DATA EVALUATION RECORD

STUDY 7

CHEM 030703 Naptalam sodium salt §164-1

FORMULATION--15--SOLUBLE CONCENTRATE (SC/L)

STUDY ID 40069101
McManus, J.P. 1987c. Soil field dissipation of Alanap in Lafayette, Indiana. Project No. 8552. Unpublished study performed by Uniroyal Chemical Company, Inc., Naugatuck, CT, and submitted by Uniroyal Chemical Company, Inc., Middlebury, CT.

STUDY ID 40488901
McManus, J.P. 1987d. Soil field dissipation of Alanap in Lafayette, Indiana. Replacement for Study ID 40069101. Project No. 8552. Unpublished study performed by Uniroyal Chemical Company, Inc., Naugatuck, CT, and submitted by Uniroyal Chemical Company, Inc., Middlebury, CT.

DIRECT REVIEW TIME = 5

REVIEWED BY: L. Binari TITLE: Staff Scientist

EDITED BY: S. Syslo TITLE: Staff Scientist
K. Patten Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD
TEL: 468-2500

APPROVED BY: H. Manning
TITLE: Microbiologist
ORG: EFGWB/EFED/OPP
TEL: 557-7323

SIGNATURE: H. J. Manning

CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study cannot be used to fulfill data requirements.

2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of naptalam residues.

3. This study is unacceptable for the following reasons:

   the analytical method was not specific, it could not distinguish between naptalam and its degradates; and

   the sampling intervals were inadequate to accurately establish the half-life of the test substance, >50% degraded between two sampling times.

4. Because the analytical method could not distinguish between naptalam and its degradates and because the sampling intervals were inadequate to establish the half-life of the test substance, the problems with this study cannot be resolved with the submission of additional data. A new study is required.

METHODOLOGY:

Naptalam (Alanap-L, formulation not further identified) was sprayed at 4 lb ai/A as a tank-mix with bensulide (Prefar, formulation and source unidentified, 6 lb ai/A) to a field plot (75 x 200 feet) of silty clay loam soil (8.8% sand, 55.6% silt, 35.6% clay, 1.43% organic matter, pH 6.2, CEC 16.53 meq/100 g) planted to melons (type and growth stage unspecified) and located in Lafayette, Indiana; the application occurred on May 20, 1985. Immediately following treatment, the soil was cultivated to a 2-inch depth to incorporate the herbicides plus a soil conditioner (undescribed). An untreated plot (size and location unspecified) was maintained as a control. Ten to fifteen soil cores (diameter unspecified; 0- to 6- and 6- to 12-inch depths) were taken from the treated plot prior to treatment and at 0, 3, 7, 14, 30, and 60 days posttreatment. Soil samples were stored (storage conditions were not adequately described) approximately 520 days prior to analysis.

Soil samples were thawed, air-dried, ground, and homogenized. A subsample (50 g) was distilled with a mixture of 50% sodium hydroxide solution:distilled water:20% titanous trichloride (250:100:5, v;v:v), antifoam solution, and mossy zinc to hydrolyze naptalam and its degradate N-1-naphthylphthalimide to 1-naphthylamine. The distillate was collected, and an aliquot was analyzed for total residues as 1-naphthylamine using reverse-phase HPLC with acetonitrile:0.15 M phosphoric acid (30:70, v:v) as the mobile phase and an electrochemical detector fitted with a glassy carbon electrode. The detection limit was 0.1 ppm. Recovery efficiencies from control soil fortified with naptalam at 0.15-1.02 ppm ranged from 94.1 to 113.3% of the applied (Appendix II). Concentrations of total naptalam residues detected in the field soil samples were corrected for the storage stability recovery (0.874).

DATA SUMMARY:

Total naptalam residues (measured as 1-naphthylamine) dissipated with an observed half-life of 3-7 days in the 0- to 6-inch depth of silty clay loam soil planted to melon in Indiana that was treated with a tank mixture of naptalam (Alanap-L, formulation not further identified) at 4 lb ai/A and bensulide (Prefar, formulation unspecified) at 6 lb ai/A on May 20, 1985. In the 0- to 6-inch soil depth, total naptalam residues decreased from 1.1 ppm immediately posttreatment to 0.8 ppm at 3 days, $\leq$0.2 ppm at 7-30 days, and <0.1 ppm (detection limit) at 60 days posttreatment (Table II). In the 6- to 12-inch soil depth, total naptalam residues were 0.12 ppm immediately posttreatment, <0.1 ppm at 3 days, 0.4 ppm at 7 days, and were not detected (<0.1 ppm) after 14 days.

During the study, rainfall plus irrigation totaled 6.86 inches and air temperatures ranged from 44 to 92 F.

COMMENTS:

1. The analytical method was not specific; it could not distinguish between naptalam and its degradates. The analytical procedure hydrolyzed naptalam and its degradate N-1-naphthylphthalimide to 1-naphthylamine.

2. The sampling intervals were inadequate to accurately establish the half-life of the test substance; >50% degraded between two sampling times. Between 3 and 7 days posttreatment, the concentration of alanap (presented as total 1-naphtylamine residues) in the 0- to 6-inch depth decreased from 0.8 ppm to <0.1 ppm (the detection limit) [Table II].

3. Two versions of this study were submitted (MRIDs 40488901 and 40069101), with MRID 40488901 being submitted to replace 40069101. The only apparent difference between the two reports was that in a storage stability study in soil (Appendix II in both reports) the peak heights used to generate a standard curve were different (Table II in each report).

   Soil samples were fortified with naptalam at 1.0 ppm and stored frozen (-20 to -30 C) for 8 months. After 8 months of storage, it was determined in MRID 40069101 that recovery of naptalam residues (determined as 1-naphthylamine) was only 79.8% (standard curve with r^2 = 0.9978; Table II); the recovery from the fortified control soil was reported to be 96.8%. Concentrations of total naptalam residues detected in the field soil samples were corrected for the storage stability recovery (0.798). However, in MRID 40488901, a new standard curve was presented (registrant-calculated r^2 = 0.9993 and reviewer-calculated r^2 = 0.9804; Table II) which resulted in a recovery of 87.4%; recovery from a fortified control was 96.8%. Naptalam residues in all of the field soil samples were then

recalculated based on the new recovery value (0.874). The registrant provided no explanation as to the source of the new peak heights or why it was necessary to generate a new standard curve. The values used in this review were taken from MRID 40488901.

In addition, the recovery values for naptalam residues as determined in this report are not consistent with other storage stability studies reviewed in this report. In another terrestrial field dissipation study in this report (Study 6, MRID 41385403), total naptalam residues were stable when soil samples were fortified with naptalam at 0.5 ppm and stored frozen for up to 1 year.

4. Soil samples were stored for approximately 520 days prior to analysis. The soil samples were stored frozen after they were received at Uniroyal Chemical, Naugatuck, CT, on August 6, 1985. Storage of the samples prior to that was not described (samples were taken from the test plot between May 20 and July 19, 1985). Soil samples were analyzed October 20-23, 1986.

5. The soil was not sampled deep enough to define the extent of leaching. Soil samples were taken only to a depth of 12 inches; total naptalam residues were detected in the 6- to 12-inch soil depth at a maximum 0.4 ppm (Table II). To define the extent of leaching, samples should be taken deep enough to show no detectable residues at two consecutive sample depths.

6. It was reported that ten to fifteen soil cores were taken for each sampling interval, but only one data value for total naptalam residues was presented. It could not be determined if the soil cores were composited prior to extraction, or if the soil samples were analyzed individually and the results were averaged following analysis.

7. The test substance was incompletely characterized. The test substance was described as Alanap-L, but the formulation concentration was not reported.

8. The test substance was applied as a tank mixture with bensulide (Prefar, formulation not identified). It is preferred that the test substance be applied alone.

9. Except for the irrigation of the test plot, field maintenance practices were not described. An uncharacterized soil conditioner was applied to the soil at treatment.

10. Soil temperature data were not provided.

11. The depth to the water table was 35 feet, and the slope of the field was <1%.

Project No. 8552

MRID 40488901

Table II. Analysis of Soil for ALANAP as Total 1-Naphthylamine Residue in Soil Regions 0-6" and 6-12"

Time	ppm Residue*	
	0-6"	6-12"
Pretreatment		
Day 0	1.1	0.12
Day 3	0.8	<0.1
Day 7	<0.1	0.4
Day 14	0.2	ND
Day 30	0.2	ND
Day 60	<0.1	ND

* Limit of detection 0.1 ppm.

Project No. 8552

MRID 40069101

Table II. Analysis of Soil for ALANAP as Total 1-Naphthylamine Residue in Soil Regions 0-6" and 6-12"

Time	ppm Residue*	
	0-6"	6-12"
Pretreatment		
Day 0	1.3	0.12
Day 3	0.8	<0.1
Day 7	<0.1	0.4
Day 14	0.2	ND
Day 30	0.2	ND
Day 60	<0.1	ND

* Limit of detection 0.1 ppm.

Project No. 8628

MRID 40488901

Appendix II

Table II. ALANAP Recovery from the 1N-010 Soil Fortified at 1 ppm and Freezer-Aged

Sample ID AC-917-68, - 82	Months in the freezer	Sample size (g)	1-NA Peak Ht. mm	1-NA Recovered µg/mL (1)	1-NA Recovered µg (2)	ALANAP Equivalent µg (3)
1	8	49.80	88	0.097	19.4	42.5
2	8	50.30	97	0.107	21.4	46.8
3	8	60.83	110	0.120	24.0	52.5
					Total	141.8

Total sample wt, 160.93

	% Recovery
ALANAP Recovered: 141.8 µg	87.4
ALANAP Added to 160.93 g sample: 162.3 µg	

1. From linear regression analysis of std. plot
2. 1-NA, µg/mL x sample tot. vol. (200 mL)
3. 1-NA, µg/0.457 (corrected for mol. wt.)

1-NA Std. Plot (AC-917-63)

µg/mL	Peak Ht. mm
0.148	140
0.099	84
0.050	22
0.025	23

r = 0.9993

MRID 40069101

Appendix II

Table II. ALANAP Recovery from the 1N-010 Soil Fortified at 1 ppm and Freezer-Aged

Sample ID AC-917-68, - 82	Months in the freezer	Sample size (g)	1-NA Peak Ht. mm	1-NA Recovered µg/mL (1)	1-NA Recovered µg (2)	ALANAP Equivalent µg (3)
1	8	49.80	88	0.089	17.8	38.9
2	8	50.30	97	0.097	19.4	42.5
3	8	60.83	110	0.110	22.0	48.1
Total sample wt,		160.93				Total 129.5

	% Recovery
ALANAP Recovered: 129.6 µg	79.8
ALANAP Added to 160.93 g sample: 162.3 µg	

1. From linear regression analysis of std. plot
2. 1-NA, µg/mL x sample tot. vol. (200 mL)
3. 1-NA, µg/0.457 (corrected for mol. wt.)

1-NA Std. Plot (AC-917-63)

µg/mL	Peak Ht. mm
0.148	154
0.099	94
0.050	45
0.025	23

r = 0.9978

Appendix II

1-Naphthylamine Recovery from Control Soil Fortified with ALANAP

Sample ID	Sample size g	ALANAP Added 8.23 µg/mL mL	ppm (1)	1-NA Recovered peak ht. mm	µg/mL (2)	ALANAP Equivalent ppm (3)	%Recovery (X)	(X̄)	±(S.D.)
1. (AC-917-53)	50.1	1	0.15	14	0.02	0.17	113.3	113.3	-
2. (AC-917-53)	50.2	1	0.15	14	0.02	0.17	113.3		
3. (AC-917-61)	52.4	4	0.56	54	0.07	0.58	103.6	104.5	1.3
4. (AC-917-61)	52.2	4	0.56	56	0.07	0.59	105.4		
5. (AC-917-61)	52.9	7	0.97	102	0.12	0.99	102.1	96.8	4.6
6. (AC-917-61)	50.4	7	1.02	98	0.11	0.96	94.1		
7. (AC-917-53)	50.4	7	1.02	100	0.11	0.96	94.1		

1. ALANAP, µg/mL x mL added x 0.894/sample wt, g, where 0.894 is the standard purity
2. Values obtained by linear regression analysis of the 1-NA standards.
3. $\frac{\mu g/mL\ (1\text{-NA}) \times 200\ mL}{(0.457)\ (\text{sample wt})}$, where 200 mL is the analyte volume and 0.457 is the correction for molecular weight.

1-NA Std. Plot (AC-917-54)

µg/mL	peak ht. mm
0.177	158
0.117	104
0.059	47
0.030	23

r = 0.9997

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RESULTS AND DISCUSSION

A melon field in Lafayette, Indiana was treated under realistic conditions with ALANAP-L at 8 quarts plus Prefar at 6 quarts per acre, (active amounts: ALANAP 4 lbs/A and Prefar 6 lbs/A). The herbicides were incorporated immediately with a soil conditioner to a depth of 2 inches. Soil samples were taken with a standard soil core sampler with 10 to 15 samples taken at each date in an X pattern across the plot. Sample times were pretreatment, immediately post treatment (zero time), 3 days, 7 days, 14 days, 30 days and 60 days. At each time point, samples were taken at 0-6" and 6-12" soil regions. Weather conditions over the course of the study are included in Appendix I of this report. Soil characteristics are shown in Table I.

All soil samples were analyzed at UNIROYAL laboratories for residues of ALANAP. The method and results of these analysis are included in Appendix II of this report.

Soils were analyzed for total 1-naphthylamine content and reported as the sodium salt of ALANAP. The residues dissipated very rapidly from 1.0 ppm on day zero to less than 0.1 ppm on day seven in the 0-6" soil region. There was no detectable 1-naphthylamine containing residues in the 6-12" soil region after fourteen days. The analysis results are summarized in Table II. The data are represented in Figure 1 with the semilog plot showing a half-life of ALANAP in Figure 2.

Project No. 8552

MRID 40069101

RESULTS AND DISCUSSION

A melon field in Lafayette, Indiana was treated under realistic conditions with ALANAP-L at 8 quarts plus Prefar at 6 quarts per acre, (active amounts: ALANAP 4 lbs/A and Prefar 6 lbs/A). The herbicides were incorporated immediately with a soil conditioner to a depth of 2 inches. Soil samples were taken with a standard soil core sampler with 10 to 15 samples taken at each date in an X pattern across the plot. Sample times were pretreatment, immediately post treatment (zero time), 3 days, 7 days, 14 days, 30 days and 60 days. At each time point, samples were taken at 0-6" and 6-12" soil regions. Weather conditions over the course of the study are included in Appendix I of this report. Soil characteristics are shown in Table I.

All soil samples were analyzed at UNIROYAL laboratories for residues of ALANAP. The method and results of these analysis are included in Appendix II of this report.

Soils were analyzed for total 1-naphthylamine content and reported as the sodium salt of ALANAP. The residues dissipated very rapidly from 1.0 ppm on day zero to less than 0.1 ppm on day seven in the 0-6" soil region. There was no detectable 1-naphthylamine containing residues in the 6-12" soil region after fourteen days. The analysis results are summarized in Table II. The data are represented in Figure 1 with the semilog plot showing a half-life of ALANAP in Figure 2.

Project No. 8628

MRID 40488901

RESULTS AND DISCUSSION - Appendix II

The mean recovery from the unaged control (1N-010) soil fortified at 1 ppm with ALANAP was 96.8% ± 4.6%, as shown in the Analytical Method for Determining ALANAP Residue in Soil, Project No. 8627. The recovery from the freezer-aged control (1N-010) soil fortified at the same level was only 87.4%. This indicates that some degradation or binding of ALANAP did occur during the storage of the soils in the freezer. Thus, the lower recovery figure was used to calculate ALANAP residues in this study.

At Day-0, the concentration of ALANAP was 1.1 ppm. It decreased to <0.1 ppm on Day-60 in the 0-6" depth samples. The data and the calculations are summarized in Table III.

For the samples from 6-12" depth, the largest concentration of ALANAP, 0.4 ppm, was found in the Day-7 sample and none was detected in the Day-14 and in all the subsequent samples (Table IV). The migration of ALANAP below 6 inches depth appears to correlate with the precipitation activity reported for the period. It rained on Days 1, 2 and 7.

The dissipation of ALANAP at both depths is shown in Figure 1.

The half-life for ALANAP in Ockley silt loam soil was 6-7 days as determined from a semilog plot of total ALANAP residues found at both depths vs time (Figure 2).

Figure 3 shows typical chromatograms of the analytes from the control soil and the control fortified at 1 ppm with ALANAP.

CONCLUSIONS

ALANAP was found to be a non-persistent herbicide with a half-life of 6-7 days. ALANAP residues were confined, for the most part, to the first six inches of the top soil.

Project No. 8628

MRID 40069101

RESULTS AND DISCUSSION - Appendix II

The mean recovery from the unaged control (1N-010) soil fortified at 1 ppm with ALANAP was 96.8% ± 4.6%, as shown in the Analytical Method for Determining ALANAP Residue in Soil, Project No. 8627. The recovery from the freezer-aged control (1N-010) soil fortified at the same level was only 79.8%. This indicates that some degradation or binding of ALANAP did occur during the storage of the soils in the freezer. Thus, the lower recovery figure was used to calculate ALANAP residues in this study.

At Day-0, the concentration of ALANAP was 1.3 ppm. It decreased to <0.1 ppm on Day-60 in the 0-6" depth samples. The data and the calculations are summarized in Table III.

For the samples from 6-12" depth, the largest concentration of ALANAP, 0.4 ppm, was found in the Day-7 sample and none was detected in the Day-14 and in all the subsequent samples (Table IV). The migration of ALANAP below 6 inches depth appears to correlate with the precipitation activity reported for the period. It rained on Days 1, 2 and 7.

The dissipation of ALANAP at both depths is shown in Figure 1.

The half-life for ALANAP in Ockley silt loam soil was 5 days as determined from a semilog plot of total ALANAP residues found at both depths vs time (Figure 2).

Figure 3 shows typical chromatograms of the analytes from the control soil and the control fortified at 1 ppm with ALANAP.

CONCLUSIONS

ALANAP was found to be a non-persistent herbicide with a half-life of five days. ALANAP residues were confined, for the most part, to the first six inches of the top soil.