DATA EVALUATION RECORD

STUDY 3

CHEM 129034 Flumioxazin §162-1
CAS No. 103361-09-7
FORMULATION–00-ACTIVE INGREDIENT

STUDY ID 44295040
Pensyl, J. W. 1996. Half-life of flumioxazin in four soils under laboratory conditions. Laboratory Project ID: VP-11579. Unpublished study performed and submitted by Valent U.S.A. Corp., Valent Technical Center, Dublin, CA.

DIRECT REVIEW TIME = 52 Hours

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CONCLUSIONS

Metabolism - Aerobic Soil

1. This study is scientifically valid and provides useful information on the aerobic soil metabolism of flumioxazin. However, degradates were not determined.

2. Nonradiolabeled flumioxazin, at a nominal application rate of 0.1 ppm, degraded with registrant-calculated half-lives of 5.0 days ($r^2 = 0.96$) in Wheeling sandy loam, 18.6 days ($r^2 = 0.98$) in Drummer clay loam, 18.9 days ($r^2 = 0.88$) in Dothan sand, and 15.6 days ($r^2 = 0.96$) in Webster loam soils incubated in darkness at 25.1 °C for up to 44 days. Soil samples were adjusted to 79-84% of 0.33 bar moisture content and homogenized by stirring, and the test vessels were capped. All data are the means of duplicate samples analyzed by gas chromatography. In the sandy loam soil, the parent was initially 0.1 ppm, decreased to 0.054 ppm by 6 days and 0.023 ppm by 13 days posttreatment, and was below the LOQ by 16 days. In the clay loam soil, the parent was initially 0.098 ppm, decreased to 0.051 ppm by 16 days posttreatment, was 0.046 ppm at 21 days, and was 0.017 ppm at 44 days. In the sand soil, the parent was initially 0.097 ppm, decreased to 0.053 ppm by 9 days and 0.043 ppm by 13 days posttreatment, and was last detected at 0.034 ppm at 28 days. In the loam soil, the parent was initially 0.097 ppm, was 0.054 ppm at 9 days posttreatment, and was last detected at 0.026 ppm at 28 days. Degradate compounds were not monitored.

METHODOLOGY

Samples (approximately 20 g dry weight) of sieved (2 mm) Wheeling sandy loam (collected from Charlestown, IN; 56% sand, 31% silt, 13% clay, pH 6.6, 3.3% organic matter, CEC 11.7 Meq/100 g; p. 13), Drummer clay loam (collected from Champaign Co., IL; 20% sand, 49% silt, 31% clay, pH 6.4, 4.9% organic matter, CEC 31.3 Meq/100 g), Dothan sand (collected from Hawkinsville, GA; 90% sand, 5% silt, 5% clay, pH 7.5, 1.09% organic matter, CEC 4.2 Meq/100 g), and Webster loam (collected from Boone Co., IA; 50% sand, 29% silt, 21% clay, pH 6.3, 4.3% organic matter, CEC 28.4 Meq/100 g) soils were each weighed into glass bottles and treated with nonradiolabeled flumioxazin {V-53482; 7-fluoro-6-((3,4,5,6-tetrahydro)phthalimido)-4-(2-propynyl)-1,4-benzoxazin-3(2H)-one; chemical purity 99.8%}, dissolved in acetone, at a nominal application rate of 0.1 ppm (p. 14). Soil samples were adjusted to 79-84% of 0.33 bar moisture content and homogenized by stirring, and the test vessels were capped (see Comment #5). The samples were placed in a plastic tray, and incubated in darkness in an incubator at 25.1°C for up to 44 days; the temperature was maintained by passing humidified air through the plastic sample tray (diagram not provided). Duplicate samples were removed for analysis at 0, 1, 3, 6, 9, 13, 16, 21, 28, and 44 days posttreatment (see Comment #6).

At each sampling interval, soil samples were extracted by shaking with acetone:0.1*N* HCl (5:1, v:v) and filtered (Whatman #1; Appendix II, p. 50); the filtrate was collected, combined with 5% aqueous sodium chloride, and extracted twice by shaking with dichloromethane in a separatory funnel. The extract was filtered (sodium sulfate and solvent-rinsed glass wool), and the filtrate was collected, evaporated to dryness by rotary evaporation, reconstituted in ethyl acetate, diluted with hexane, and sonicated. The extract was cleaned-up by glass column chromatography (Florisil) using hexane:ethyl acetate (2:1, v:v). The eluate containing the parent compound was collected, evaporated to dryness using rotary evaporation, reconstituted in acetone, and analyzed by gas chromatography (DB-17 column) with a nitrogen-phosphorus specific flame-ionization detector (p. 15; Appendix II, p. 51); the limits of detection and quantitation were 0.005 and 0.01 ppm, respectively.

Mean concurrent recoveries from soil samples fortified with the parent at 0.010, 0.050, or 0.100 ppm were 89-108% of the applied radioactivity (with the exception of 72%; Table II, p. 25).

DATA SUMMARY

Nonradiolabeled flumioxazin, at a nominal application rate of 0.1 ppm, degraded with registrant-calculated half-lives of 5.0 days ($r^2 = 0.96$) in Wheeling sandy loam, 18.6 days ($r^2 = 0.98$) in Drummer clay loam, 18.9 days ($r^2 = 0.88$) in Dothan sand, and 15.6 days ($r^2 = 0.96$) in Webster loam soils incubated in darkness at 25.1°C for up to 44 days (Figures 1-4, pp. 27-30). All data are the means of duplicate samples analyzed by gas chromatography. In the sandy loam soil, the parent compound was initially present at 0.1 ppm, decreased to 0.054 ppm by 6 days posttreatment, was 0.031 ppm at 9 days, was 0.023 ppm at 13 days posttreatment, and was below the LOQ by 16 days posttreatment (Table I, p. 24; tables on pp. 27-30). In the clay loam soil, the parent was initially present at 0.098 ppm, decreased to 0.051 ppm by 16 days posttreatment, was 0.046 ppm at 21 days, and was 0.017 ppm at 44 days posttreatment. In the sand soil, the parent was initially present at 0.097 ppm, decreased to 0.053 ppm by 9 days and 0.043 ppm by 13 days posttreatment, and was last detected at 0.034 ppm at 28 days posttreatment. In the loam soil, the parent was initially present at 0.097 ppm, was 0.054 ppm at 9 days and 0.050 ppm at 13 days posttreatment, and was last detected at 0.026 ppm at 28 days posttreatment. Degradate compounds were not monitored (see Comment #2).

Material balances were not determined (see Comment #1).

COMMENTS

1. Material balances were not determined. Subdivision N Guidelines require that material

balances of 90-110% be demonstrated. Material balances could not be determined in this study because only the parent compound was quantified, while degradates and volatiles were not addressed. Generally, in a study conducted with a nonradiolabeled compound, the material balance is calculated from the sum of the parent compound, degradates, and volatiles. The reviewer notes, however, that in two submitted photodegradation on soil studies (MIRDs 44295038 and 44295039) of the radiolabeled parent compound (different labels), volatiles were negligible (also see Comment #2).

2. The study author did not address the patterns of formation and decline of degradates. Subdivision N Guidelines require the identification of degradates present at ≥10% of the applied. However, the study author stated that no major degradates were observed in previously submitted aerobic soil metabolism studies using either uniformly phenyl ring-labeled [^{14}C]flumioxazin (MRID 42684906) or tetrahydrophthalimido ring-labeled [1,2-^{14}C]flumioxazin (MRID 42884009; p. 11). The study author stated the current study was performed as a supplement to the previous aerobic soil metabolism studies and to reconfirm the aerobic soil metabolism half-life in four soils.

3. Soil viability was not confirmed at the termination of the incubation period. Although analysis was conducted to determine the viability of the soils prior to treatment, the data were not obtained in a manner that conforms with Good Laboratory Practices as required under FIFRA (p. 36; Appendix IV, p. 79).

4. The study author did not describe the preparation of untreated control samples in the text; however, at each sampling interval, untreated control samples were analyzed with each set of soil samples (p. 16). The study author stated that the parent compound was below the detection limit in the untreated control samples (tabular data were not reported; p. 17). The reviewer notes, however, that the use of treated sterile soil samples, rather than untreated viable samples, would have been the appropriate control for an aerobic soil metabolism study.

5. The study author stated that soil samples were adjusted to 79-84% of the field moisture capacity. Subdivision N Guidelines require that the soil be adjusted to and maintained at 75% of 0.33 bar moisture content (also referred to as "field capacity").

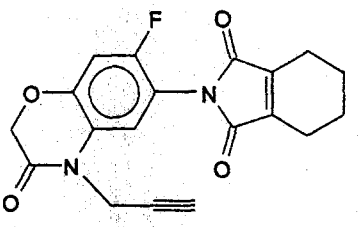
6. Samples were not removed for analysis on 21, 28 and 44 days posttreatment for the Wheeling sandy loam, and 44 days for the Dothan sand and Webster loam soils (Table I, p. 24).

7. The study author stated that the nominal treatment rate (0.1 ppm) for flumioxazin was based on the approximate maximum seasonal broadcast rate of 45 g ai/A (p. 17).

MATERIALS

TEST SUBSTANCE / REFERENCE STANDARD

Analytical grade flumioxazin was used as the test substance and reference standard in this study. Flumioxazin is the active ingredient in Valent's V-53482 WP Herbicide. The test substance used in this study was provided by Sumitomo Chemical and certified by Valent prior to study initiation.



Common Names: flumioxazin, V-53482
Chemical Name: 7-fluoro-6-((3,4,5,6,-tetrahydro)phthalimido)-4-(2-propynyl)-1,4-benzoxazin-3(2H)-one
CAS#: 103361-09-7
Lot#: As 1663g
% Purity: 99.8

SOIL CHARACTERIZATION

Soils for this study were all topsoils common to the soybean and peanut growing regions of the U.S. The soil series used in this study and the source of these samples are shown in the following table:

Identification of Soils

Soil Series Name	Source of Soil
Wheeling	PTRL East - Charlestown, IN
Drummer Flanagan	Valent MWARC - Champaign Co., IL
Dothan	Georgia Agri-Scientific - Hawkinsville, GA
Webster	H. Shepard* - Boone Co., IA

* H. Shepard is a Valent Field Marketing Specialist.

Soil characteristics for each of these soils were determined by Agvise Laboratories. The data obtained are summarized in the following table:

Table I

Residues of Flumioxazin in Four Soils
Approximate Dry Weight Basis

Day of Study	ppm Flumioxazin Found			
	Wheeling	Drummer	Dothan	Webster
0	0.099 0.100	0.098 0.098	0.097 0.097	0.099 0.095
1	0.091 0.091	0.088 0.090	0.080 0.080	0.079 0.084
3	0.071 0.070	0.084 0.080	0.071 0.066	0.058 0.074
6	0.055 0.052	0.082 0.079	0.060 0.053	0.062 0.058
9	0.030 0.032	0.069 0.067	0.054 0.052	0.054 0.053
13	0.022 0.023	0.066 0.063	0.043 0.043	0.050 0.049
16	0.009 0.009	0.051 0.050	0.041 0.037	0.036 0.037
21	*	0.048 0.043	0.035 0.034	0.032 0.032
28	*	0.043 0.037	0.034 0.033	0.026 0.026
44	*	0.017 0.017	*	*

* No sample taken.

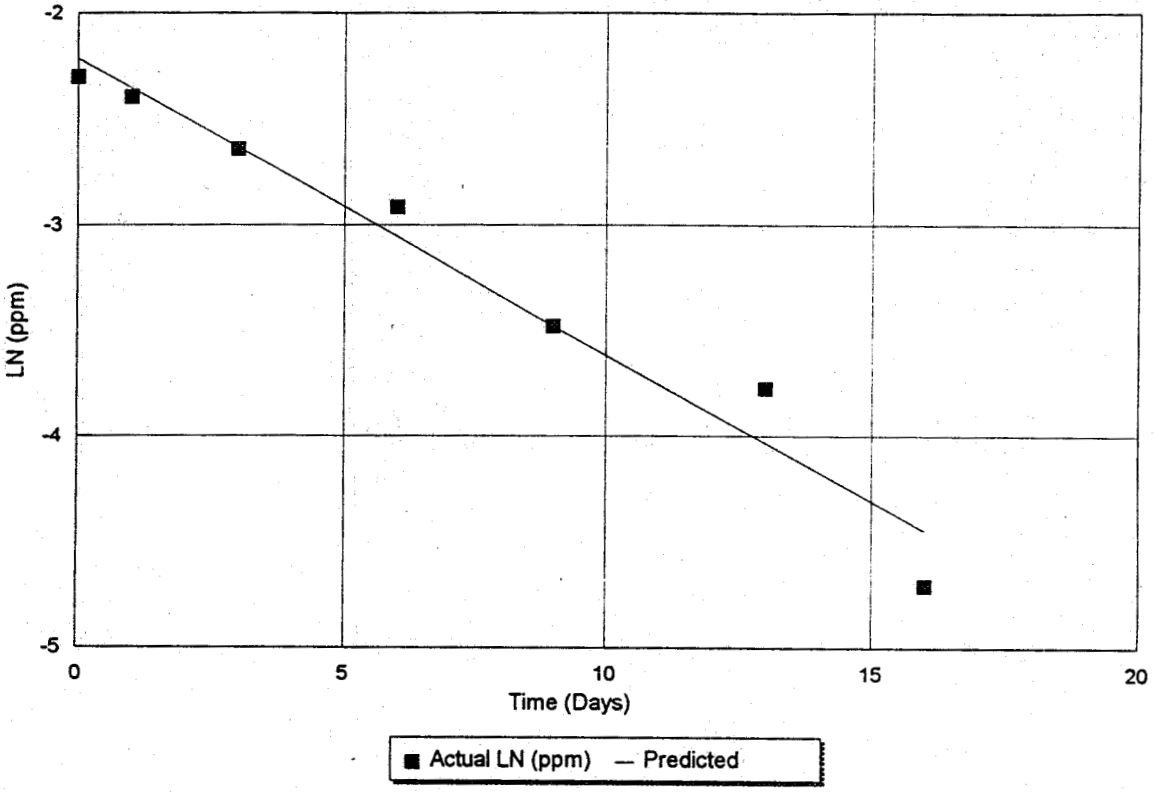
Table II

Recovery of Flumioxazin From Soil

Date Analyzed	Soil Series Name	Amount Added (ppm)	Amount Found (ppm)	% Recovery
6/5/96	Drummer	0.050	0.054	108
6/6/96	Dothan	0.050	0.050	98
6/14/96	Drummer	0.050	0.054	106
6/19/96	Dothan	0.050	0.051	101
6/24/96	Webster	0.050	0.050	98
6/26/96	Drummer	0.050	0.050	100
7/5/96	Drummer	0.050	0.051	101
7/25/96	Drummer	0.050	0.036	72
		Mean Recovery	(0.050 ppm level)	98
		CV (n = 8)		11.3
6/10/96	Webster	0.100	0.103	102
6/11/96	Wheeling	0.100	0.095	95
		Mean Recovery	(0.100 ppm level)	99
		CV (n =2)		5.03
5/29/96	Wheeling	0.010	0.009	89
5/29/96	Wheeling	0.010	0.009	90
5/29/96	Wheeling	0.010	0.009	91
		Mean Recovery	(0.010 ppm level)	90
		CV (n =3)		1.11

FIGURE 1

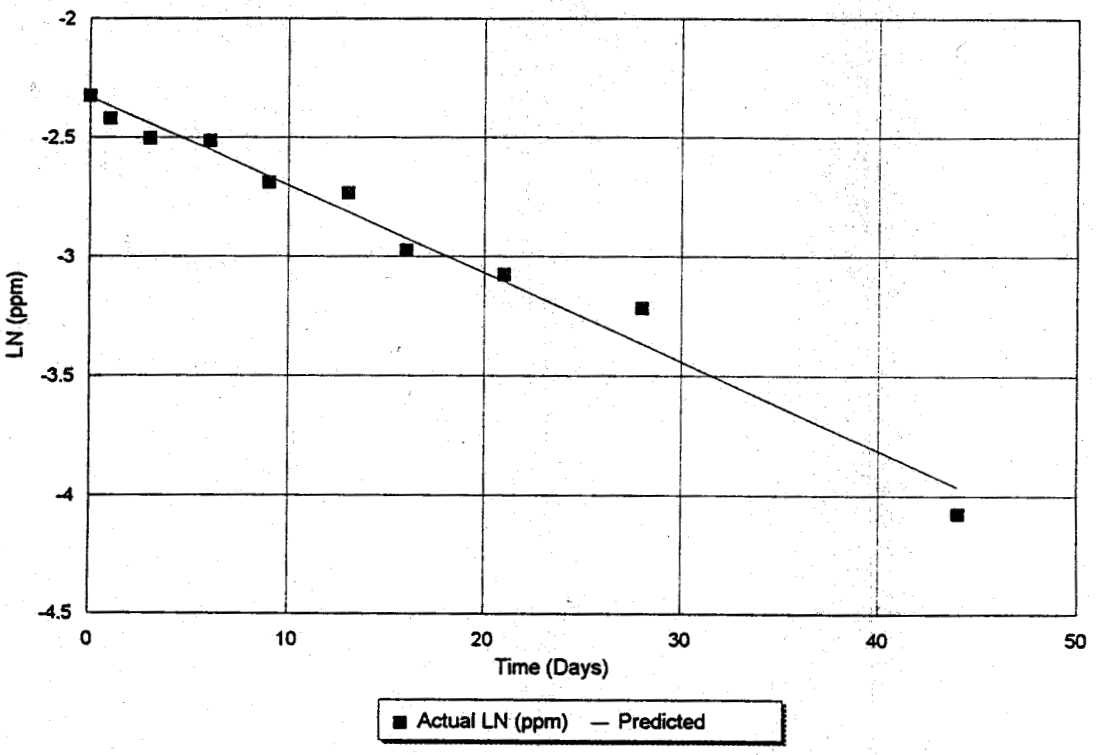
Decline of Flumioxazin From Wheeling Soil



Log Least Square Estimate of 'm' and 'b' for: Y = b*EXP(mX) (or LN Y = mX + LN b) and for correlation coefficient 'r'.					
m=	-0.13978				
LN b=	-2.21576				
b=	0.10907				
Half-life=	4.96 Days				
r=	-0.98186				
X (Days)	Y (ppm)	LN Y	LN YP	YP	Residual
0	0.1	-2.30259	-2.21576	0.109071	0.086826
1	0.091	-2.3969	-2.35554	0.094842	0.041354
3	0.071	-2.64508	-2.63511	0.071711	0.009968
6	0.054	-2.91877	-3.05446	0.047148	-0.13568
9	0.031	-3.47377	-3.4738	0.030999	-3.6E-05
13	0.023	-3.77226	-4.03294	0.017722	-0.26067
16	0.009	-4.71053	-4.45228	0.011652	0.258247

FIGURE 2

Decline of Flumioxazin From Drummer Soil

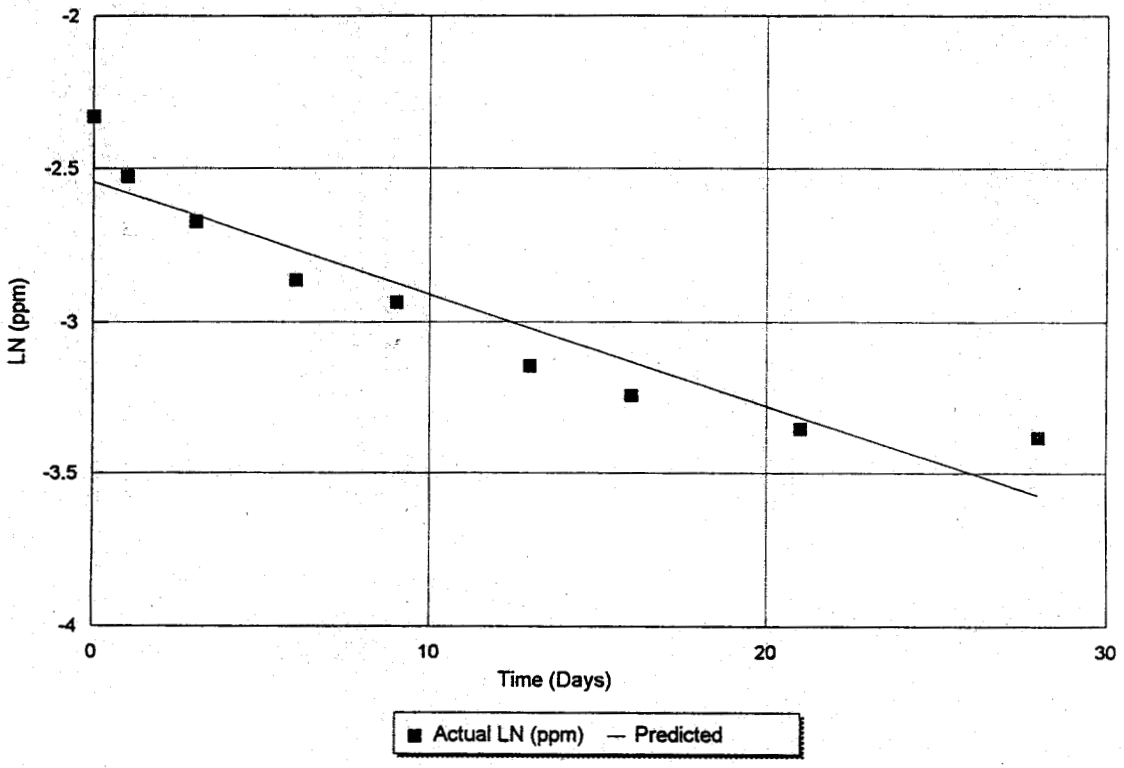


Log Least Square Estimate of 'm' and 'b' for: Y = b*EXP(mX) (or LN Y = mX + LN b) and for correlation coefficient 'r'.		
m=	-0.03722	
LN b=	-2.32789	
b=	0.09750	
Half-life=	18.63	Days
r=	-0.98902	

X (Days)	Y (ppm)	LN Y	LN YP	YP	Residual
0	0.098	-2.32279	-2.32789	0.097501	-0.0051
1	0.089	-2.41912	-2.36511	0.093939	0.054013
3	0.082	-2.50104	-2.43954	0.087201	0.061499
6	0.081	-2.51331	-2.55118	0.077989	-0.03788
9	0.068	-2.68825	-2.66283	0.06975	0.025416
13	0.065	-2.73337	-2.8117	0.060103	-0.07833
16	0.051	-2.97593	-2.92334	0.053754	0.052587
21	0.046	-3.07911	-3.10942	0.044627	-0.03031
28	0.04	-3.21888	-3.36993	0.034392	-0.15106
44	0.017	-4.07454	-3.96538	0.018961	0.109157

FIGURE 3

Decline of Flumioxazin From Dothan Soil



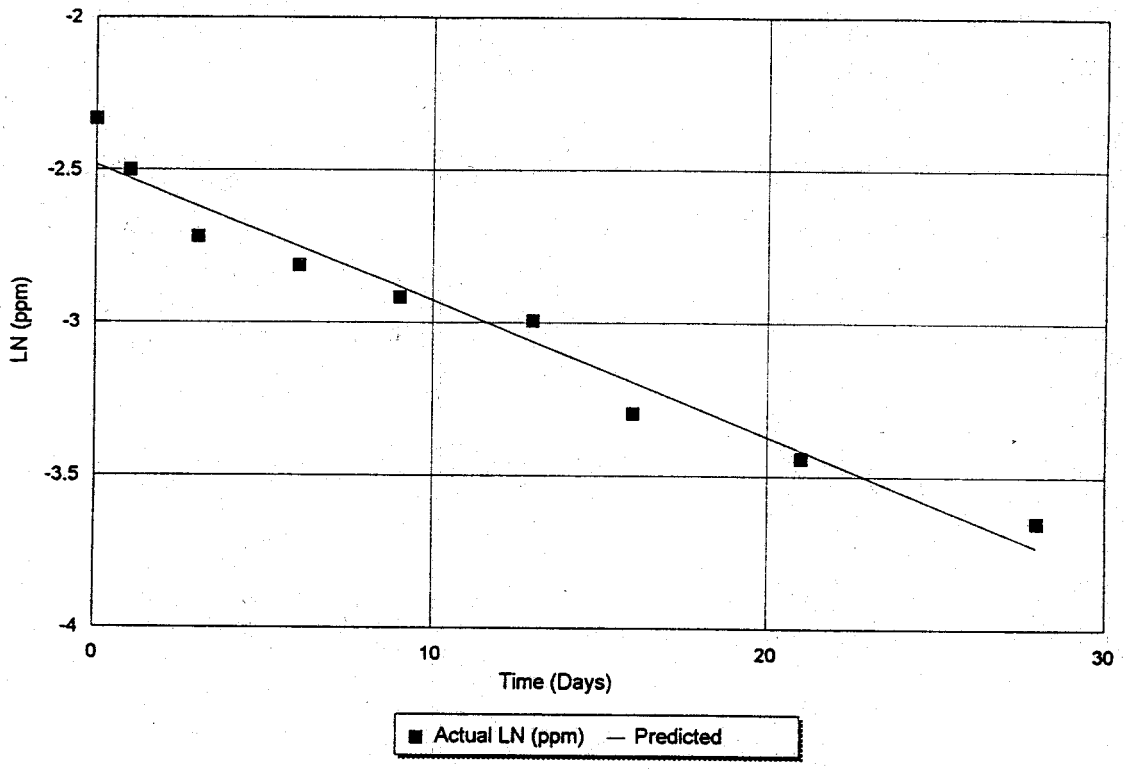
Log Least Square Estimate of 'm' and 'b' for:
Y = b*EXP(mX) (or LN Y = mX + LN b) and for correlation coefficient 'r'.

m=	-0.03676	
LN b=	-2.54369	
b=	0.07858	
Half-life=	18.86	Days
r=	-0.94090	

X (Days)	Y (ppm)	LN Y	LN YP	YP	Residual
0	0.097	-2.33304	-2.54369	0.078576	-0.21065
1	0.08	-2.52573	-2.58046	0.075739	-0.05473
3	0.069	-2.67365	-2.65398	0.070371	0.019669
6	0.057	-2.8647	-2.76426	0.063022	0.100439
9	0.053	-2.93746	-2.87455	0.056442	0.062913
13	0.043	-3.14656	-3.0216	0.048723	0.124958
16	0.039	-3.24419	-3.13188	0.043636	0.112311
21	0.035	-3.35241	-3.31569	0.036309	0.036716
28	0.034	-3.38139	-3.57302	0.028071	-0.19163

FIGURE 4

Decline of Flumioxazin From Webster Soil



Log Least Square Estimate of 'm' and 'b' for: Y = b*EXP(mX) (or LN Y = mX + LN b) and for correlation coefficient 'r'.						
m=	-0.04448					
LN b=	-2.48373					
b=	0.08343					
Half-life=	15.58	Days				
r=	-0.97978					
X (Days)	Y (ppm)		LN Y	LN YP	YP	Residual
0	0.097		-2.33304	-2.48373	0.083431	-0.15069
1	0.082		-2.50104	-2.52822	0.079801	-0.02718
3	0.066		-2.7181	-2.61719	0.073008	0.100915
6	0.06		-2.81341	-2.75064	0.063887	0.062771
9	0.054		-2.91877	-2.88409	0.055905	0.034676
13	0.05		-2.99573	-3.06203	0.046792	-0.0663
16	0.037		-3.29684	-3.19549	0.040947	0.101349
21	0.032		-3.44202	-3.41791	0.032781	0.024106
28	0.026		-3.64966	-3.72931	0.024009	-0.07965

AGVISE LABORATORIES

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MICROBIAL RESULTS

Grower: VP-11579
Submitter:
Valent USA Corp
PO Box 8025
Walnut Creek, CA 54596-8025

Date Received: 5/16/96
Date Analyzed: 5/21/96
Print Date: 5/28/96

Sample#	Reference #	Matrix	Parameter	Result	Units*
1309	VTC-55-96	Soil			
			Actinomycetes	8,900,000	CFU/g of dry soil
			Bacteria	3,900,000	CFU/g of dry soil
			Fungi	69,000	CFU/g of dry soil
1310	VTC-56-96	Soil			
			Actinomycetes	8,100,000	CFU/g of dry soil
			Bacteria	5,400,000	CFU/g of dry soil
			Fungi	57,000	CFU/g of dry soil
1311	VTC-57-96	Soil			
			Actinomycetes	2,700,000	CFU/g of dry soil
			Bacteria	610,000	CFU/g of dry soil
			Fungi	9,100	CFU/g of dry soil
1312	VTC-58-96	Soil			
			Actinomycetes	4,600,000	CFU/g of dry soil
			Bacteria	600,000	CFU/g of dry soil
			Fungi	9,300	CFU/g of dry soil

*CFU = Colony Forming Unit

Comments:

Chemist - Dan Stetler Date- 5-28-96 QC- Rebecca A Bower Date- 5/28/96