	0.000		0.0		
NE1P1E01B8	12	<10	<10	<10		3.04	0.012	0.006		3,749.9		
NE1P1E01B9	30	<10	<10	<10		3.04	0.03	0.016		9,374.8		
NE1P1E01B10	44	<10	<10	<10		3.04	0.044	0.023		13,749.8		
NE1P1E01B11	22	<10	<10	<10		3.04	0.022	0.012		6,874.9		
NE1P1E01B12	24	<10	<10	<10		3.04	0.024	0.013		7,499.9		
NE1P1E01B13	24	<10	<10	<10		3.04	0.024	0.013		7,499.9		
NE1P1E01B14	16	<10	<10	<10		3.04	0.016	0.008		4,999.9		
NE1P1E01B15	18	<10	<10	<10		3.04	0.018	0.010		5,624.9		
NE1P1E01B16	14	<10	<10	<10		3.04	0.014	0.007		4,374.9		
NE1P1E01B17	12	<10	<10	<10		3.04	0.012	0.006		3,749.9		
NE1P1E01B18	14	<10	<10	<10		3.04	0.014	0.007		4,374.9		
NE1P1E01B19	11	<10	<10	<10		3.04	0.011	0.006		3,437.4		
NE1P1E01B20	12	<10	<10	<10		3.04	0.012	0.006		3,749.9		
NE1P1E01B21	0	<10	<10	<10		3.04	0	0.000		0.0		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE1P1E01B22	0	<10	<10	<10		3.04	0	0.000		0.0		
NE1P1E01B23	14	<10	<10	<10		3.04	0.014	0.007		4,374.9		
NE1P1E01B24	15	<10	<10	<10		3.04	0.015	0.008		4,687.4		
							Field Event Average loss		0.0781			
NE1P1E02B1	318	<10	<10	11			0.318	0.061		36,284.3		
NE1P1E02B2	463	21	<10	22			0.463	0.090		52,829.1		
NE1P1E02B3	83	17	<10	21			0.083	0.016		9,470.4		
NE1P1E02B4	540	13	<10	18			0.54	0.104		61,614.9		
NE1P1E02B5	358	<10	<10	15			0.358	0.069		40,848.4		
NE1P1E02B6	430	<10	<10	11			0.43	0.083		49,063.7		
NE1P1E02B7	383	<10	<10	15			0.383	0.074		43,700.9		
NE1P1E02B8	244	<10	<10	<10			0.244	0.047		27,840.8		
NE1P1E02B9	234	<10	<10	<10			0.234	0.045		26,699.8		
NE1P1E02B10	288	<10	<10	<10	341.50	1.11	0.288	0.056	0.0659	32,861.3	1	20
NE1P1E02B11	310	<10	<10	<10			0.31	0.060		35,371.5		
NE1P1E02B12	378	<10	<10	<10			0.378	0.073		43,130.4		
NE1P1E02B13	353	<10	<10	11			0.353	0.068		40,277.9		
NE1P1E02B14	508	<10	<10	18			0.508	0.098		57,963.6		
NE1P1E02B15	393	<10	<10	13			0.393	0.076		44,842.0		
NE1P1E02B16	223	<10	<10	<10			0.223	0.043		25,444.7		
NE1P1E02B17	333	<10	<10	<10			0.333	0.064		37,995.9		
NE1P1E02B18	333	<10	<10	13			0.333	0.064		37,995.9		
NE1P1E02B19	315	12	<10	<10			0.315	0.061		35,942.0		
NE1P1E02B20	343	15	<10	38			0.343	0.066		39,136.9		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE1P1E03B1	13	<10	<10	<10			0.013	0.002		1,336.3		
NE1P1E03B2	23	<10	<10	<10			0.023	0.004		2,364.3		
NE1P1E03B3	19	<10	<10	<10			0.019	0.003		1,953.1		
NE1P1E03B4	13	<10	<10	<10			0.013	0.002		1,336.3		
NE1P1E03B5	18	<10	<10	<10			0.018	0.003		1,850.3		
NE1P1E03B6	19	<10	<10	<10			0.019	0.003		1,953.1		
NE1P1E03B7	30	<10	<10	<10			0.03	0.005		3,083.8		
NE1P1E03B8	20	<10	<10	<10	17.75	1	0.02	0.003	0.003	2,055.9	1	20
NE1P1E03B9	23	<10	<10	<10			0.023	0.004		2,364.3		
NE1P1E03B10	11	<10	<10	11			0.011	0.002		1,130.7		
NE1P1E03B11	15	<10	<10	<10			0.015	0.003		1,541.9		
NE1P1E03B12	12	<10	<10	<10			0.012	0.002		1,233.5		
NE1P1E03B13	12	<10	<10	<10			0.012	0.002		1,233.5		
NE1P1E03B14	14	<10	<10	<10			0.014	0.002		1,439.1		
NE1P1E03B15	17	<10	<10	<10			0.017	0.003		1,747.5		
NE1P1E03B16	25	<10	<10	<10			0.025	0.004		2,569.9		
NE1P1E04B1	0	<10	<10	<10	0.00		0	0.000	0.00	0.0	1	20
NE1P1E04B2	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B3	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B4	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B5	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B6	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B7	0	<10	<10	<10		1	0	0.000		0.0		
NE1P1E04B8	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B9	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B10	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B11	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B12	0	<10	<10	<10			0	0.000		0.0		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE1P1E04B13	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B14	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B15	0	<10	<10	<10			0	0.000		0.0		
NE1P1E04B16	0	<10	<10	<10			0	0.000		0.0		
NE1P1E05B1	0	<10	<10	<10			0	0.000		0.0		
NE1P1E05B2	0	<10	<10	<10			0	0.000		0.0		
NE1P1E05B3	0	<10	<10	<10	0.00	1	0	0.000	0.0	0.0	1	20
NE1P1E05B4	0	<10	<10	<10			0	0.000		0.0		
NE1P1E05B5	0	<10	<10	<10			0	0.000		0.0		
NE1P1E06B1	0	<10	<10	<10	5.50	1	0	0.000	0.001	0.0	1	20
NE1P1E06B2	11	<10	<10	<10			0.011	0.002		1,130.7		
NE1P1E07B1	17	<10	<10	<10			0.017	0.003		1,747.5		
NE1P1E07B2	16	<10	<10	<10			0.016	0.003		1,644.7		
NE1P1E07B3	15	<10	<10	<10			0.015	0.003		1,541.9		
NE1P1E07B4	15	<10	<10	<10			0.015	0.003		1,541.9		
NE1P1E07B5	17	<10	<10	<10			0.017	0.003		1,747.5		
NE1P1E07B6	15	<10	<10	<10	15.73	1	0.015	0.003	0.003	1,541.9	1	20
NE1P1E07B7	11	<10	<10	<10			0.011	0.002		1,130.7		
NE1P1E07B8	15	<10	<10	<10			0.015	0.003		1,541.9		
NE1P1E07B9	15	<10	<10	<10			0.015	0.003		1,541.9		
NE1P1E07B10	19	<10	<10	<10			0.019	0.003		1,953.1		
NE1P1E07B11	18	<10	<10	<10			0.018	0.003		1,850.3		

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE1P1E08A	0	<10	<10	<10		1	0	0.000		0.0	1	20
NE1P1E08B1	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B2	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B3	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B4	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B5	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B6	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B7	0	<10	<10	<10	1.93	1	0	0.000	0.000	0.0	1	20
NE1P1E08B8	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B9	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B10	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B11	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B12	0	<10	<10	<10			0	0.000		0.0		
NE1P1E08B13	14	<10	<10	<10			0.014	0.002		1,439.1		
NE1P1E08B14	13	<10	<10	<10			0.013	0.002		1,336.3		
							Field Event Weighted loss		0.00			
NE1P1E09B1	0	<10	<10	<10			0	0.000		0.0		
NE1P1E09B2	0	<10	<10	<10			0	0.000		0.0		
NE1P1E09B3	0	<10	<10	<10			0	0.000		0.0		
NE1P1E09B4	11	<10	<10	<10			0.011	0.002		1,130.7		
NE1P1E09B5	12	<10	<10	<10	1.93	1	0.012	0.002	0.001	1,233.5	1	20
NE1P1E09B6	0	<10	<10	<10			0	0.000		0.0		
NE1P1E09B7	0	<10	<10	<10			0	0.000		0.0		
NE1P1E09B8	11	<10	<10	<10			0.011	0.002		1,130.7		
NE1P1E09B9	10	<10	<10	<10			0.01	0.002		1,027.9		

Soil Texture = silt loam
Mean edge of field loss = 0.017%
Total edge of field loss = 0.152%

Table 21. Fipronil edge of field loss summary for the Nebraska 5 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE5P2E01A	3600	167	435	218			3.6	1.317		777,124.8		
NE5P2E01B	1338	37	24	73	1927.33	2.1	1.338	0.489	0.705	288,831.4	3	12
NE5P2E01C	844	24	10	25			0.844	0.309		182,192.6		
NE5P2E02G1	13	<10	<10	<10			0.013	0.003		1,737.2		
NE5P2E02A	450	50	165	69	248.00	1.3	0.45	0.102	0.056	60,134.7	3	12
NE5P2E02B	31	<10	<10	<10			0.031	0.007		4,142.6		
NE5P2E02C	498	<10	<10	21			0.498	0.113		66,549.0		
NE5P2E03G1	0	<10	<10	<10			0	0.000		0.0		
NE5P2E03A	0	<10	<10	<10	0.00	0.2	0	0.000	0.0	0.0	3	12
NE5P2E03B	0	<10	<10	<10			0	0.000		0.0		

Soil Texture = silt loam
Mean edge of field loss = 0.253%
Total edge of field loss = 0.761%

Table 22. Fipronil edge of field loss summary for the Nebraska 11 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
NE11P1E01G1	29	<10	<10	<10			0.029	0.015		8,883.5		
NE11P1E01A	(37)	<10	<10	<10	25.5		0.037	0.019	0.013	11,334.1		45
NE11P1E01B	10	<10	<10	<10			0.01	0.005		3,063.3	0.75	
NE11P1E01C	26	<10	<10	<10		2.98	0.026	0.013		7,964.5		
NE11P2E01G1	0	<10	<10	<10	0.00		0	0.000	0.0	0.0		45
NE11P2E01A	0	<10	<10	<10	0.00		0	0.000		0.0	0.75	45
							Field Event Average loss		0.007			
NE11P1E02G1	(38)	<10	<10	<10			0.038	0.007		4,414.0		
NE11P1E02A	28	<10	<10	<10	29.67	1.13	0.028	0.006	0.006	3,252.4	0.75	45
NE11P1E02B	23	<10	<10	<10			0.023	0.005		2,671.6		

Soil Texture = silt loam
Mean edge of field loss = 0.007%
Total edge of field loss = 0.013%

Table 23. Fipronil edge of field loss summary for the Ohio 3 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
OH3P1E01G2	171	<10	<10	<10	171.00	1.16	0.171	0.035	0.035	20,390.3	3	31
OH3P1E02G2	73	<10	<10	<10	73.00	0.8	0.073	0.010	0.010	6,003.2		
OH3P1E03G2	56	<10	<10	<10	56.00	1.55	0.056	0.015	0.015	8,922.5		
OH3P1E05A	118	<10	<10	18	118.00	0.9	0.118	0.018	0.0195	10,916.8	3	31
OH3P1E05B	136	<10	<10	18	136.00		0.136	0.021		12,582.0		

Soil Texture = silt loam
Mean edge of field loss = 0.007%
Total edge of field loss = 0.0199%

Table 24. Fipronil edge of field loss summary for the Ohio 4 site.

Sample ID	FIPRONIL (ppt)	MB 46513 (ppt)	MB 45950 (ppt)	MB 46136 (ppt)	Sample Avg. (ug)	Rain Event (in)	Concentration (ug/L)	%Loss	Average % Loss	ug Lost	Slope (%)	Drainage Area (ac)
OH4P1E01G2	2188	216	17	101	2188.00	0.88	2.188	0.335	0.335	197,924.2	3	30
OH4P1E02G2	355	<10	<10	<10	355.00	0.42	0.355	0.026	0.026	15,326.6		
OH4P1E04A	125	31	<10	21			0.125	0.016		9,380.0		
OH4P1E04B	136	23	<10	16	166.00	0.73	0.136	0.017	0.021	10,205.4	3	30
OH4P1E04C	237	<10	12	26			0.237	0.030		17,784.4		

Soil Texture = fine sandy loam
Mean edge of field loss = 0.042%
Total edge of field loss = 0.127%

SAMPLING AND ANALYTICAL METHODS:

Sampling Methods-

Three sampling methods were used in the study including grab sample, siphon sample, and automatic sample collection. Siphon collection employed a simple siphon bottle with an inlet port for surface water and exhaust port for displaced air. Automatic sampling was conducted using a fraction collector connected to a peristaltic pump. Water samples were taken at triplicate, sequential time intervals of 2, 5, 10, 30, 45 minutes; and 1, 2, and 4 hours.

Analytical Methods-

Analytical methods were designed to determine concentrations of fipronil, MB 45950, MB 46513, and MB 46136. Water samples collected at each site were kept cooled and then shipped via overnight courier to the BASF laboratory. At the laboratory, the samples were frozen and stored (< -5°C) prior to analysis. Samples were initially analyzed using an analytical method by Bayer[1]. The method limit or quantification is 10 ng/L (10 ppt) and the limit of detection is 2 ng/L (2 ppt). Frozen samples were thawed, shaken vigorously and filtered. Acetonitrile was added to each sample prior to chemical analysis. Samples were analyzed using LC/MS/MS in negative ion mode.

BASF modified the Bayer analytical method to account for turbidity of surface waters. Water samples were shaken vigorously prior to sampling. A 5 ml aliquot was removed and then filtered through a 4.5 µ telflon filter. An aliquot of the filtered extract was spiked with acetonitrile and then analyzed using HPLC-MS/MS. LC-MS/MS analysis was based on the following transitions m/z 434.9-330.0 for fipronil, m/z 386.9-351 for MB46513, m/z 418.9 - 383.1 for MB 45950, and m/z 450.9 - 415 for MB46136. Because the Bayer analytical method was modified during study, several samples were not be analyzed using the new BASF method due to destructive sample extraction method. Sample recoveries from fortified surface water at 10 ng/L are as follows: 99±15% for fipronil, 86± 9% for MB46513, 82±7 for MB45950, and 60±4 for MB46136.

Calculation of Compound Edge of Field Loss

Equations used for estimating mass of fipronil loss and % of runoff are as follows:

µg Lost = runoff volume (inches)/12 (inches)* 1233531 L/ac ft* concentration (µg/L)

% edge of field loss = µg/ac applied/µg lost* 100 (This equation should be reversed.)

[1] "Insecticides, Fipronil: Method of Analysis for possible Residues of Fipronil, MB46513, MB45950, and MB46136 in Water-Revision 4" Issued May 21, 2002.

In **Table 25**, cultural practices that may have contributed to fipronil runoff have been listed. Based on total and average field loss, soil disturbance (Cultural practices) did not appear to produce any general trend that could be simply summarized.

Table 25. Summary of cultural practices having soil disturbance impacts.

Site ID	Post-plant Soil Disturbance?	Comments
IA9	None.	
IA10	None.	
IL3	None.	
IL4	None.	
IL5	None.	
IL6	None.	
IN10	Nitrogen side dressing once during season.	Very little soil disturbance
IN12	Nitrogen side dressing 6 to 8 weeks after planting.	Very little soil disturbance
IN13	Nitrogen side dressing 6 to 8 weeks after planting.	Very little soil disturbance
IN7	Cultivated for weeds and nitrogen side dressed at the end of the second week of June .	
IN8	None.	
KY4	None.	
MI3	Rotary hoed 2 to 3 times during the season. Nitrogen side dressing once.	They had so much rain they had to break up the crust.
MN1	None.	
MO4	None.	
NE1	Cultivated once for weeds -beginning of June. Hilled for irrigation rows - end of June.	Hilling piles the soil up around the corn.
NE11	Cultivated once for weeds -beginning of June. Hilled for irrigation rows - end of June.	Hilling piles the soil up around the corn.
NE5	Cultivated once for weeds.	
OH3	Nitrogen side dressing once during season.	Very little soil disturbance
OH4	None.	

The edge of field loss presented in **Tables 5 - 24** have been plotted by event with the rainfall amount associated with the event. The results of plotting this data can be found in **Figures 27 - 42**. **Figure 43** is a graph of water concentration in the MO4 pond site. **Figure 44** is a probability distribution of fipronil edge of field loss based on all events, and **Figure 45** is a probability distribution of fipronil edge of field loss based on total field loss. **Table 29** is a summary of runoff events by field presented in **Figure 45**.

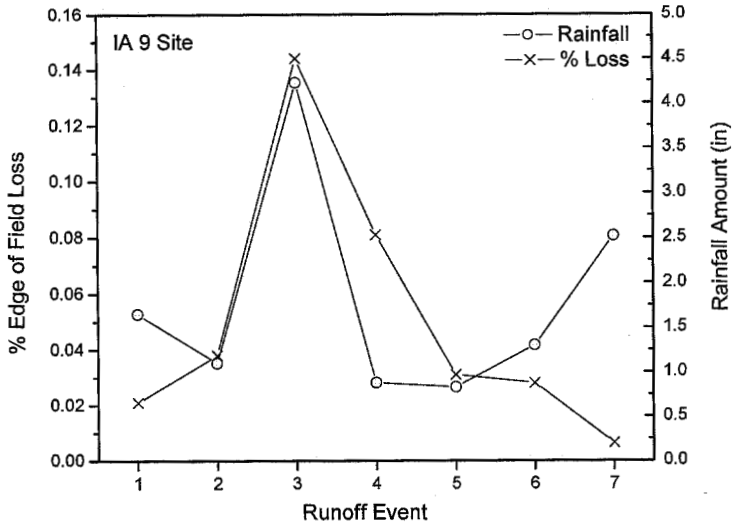

Figure 27 Fipronil edge of field loss for each rainfall event at the Iowa 9 site.

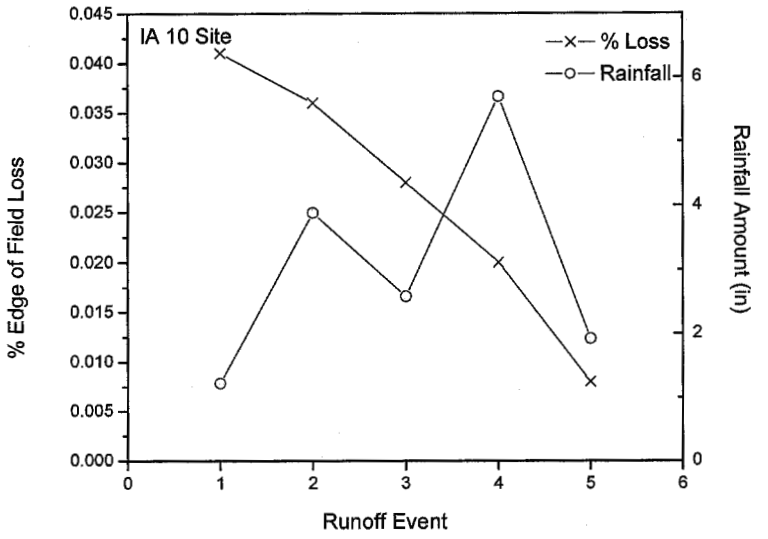

Figure 28 Fipronil edge of field loss for each rainfall event at the Iowa 10 site.

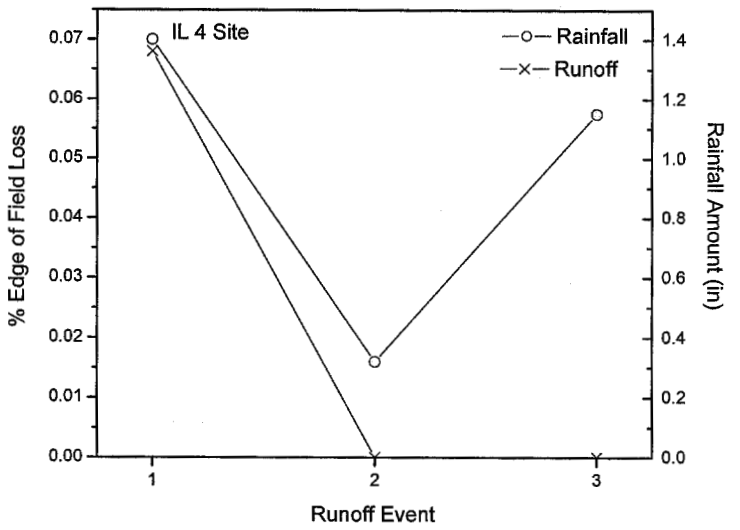

Figure 29 Fipronil edge of field loss for each rainfall event at the Illinois 4 site.

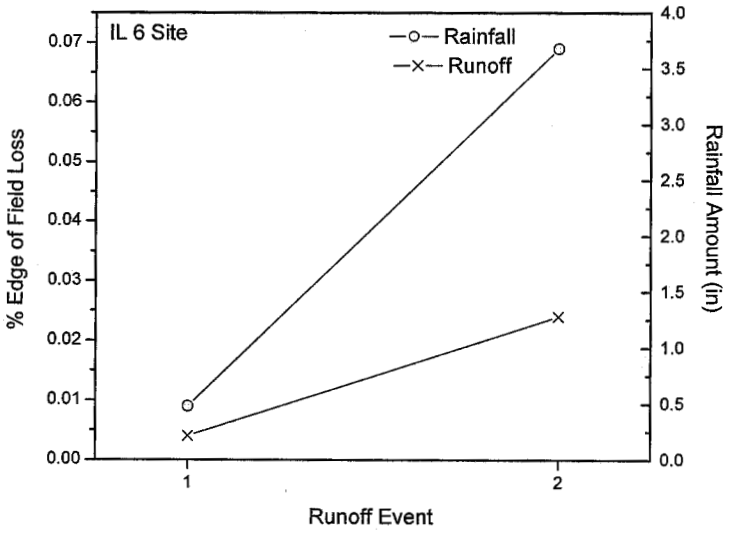

Figure 30 Fipronil edge of field loss for each rainfall event at the Illinois 6 site.

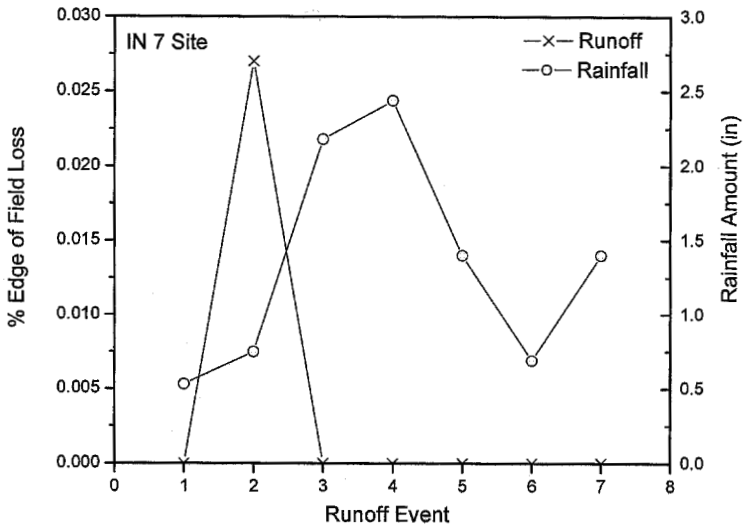

Figure 31 Fipronil edge of field loss for each rainfall event at the Indiana 7 site.

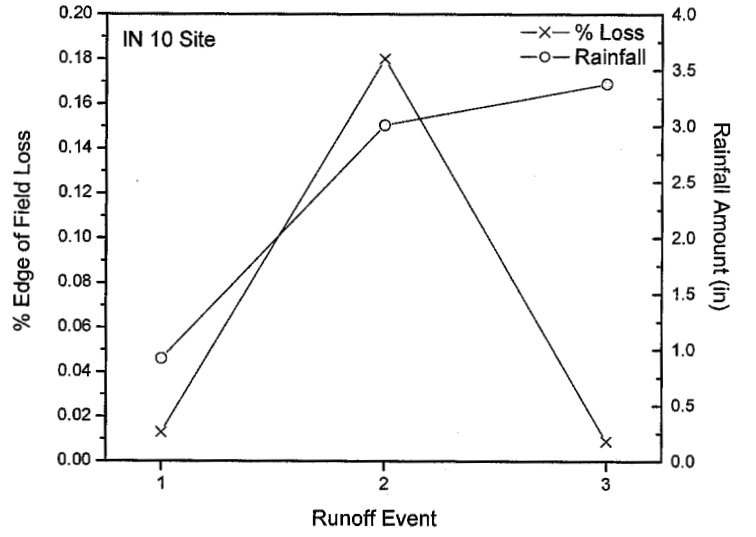

Figure 32 Fipronil edge of field loss for each rainfall event at the Indiana 10 site.

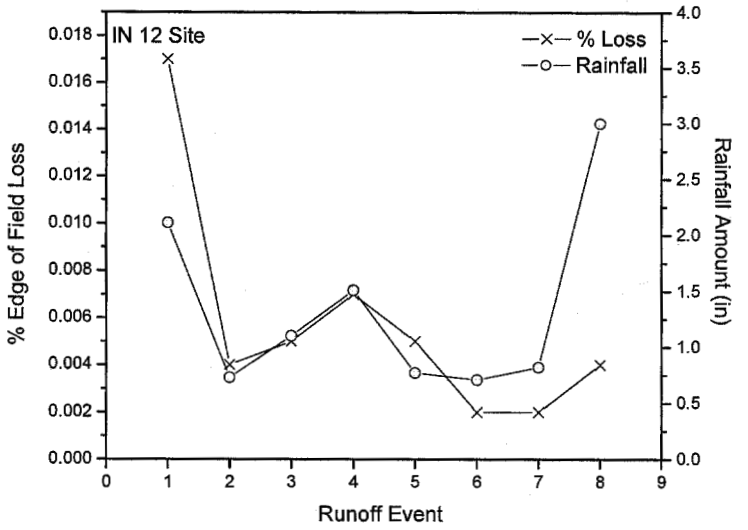

Figure 33 Fipronil edge of field loss for each rainfall event at the Indiana 12 site.

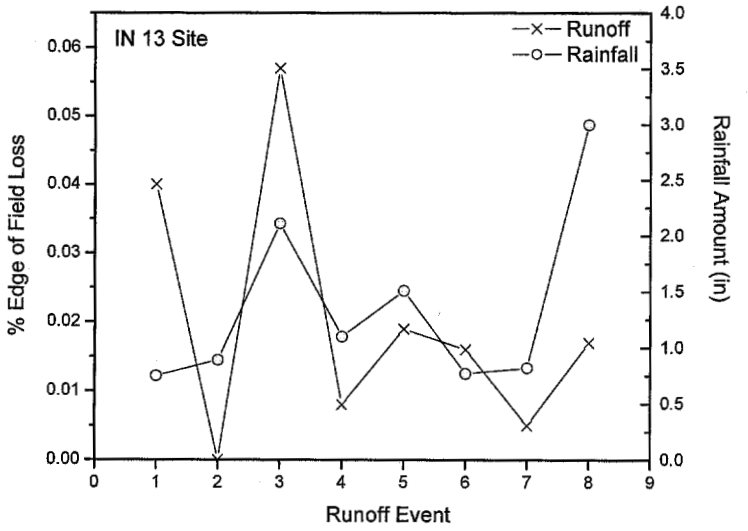

Figure 34 Fipronil edge of field loss for each rainfall event at the Indiana 13 site.

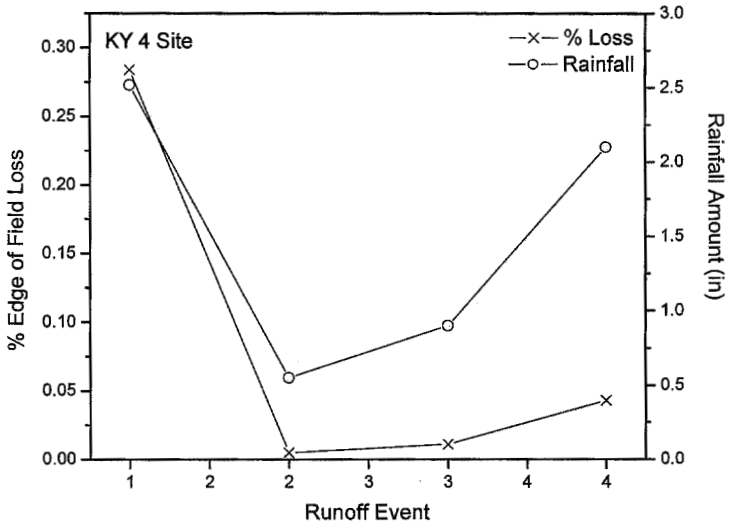

Figure 35 Fipronil edge of field loss for each rainfall event at the Kentucky 4 site.

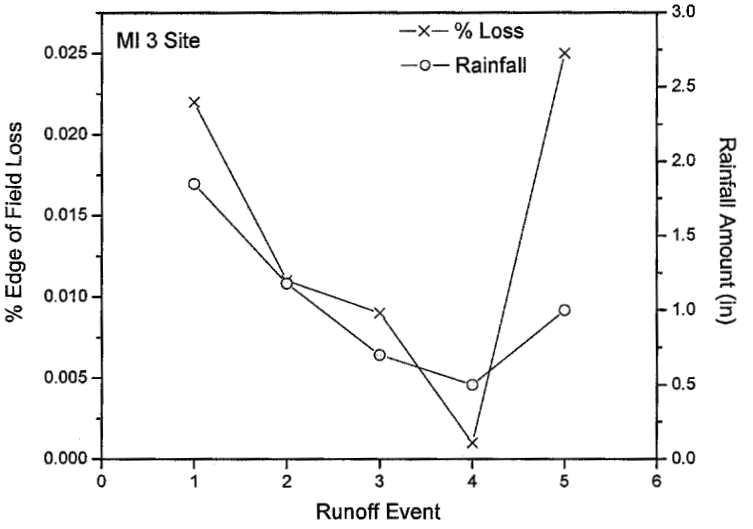

Figure 36 Fipronil edge of field loss for each rainfall event at the Michigan 3 site.

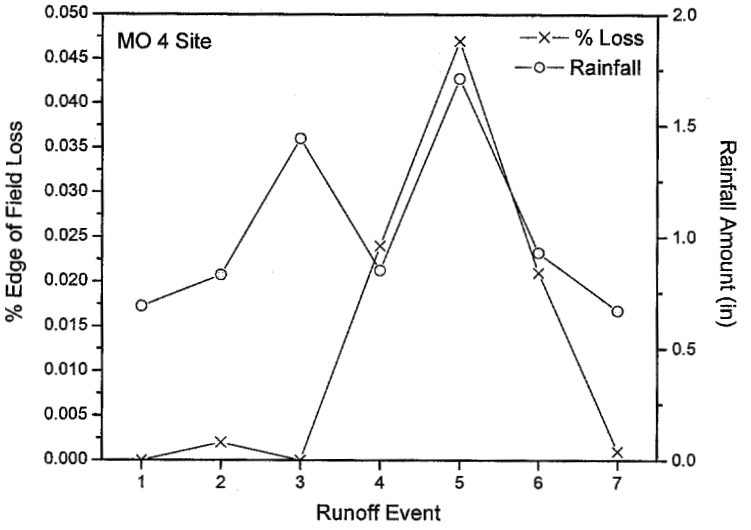

Figure 37 Fipronil edge of field loss for each rainfall event at the Missouri 4 site.

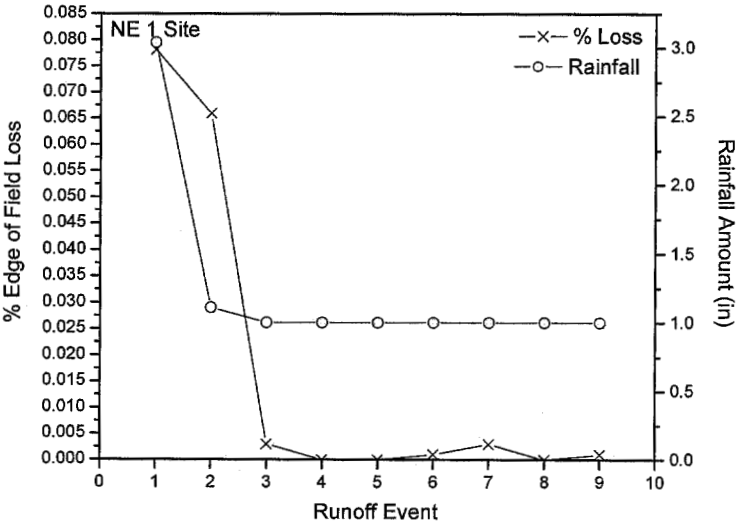

Figure 38 Fipronil edge of field loss for each rainfall event at the Nebraska 1 site.

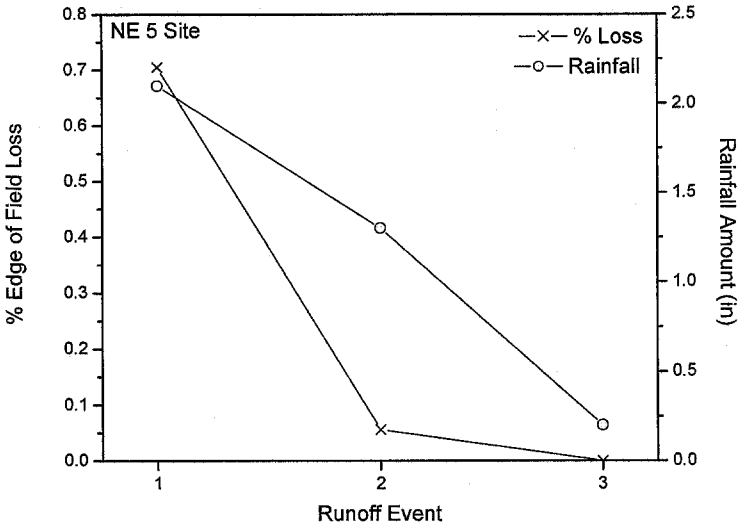

Figure 39 Fipronil edge of field loss for each rainfall event at the Nebraska 5 site.

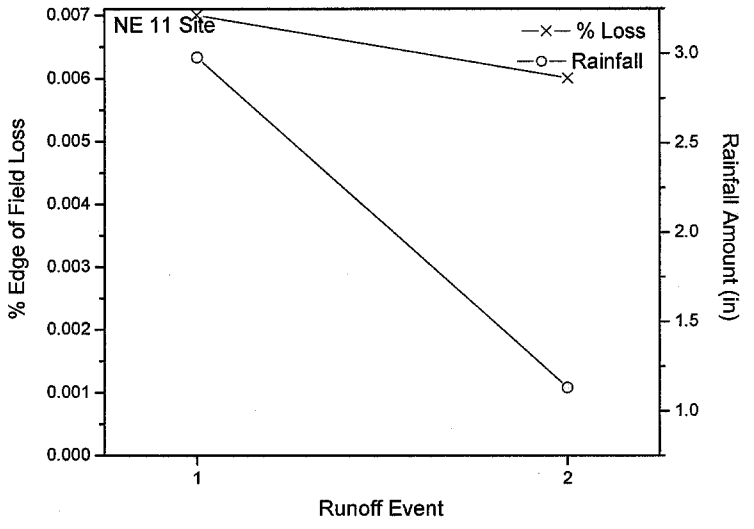

Figure 40 Fipronil edge of field loss for each rainfall event at the Nebraska 11 site.

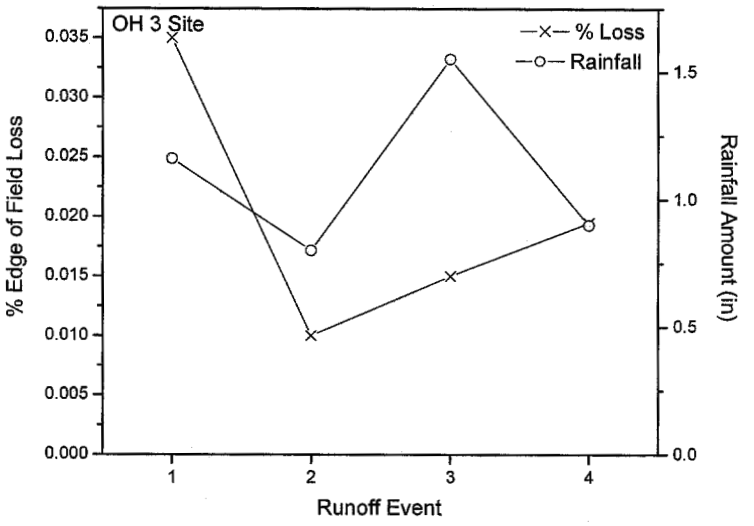

Figure 41 Fipronil edge of field loss for each rainfall event at the Ohio 3 site.

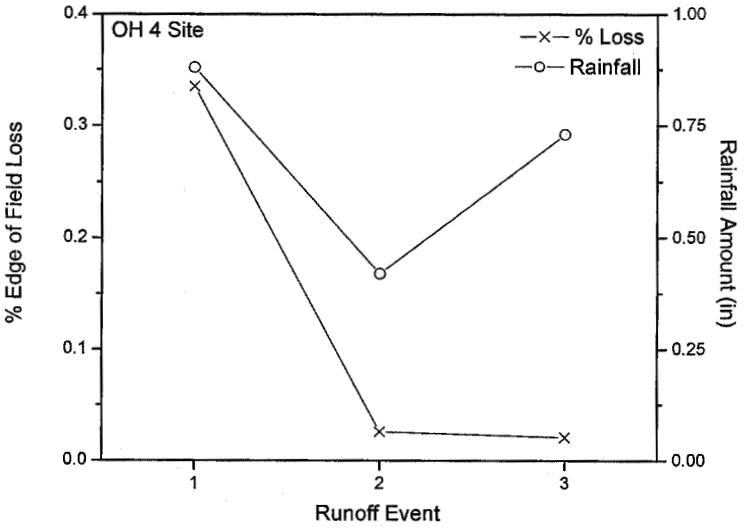

Figure 42 Fipronil edge of field loss for each rainfall event at the Ohio 4 site.

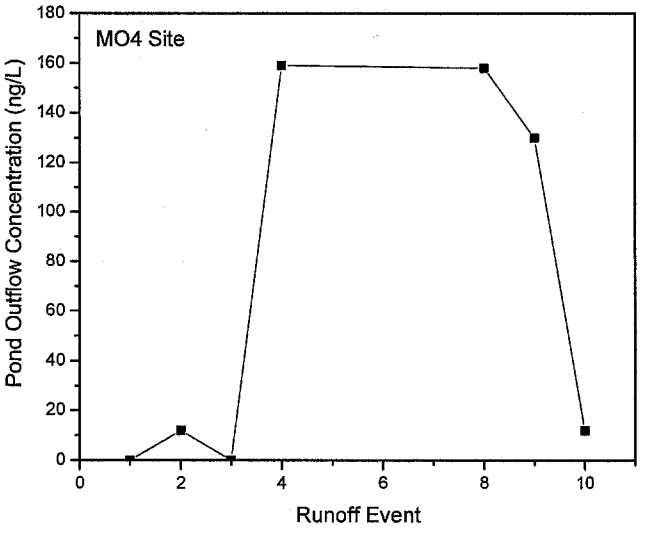

Figure 43 Missouri 4 pond concentrations.

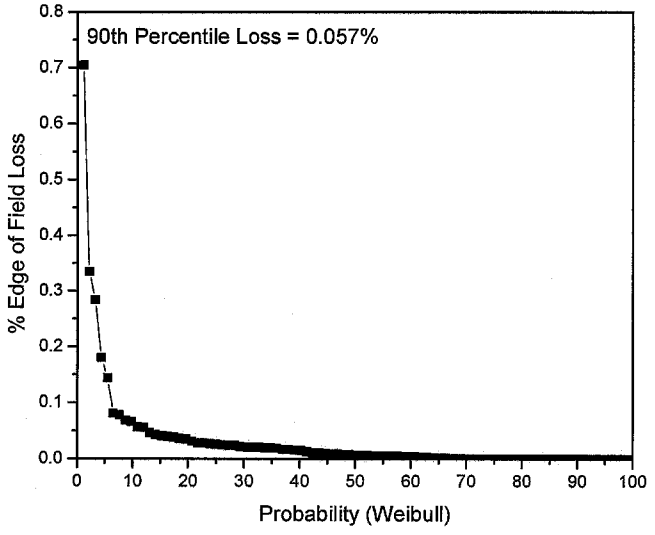

Figure 44 Probability distribution of fipronil edge of field loss - All events.

general, degradates of fipronil were not detected in the runoff water samples. Since we were measuring edge of field loss, it was not anticipated that degradates would be found commonly. There were three ponds sampled (IL3, IN10 and MO4) as part of this study in addition to the runoff data generated. At the IL3 and IN10 sites, there were no fipronil residues found in pond samples. However, at the IN10 site there was a detect of the MB 46513 degradate at 24 ng/L. At the MO4 site fipronil concentrations ranged from 0 to 159 ng/L. A backward, stepwise multiple regression was performed on the dataset to determine if a specific causal factor leading to residue runoff could be determined. Factors used in the multiple regression included data on site slope, soil texture, drainage area, and rainfall amount as predictors of edge of field loss, but none were predictive. Rainfall amount provided the best predictor of compound loss but the relationship (correlation to % edge of field loss to rainfall) was only a trend (**Figure 46**).

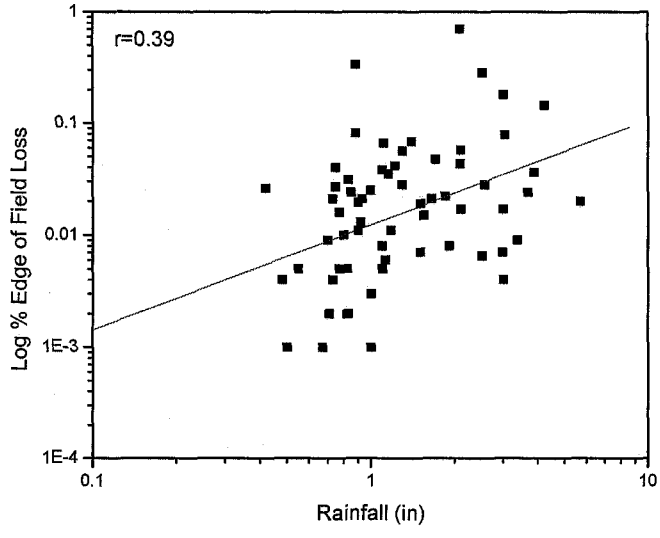

Figure 46 Relationship between rainfall and % edge of field loss.

A great deal of work on edge of field loss for various compounds can be found in the literature. We examined many papers as part of our work and several insightful papers are summarized in this document.

Southwick et al. (2003) studied atrazine and metolachlor runoff over a three year period in southern Louisiana. In the Southwick et al. study losses of atrazine in runoff water were 5.2%, 10.8%, and 6.0% for years 1995, 1996, and 1997 respectively. Losses of metolachlor in runoff water were 3.7%, 8.0%, and 5.0% for years 1995, 1996, and 1997 respectively. In the Southwick et al. study, edge of field loss corresponded to rainfall in decreasing response (e.g. 1st event, highest loss). The percent edge of field loss for both atrazine and metolachlor were much higher than those for fipronil based on the

Autosampler results

Five sites were setup to collect samples using a Sigma autosampler. The goal for using the autosamplers was to gain understanding about the behavior of residue leaving fields as runoff water in a way not possible with fixed time sampling (e.g. grab samples). Decline in residues from sample initiation to completion indicated that concentrations fluctuated throughout the sampling events and did not produce a general trend (e.g. decline from 1st sample to last). One phenomenon that was observed with most autosampler chronoseries we measured was a bimodal behavior for residue concentration. The bimodal behavior was not correlated to rainfall pattern. The bimodal pattern followed the anticipated peak in concentration followed by concentration decline, but this occurred twice in the full series of samples. A typical autosampler residue sequence can be observed in **Figure 47**. The first fours hours of the runoff event in **Figure 47** are presented in **Figure 48**. Residue concentrations in the sequence slowly increased from 1 hour to the 4 hour sampling, but rainfall fell as a slow steady rainfall event.

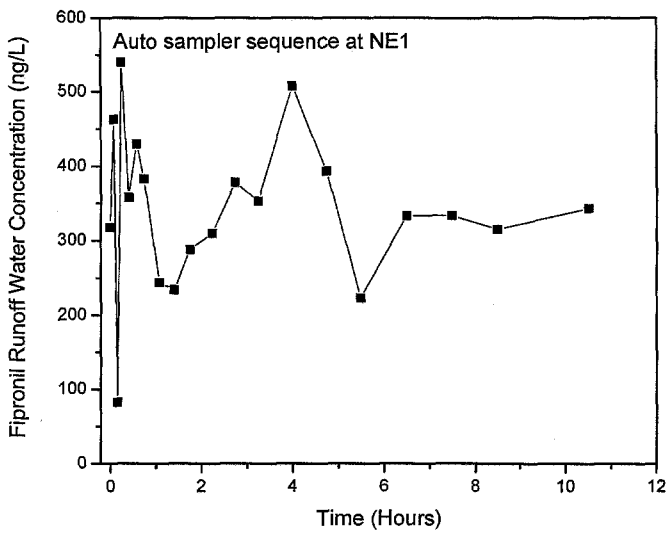

Figure 48. Residue concentrations from the autosampler at the NE1 site.

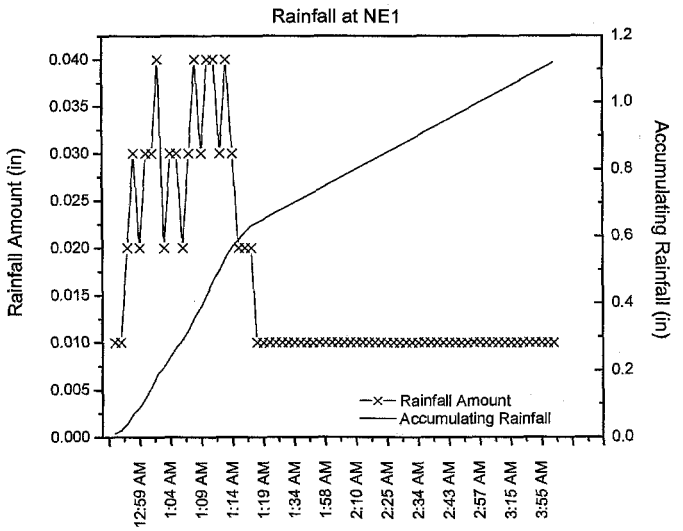

Figure 49. Rainfall pattern corresponding to autosampler sequence in Figure 47. Runoff continued past the end of the rainfall event.

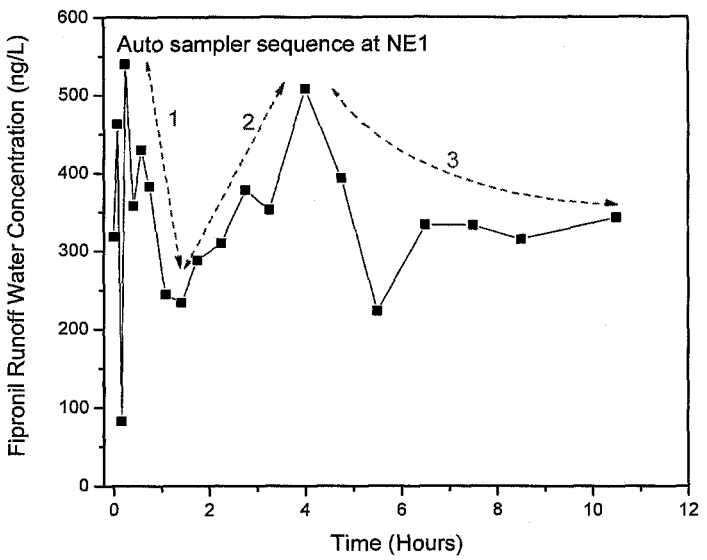

Figure 50. Diagram with the three phases of a runoff event displayed.

In **Figure 49**, three areas (phases) have been labeled that indicate runoff responses that we commonly observed in this study. In phase one, an initial event highest concentration is observed but declines as the soil becomes saturated. In phase two, the rain falls as a slow but steady event. However, once the soil became saturated,