10-26-94

DATA EVALUATION RECORD

CHEM 129086 Phostebupirim 164-1

FORMULATION--04--GRANULAR

STUDY ID 43092702
Dehart, B.A. and G.C. Mattern. 1994. Field dissipation of phostebupirim (MAT 7484) on Illinois soil. Unpublished study performed by Agri-Growth Research Inc., Geneseo, IL and Miles Environmental Fate Analytical, Kansas City, MO for Miles, Inc., Kansas City, MO.

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CONCLUSIONS:

Dissipation - Terrestrial Field

1. This study provides supplemental data towards the fulfillment of the terrestrial field dissipation guideline for phostebupirim (MAT 7484).

2. MAT 7484 ((O-[2-(1,1-dimethylethyl)-5-pyrimidinyl] O-ethyl O-(1-methylethyl) phosphorothioate) dissipated with a calculated half-life of 39 days (r^2= 0.43) from the top 6 inches of a sand/loamy sand soil in Illinois. MAT 7484 varied without pattern from 2-274 days posttreatment and was below detection by 343 days. The compound was applied as a granular formulation in a 7-inch band at a nominal rate of 0.168 lb ai/A. MAT 7484 was applied with 0.1% cyfluthrin at corn planting and was incorporated to a depth of 1/4 inch.

   No detectable MAT 7484 residues leached below the 6-inch depth; there were no detections of degradates OMAT (MAT 7484 oxygen analog) or IMATS (isopropyl MAT 7484 s-ester isomer).

3. The study does not provide acceptable data because the route of dissipation is unclear and the sampling

procedure of random samples may not be appropriate for band applications of pesticides.

METHODOLOGY:

Site and application: The test plot, located near Geneseo, Illinois, was 90 x 100 ft with 10 foot buffer strips on each side. The soil at the site was characterized as a sand for the top 6 inches, a loamy sand for the 6- to 12-inch depth and as a sand from 18- to 42-inches: soil characterization by depth is in Table 3. The slope of the plot was <1% and the water table was at 8-10 feet. This is in a corn growing area and the plot had been planted to soybeans and corn in the three years prior to the study.

Planting and application were accomplished with a Nobel applicator attached to the corn planter on August 6, 1991. Corn seed was planted with a furrow type planter which closed the furrow prior to application of granular material in a 7-inch band. The application was incorporated to a 1/4 inch depth with a drag chain. Aztec 2.1% ai formulation was 2.04% phostebupirim and 0.1% cyfluthrin; the application rate was 0.168 ai/A for the overall plot and 0.73 lb ai/A within the 7-inch band.

Plot maintenance and weather: There were three rainfall events within the first week after application: 0.85 inches at day 1, 0.5 and 0.6 inches at days 2 and 7, respectively. The plot was to be irrigated at 90% of the 10 year monthly precipitation average at the Moline IL NOAA weather station. The total irrigation plus rainfall for the study was 48.55 inches which was 125% of the 10 year monthly average. The soil temperatures at 2 inches ranged from 72-92 F for the first 28 days after treatment.

Sampling: The plot was divided into five sections designated A-E, each section was divided into 72 subsections totaling 360 subsections (5 x 5 ft). Samples were removed prior to application, immediately after application, and at 1, 2, 4, 7, 10, 14, 21, 28, 62, 100, 198, 274, 343, and 366 days posttreatment. At each sampling interval, a core was taken from three subsections in each of the five subplots; the cores were taken randomly without respect to the pesticide band. A corer using plastic liners with a 1.75 inch inner diameter was used for all samples. A 0- to 6-inch core was taken immediately after application and a 0- to 37-inch core was taken at all other sampling intervals. Cores in the plastic liners were capped, frozen, and transported to the analytical laboratory. At the analytical laboratory, cores were thawed, segmented into 0-

to 6-, 6- to 12-, 12- to 18-, 18- to 24-, 24- to 30-, 30- to 36-inch, and 36-inches to the end of core. Samples were composited by depth with one core from each of the five subplots making one sample resulting in three composited samples per interval.

Field spikes: At each sampling interval, three soil samples (50 grams each) from an untreated plot were fortified with MAT 7484 at 1 ug/g; separate samples were fortified with OMAT. One untreated sample was included as a control. These samples were transported and frozen with the cores from the treated plot.

Sample extraction and analysis: Each composite sample was thawed, mixed, and sieved to 2-mm. A 25-g portion of sample was Soxhlet extracted with chloroform:methanol (7:3) for 4 hours. The resulting mixture was cooled, the solvents were evaporated to dryness and the solids were extracted with three 50-mL portions of hexane by shaking for one minute. The hexane extracts were poured through anhydrous sodium sulfate and combined. The hexane was concentrated to dryness and the residue was dissolved in methanol (2.5 mL). The methanol was filtered and aliquots were analyzed by GC with a nitrogen/phosphorus detector. A standard curve of MAT 7484 and MAT 7484-residues at four concentrations ranging from 0.004 to 0.04 ug/g was run prior to each analytical run.

Method validation: Five soil samples were spiked with MAT 7484 and OMAT at 0.01, 0.02, and 0.05 ug/g. Additional samples were spiked with 0.01 ug/g IMATS. The samples were extracted and analyzed as described above. Recoveries were 84-105% for MAT 7484, 87-122% for OMAT, and 106-116% for IMATS. There were no differences in recoveries based on fortification rates.

DATA SUMMARY:

MAT 7484 dissipated with a registrant calculated half-life of 39 days (r^2= 0.43) from the upper 6 inch depth when applied at 0.168 lb ai/A to a sand soil in Illinois. MAT 7484 apparently did not leach below the 6-inch depth at this site.

In the top 0- to 6-inch increment, MAT residues were an average of 0.17 ug/g immediately posttreatment, 0.13 at 1 day posttreatment, ranged from 0.01 to 0.11 at 2-274 days with no pattern of dissipation, and was below detection <0.01 ug/g at 343 and 366 days posttreatment (Table 24). There were no detections below 6 inches at any sampling interval (Tables 10-23).

There were no detectable residues of OMAT or IMATS; method detection limits were 0.01 ug/g soil.

COMMENTS

1. Although in this study the calculated half-life was 39 days (r^2= 0.43), there was no clear pattern of dissipation. When samples from a band application are taken randomly as with a broadcast application, it is often not possible to determine the dissipation pattern because of the dilution effect from the non-treated areas. From the composite samples in this study, MAT 7484 decreased from 0.17 ug/g immediately posttreatment to 0.13 ug/g at day 1 and then varied without pattern from 2-274 days posttreatment and was below detection by 343 days.

   It is not clear how MAT 7484 dissipates in the environment. In the laboratory studies, hydrolysis and photolysis are the main degradation pathways. The aerobic metabolism half-life was 343 days. The lack of laboratory degradation considered with the improper sampling method suggests that the 39 day half-life is a result of dilution effects and is not an indication of field dissipation.

2. **Storage stability:** No storage stability data were submitted with this study. The registrant stated that MAT 7484 was stable in frozen soil "for the duration of a 12-month study" and that OMAT was stable "for the duration of a 6-month study". No data were provided for review and these studies were not referenced. For the field spike samples, there was a slight trend towards lower recoveries with longer frozen storage; samples stored frozen for 13-395 days ranged from 84 to 136% recoveries and samples stored frozen for 408-424 days had recoveries of 47-75%. Soil core samples were stored frozen for up to 424 days.

3. There seems to be a problem with the MAT 7484 oxygen analog field spike samples; after the 14 day sampling interval, the amounts of OMAT recovered increased from 139% at 14 days, 150-154% at 21-100 days, 209-282% recovered at 198-236 days to 540% recovered at the 366 days sampling intervals.

4. Pesticide usage history: A alachlor was applied to this plot in 1988, atrazine and tridithane were applied in 1989 and 1990; turbufos was also applied in 1990.

Table 5. Cumulative rainfall plus irrigation and sampling interval dates.

Sampling Interval	Date	Cumulative Rainfall plus Irrigation (inches)
P-0	August 6, 1991	0.00
P-1	August 7, 1991	0.85
P-2	August 8, 1991	1.35
P-4	August 10, 1991	1.35
P-7	August 13, 1991	1.95
P-10	August 16, 1991	1.95
P-14	August 20, 1991	2.35
P-21	August 27, 1991	2.35
P-28	September 3, 1991	3.10
P-62	October 7, 1991	9.70
P-100	November 14, 1991	12.71
P-198	February 20, 1992	15.87
P-274	May 6, 1992	24.59
P-343	July 13, 1992	42.41
P-366	August 5, 1992	48.55

Table 3. Soil characterization for the test site.

	Soil Depth (inches)						
	0-6	6-12	12-18	18-24	24-30	30-36	36-42
% Organic Matter	1.4	0.7	0.4	0.2	0.7	0.2	0.2
pH	5.2	4.8	4.5	4.5	4.7	4.8	4.9
CEC (meq/100g)	7.7	7.0	5.6	5.0	4.8	3.8	3.9
% Moisture at 1/3 bar	9.7	9.8	8.6	5.4	8.1	7.5	7.8
Bulk Density (g/cc)	1.32	1.32	1.39	1.40	1.41	1.42	1.42
% Sand	88	86	88	90	95	97	95
% Silt	8	8	6	6	3	1	3
% Clay	4	6	6	4	2	2	2
Texture Class (USDA)	sand	loamy sand	loamy sand	sand	sand	sand	sand

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Table 9. MAT 7484 sample analyses 0-days after application

Sampling Depth (inches)	Miles Sample Number	MAT 7484 concentration (ug/g)	average concentration(ug/g) [1]
0-6	666022	0.21	
0-6	666023	0.06	0.17
0-6	666024	0.23	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 10. MAT 7484 sample analyses 1 day after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666025	0.26	
0-6	666032	0.10	0.13
0-6	666039	0.02	
6-12	666026	<0.01	
6-12	666033	<0.01	<0.01
6-12	666040	<0.01	
12-18	666027	<0.01	
12-18	666034	<0.01	<0.01
12-18	666041	<0.01	
18-24	666028	<0.01	
18-24	666035	<0.01	<0.01
18-24	666042	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 11. MAT 7484 sample analyses 2 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666046	0.04	
0-6	666053	0.02	0.02
0-6	666060	<0.01	
6-12	666047	<0.01	
6-12	666054	<0.01	<0.01
6-12	666061	<0.01	
12-18	666048	<0.01	
12-18	666055	<0.01	<0.01
12-18	666062	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 12. MAT 7484 sample analyses 4 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666067	0.10	
0-6	666074	0.14	0.11
0-6	666081	0.10	
6-12	666068	<0.01	
6-12	666075	<0.01	<0.01
6-12	666082	<0.01	
12-18	666069	<0.01	
12-18	666076	<0.01	<0.01
12-18	666083	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 13. MAT 7484 sample analyses 7 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666088	0.07	
0-6	666095	0.03	0.06
0-6	666102	0.08	
6-12	666089	<0.01	
6-12	666096	<0.01	<0.01
6-12	666103	<0.01	
12-18	666090	<0.01	
12-18	666097	<0.01	<0.01
12-18	666104	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 14. MAT 7484 sample analyses 10 days after application

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666109	0.05	
0-6	666116	0.11	0.06
0-6	666123	0.01	
6-12	666110	<0.01	
6-12	666117	<0.01	<0.01
6-12	666124	<0.01	
12-18	666111	<0.01	
12-18	666118	<0.01	<0.01
12-18	666125	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 15. MAT 7484 sample analyses 14 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666130	0.10	
0-6	666137	0.01	0.06
0-6	666144	0.06	
6-12	666131	<0.01	
6-12	666138	<0.01	<0.01
6-12	666145	<0.01	
12-18	666132	<0.01	
12-18	666139	<0.01	<0.01
12-18	666146	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 16. MAT 7484 sample analyses 21 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666151	0.02	
0-6	666158	<0.01	0.01
0-6	666165	0.01	
6-12	666152	<0.01	
6-12	666159	<0.01	<0.01
6-12	666166	<0.01	
12-18	666153	<0.01	
12-18	666160	<0.01	<0.01
12-18	666167	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 17. MAT 7484 sample analyses 28 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666172	0.09	
0-6	666179	0.06	0.09
0-6	666186	0.11	
6-12	666173	<0.01	
6-12	666180	<0.01	<0.01
6-12	666187	<0.01	
12-18	666174	<0.01	
12-18	666181	<0.01	<0.01
12-18	666188	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 18. MAT 7484 sample analyses 62 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666193	0.02	
0-6	666200	<0.01	0.02
0-6	666207	0.06	
6-12	666194	<0.01	
6-12	666201	<0.01	<0.01
6-12	666208	<0.01	
12-18	666195	<0.01	
12-18	666202	<0.01	<0.01
12-18	666209	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 19. MAT 7484 sample analyses 90 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666214	0.08	
0-6	666221	0.03	0.05
0-6	666228	0.05	
6-12	666215	<0.01	
6-12	666222	<0.01	<0.01
6-12	666229	<0.01	
12-18	666216	<0.01	
12-18	666223	<0.01	<0.01
12-18	666230	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 20. MAT 7484 sample analyses 198 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666235	0.02	
0-6	666242	0.08	0.04
0-6	666249	0.03	
6-12	666236	<0.01	
6-12	666243	<0.01	<0.01
6-12	666250	<0.01	
12-18	666237	<0.01	
12-18	666244	<0.01	<0.01
12-18	666251	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

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Table 21. MAT 7484 sample analyses 274 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666256	0.02	
0-6	666263	0.04	0.02
0-6	666270	<0.01	
6-12	666257	<0.01	
6-12	666264	<0.01	<0.01
6-12	666271	<0.01	
12-18	666258	<0.01	
12-18	666265	<0.01	<0.01
12-18	666272	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 22. MAT 7484 sample analyses 343 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666277	<0.01	
0-6	666284	<0.01	<0.01
0-6	666291	<0.01	
6-12	666278	<0.01	
6-12	666285	<0.01	<0.01
6-12	666292	<0.01	
12-18	666279	<0.01	
12-18	666286	<0.01	<0.01
12-18	666293	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Table 23. MAT 7484 sample analyses 366 days after application.

sampling depth (inches)	Miles sample number	MAT 7484 concentration (ug/g)	average concentration (ug/g)[1]
0-6	666298	<0.01	
0-6	666305	<0.01	<0.01
0-6	666312	<0.01	
6-12	666299	<0.01	
6-12	666306	<0.01	<0.01
6-12	666313	<0.01	
12-18	666300	<0.01	
12-18	666307	<0.01	<0.01
12-18	666314	<0.01	

[1] When average was less than the limit of detection then it is reported as <0.01.

Dry residue values of each analyte where determine by the following calculation:

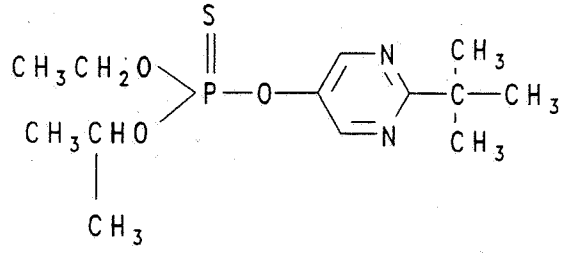
$$\text{analyte dry residue } \mu g/g = \frac{\text{sample concentration } (\mu g/g)}{1 - (\text{percent moisture}/100)}$$

total dry residue µg = dry residue µg/g x total composite sample weight g

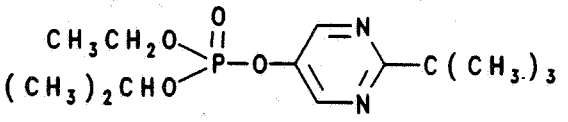
4.0 RESULTS AND DISCUSSION

4.1 Analyte Choice Justification

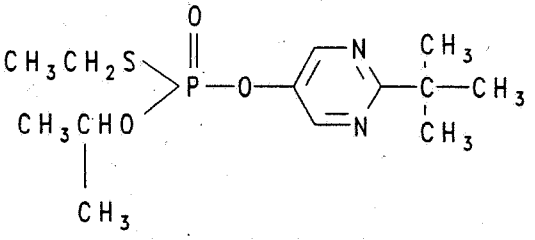
The information given in section 2.3 shows that no metabolite of MAT 7484 exceeded 10% of the total radioactivity in the aerobic or anaerobic soil metabolism studies. However, analyses were performed for the parent compound MAT 7484, and two degradates; MAT 7484 oxygen analog and IMATS. The structures of these three analytes are shown below.



MAT 7484



MAT 7484 oxygen analog



MAT 7484 IMATS

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4.2 Instrumental Response Linearity

Instrumental response linearity was demonstrated from concentrations of 0.01 to 0.2 ppm in soil. Four data points were used and the r^2 was 0.9 or better. The analytical system was considered linear up to 0.2 ppm. Samples with residue concentrations higher than 0.2 ppm were diluted before final analysis. Before each set of actual samples was analyzed, a standard curve was prepared by making duplicate injections of analytical standards with concentrations (relative to matrix) of 0.01, 0.05, 0.10, and 0.20 µg/g. This standard curve while indicating instrument linearity also was used to calculate actual sample concentration. Linear response representative chromatograms are shown in Figures 6-9.

4.3 Method Validation

Five soil samples from the test site were spiked with MAT 7484 and MAT 7484 oxygen analog at concentrations of 0.01, 0.02 and 0.05 µg/g. Soil samples were also spiked with MAT 7484 IMATS at 0.01 µg/g. The range of recoveries of MAT 7484, MAT 7484 oxygen analog, and MAT 7484 IMATS are summarized below. Additional information can be found in MR 106240-2[14]. Representative chromatograms from each method validation concentration performed are shown in Figures 10-15.

Concentration added to each of 5 soil samples (µg/g)	Range of recoveries (%) of MAT 7484	Range of recoveries (%) of MAT 7484 oxygen analog	Range of recoveries (%) of MAT 7484 IMATS
0.01	84-105	92-98	106-116
0.02	85-95	87-122	n.a.
0.05	96-103	90-102	n.a.
Average	95	99	111
Relative standard deviation (%)	7	10	5

n.a.; not available

4.4 Concurrent Recoveries

A laboratory fortified sample of MAT 7484, MAT 7484 oxygen analog, and MAT 7484 IMATS at 0.1 or 0.01 µg/g was analyzed with each group of field samples. For each analyte, the range of recoveries, average recovery and

relative standard deviation can be found below. Concurrent recovery data can also be found in MR 106240-1[14]. The concentrations of MAT 7484, MAT 7484 oxygen analog or MAT IMATS in the actual field samples from this study have not been corrected by concurrent recovery values. A representative chromatogram of a laboratory fortified concurrent recovery sample can be found in Figure 16.

Concentration added to soil samples (μg/g)	Range of recoveries (%) of MAT 7484	Range of recoveries (%) of MAT 7484 oxygen analog	Range of recoveries (%) of MAT 7484 IMATS
0.01	59-110	78-114	78-119
Average	93	99	102
Relative standard deviation (%)	13	10	14

4.5 Resultant Limit of Detection

The limit of detection is defined by Miles Inc., as being the lowest detectable residue level at which reliable measurements can be made. This is supported by acceptable recoveries from the soil matrix at that level. Also, this limit could be elevated due to any apparent residue of the test compound in the given pre-application sample. A representative chromatogram from the analysis of a pre-application sample can be found in Figure 17.

Analyses of control soil from the test plot indicated no detectable amounts of MAT 7484 were present before the application of the test compound. The lowest level of fortification performed by MEFA in the method validation was 0.01 μg/g, therefore the limit of detection for all analyses included in this report was set at 0.01 μg/g. Any apparent residue found below this limit was reported as <0.01 μg/g.

4.6 Residues of MAT 7484, OMAT and IMATS Detected

The average amount of MAT 7484 residue in the Day-0 soil samples was 0.17 μg/g, representing 43 % of the theoretical 0.40 μg/g (0.73 lbs a.i. per acre; see section 3.1.4) applied. The concentrations of MAT 7484 residues detected at each sampling interval are presented in Tables 8 to 23. The sum of total residues found is provided in Table 24. A representative chromatogram from analyses of samples taken 1 day after application and a spreadsheet are shown in Figure 18 and 19. The data for the first-order rate plot is found in Figure 20. The entire residue analysis report from MEFA can be found in 106240-2[14]

The half-life of MAT 7484 from 0 to 366 days after application was estimated to be 39 days. The half-life of MAT 7484 was calculated by the following equation:

$$t^{1/2} = \frac{\ln (2)}{- (k \text{ constant})}$$

The k constant represents the slope determined from regression analysis of time in days after compound application, versus natural log of the percent of day 0 residue found (μg/g) at each sampling interval. The residue values obtained for the regression analyses were determined by summing residues at each sampling interval (μg/g) from each depth. The half-life was calculated to be 39 days (r^2 =0.43) from 0 to 366 days with a first-order rate constant of -0.01771.

Soil samples included in this report which were analyzed for MAT 7484, MAT 7484 oxygen analog, and MAT 7484 IMATS were held in frozen storage for a maximum of 424 days from the time of sampling to the time of extraction at the analytical laboratory. Currently available freezer storage stability data are presented in section 2.3 of this report.

4.7 Dissipation and Leaching of Residues in Field Tests

Tables 8 through 23 contain the results from the analysis of the authentic field samples. The P-0 (application day) interval samples were analyzed to a depth of 6 inches and all samples from subsequent intervals were analyzed until two consecutive horizons were found residue free.

No sample contained detectable residues of MAT 7484 oxygen analog or IMATS. These data indicate that significant concentrations of the MAT oxygen analog or the MAT IMATS degradates do not form under field conditions. Because no sample contained detectable residues of MAT oxygen analog or MAT IMATS these results were not reported in a table format. The parent compound MAT 7484 was found only in the 0-6 inch samples, indicating that even when 125% of the 10 year normal precipitation is applied, the compound is immobile in the soil. No residues of MAT 7484 were detectable in samples from the P-343 or the P-366 day samples (detection limit 0.01 μg/g).

5.0 CONCLUSION

The data presented in this report depict the dissipation of MAT 7484 under actual field conditions in Illinois soil. The estimated half-life of MAT 7484 under these conditions was 39 days. Analysis was conducted for two MAT 7484 degradates, the MAT 7484 oxygen analog and IMATS, but residues of these two compounds were not detected in any actual field sample. No residue of MAT 7484 was detected below the 0-6 inch layer, thus leaching of MAT 7484 residues did not occur in this study.

Table 24. Summary of average concentrations of MAT 7484 residues found at each sampling interval.

Interval	Sum of the Average Concentrations at Each Depth (ug/g)[1]
Pre-Application	<0.01
Application	0.17
P-1	0.13
P-2	0.02
P-4	0.11
P-7	0.06
P-10	0.06
P-14	0.06
P-21	0.01
P-28	0.09
P-62	0.02
P-90	0.05
P-198	0.04
P-274	0.02
P-343	<0.01
P-366	<0.01

[1] The sum of the average concentration for each depth.