Versar inc.

MEMORANDUM

TO: Catherine Joseph

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J. Becker

FROM: Teri Schaeffer
Diane Baxter

DATE: April 29, 1999

SUBJECT: Review of *Determination of Dislodgeable Foliar Residues in Roses Treated with ORTHENE® Turf, Tree & Ornamental Spray (OTTO)* - MRID No. 447639-03

This report reviews *Determination of Dislodgeable Foliar Residues in Roses Treated with ORTHENE® Turf, Tree & Ornamental Spray (OTTO)*, submitted in support of the reregistration requirements for the insecticide acephate. The requirements for this study are specified by the U.S. Environmental Protection Agency's (US-EPA) OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Dislodgeable Foliar Residue Dissipation: Agricultural, [formerly, EPA Assessment Guidelines Subpart K, Reentry Exposure Series 132-1]. Information which may be used to identify the study includes:

Title:	*Determination of Dislodgeable Foliar Residues in Roses Treated with ORTHENE® Turf, Tree & Ornamental Spray (OTTO),* 359 pages
Sponsor:	Joseph L. Powell Valent USA Corporation P.O. Box 8025 Walnut Creek, CA 94596-8025
Performing Laboratory:	Valent U.S.A. Corporation Valent Technical Center 6560 Trinity Court Dublin, CA 94568 Plant Sciences, Inc. 342 Green Valley Road Watsonville, CA 95076
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Author & Study Director:	J.C. Lai
Report Date:	February 12, 1999
Identifying Codes:	MRID # 447639-03; Valent Laboratory Project Identification #V11654

EXECUTIVE SUMMARY

The purpose of this study was to quantify dislodgeable foliar residues (DFR) of the active ingredient (a.i.) acephate and its metabolite methamidophos on rose foliage following two greenhouse applications of ORTHENE® Turf, Tree & Ornamental (OTTO) spray. The study met most of the requirements of the Environmental Protection Agency's (US-EPA) OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Dislodgeable Foliar Residue Dissipation: Agricultural, [formerly, EPA Assessment Guidelines Subpart K, Reentry Exposure Series 132-1].

The most important deviations from EPA-OPPTS guidelines were: (1) predicted foliar residues assuming first-order dissipation kinetics deviated significantly from the actual measured DFR values obtained; (2) it is unclear whether the author corrected raw DFR data for either laboratory or field recovery losses before running the regression analysis; (3) DFR samples were collected from one location, rather than from three geographically distinct locations per formulation type as recommended in the Series 875 guidelines. It should also be noted that the report (see pp. 18-19) may contain a typographical error, since with regard to use of curve-fitting software, two 50 percent dissipation values were given. For acephate, 50 percent dissipation was calculated to occur at either 1.60 days (R^2 = unknown) or 2.03 days (R^2 = 0.961); for methamidophos the calculated value was either 1.03 days (R^2 = unknown) or 1.38 (R^2 = 0.924). (4)

DFR samples were collected from one greenhouse test plot located in Pajaro (Monterey County), California, Region X. The trial was conducted in a glass commercial greenhouse between June 15, 1998 (planting date) and September 17, 1998 (last DFR sampling date). The analytical phase of the study was completed by September 28, 1998.

The highest foliar acephate residue (i.e., 3.56 µg/cm^2) and methamidophos residue levels (i.e., 0.123 µg/cm^2) were found immediately after the second application. Acephate levels dropped below the LOQ 28 days after the second application. The methamidophos levels dropped below the LOQ 14 days after the second application.

The study author calculated dissipation half-life values for acephate and methamidophos using two methods. The first method, log linear least squares regression analysis, assumed first order dissipation kinetics. Considering acephate DFR data from Day 0 to Day 35 after the second application, the calculated half-lives were: (1) acephate - 2.74 days (R^2 = 0.924) and (2) considering methamidophos DFR data from Day 0 to Day 28, methamidophos - 4.63 (R^2 = 0.728).

The second method used employed a curve-fitting program (CurveExpert® v. 1.3) to generate an empirical exponential equation [i.e., $y = ae^{bx}$], from which was calculated the time at which 50 percent of the residues dissipated. The report (see pp. 18-19) may contain a typographical error, since two results are given. For acephate, 50 percent dissipation was calculated to occur at either 1.60 days (R^2 = unknown) or 2.03 days (R^2 = 0.961); for methamidophos the calculated value was either 1.03 days (R^2 = unknown) or 1.38 (R^2 = 0.924).

Versar re-analyzed the same data-set using the Microsoft EXCEL 97® linear regression function, considering Day 0 to Day 35 data, and calculated very similar half-life values: (1) acephate - 3.02 days ($R^2 = 0.93$) and (2) methamidophos - 4.63 days ($R^2 = 0.73$). Versar also calculated a half-life value for the combined residues of acephate and methamidophos. The half-life for combined residues was estimated to be 3.08 days ($R^2 = 0.93$). "Predicted" residues were found to deviate significantly from actual DFR values measured.

The field portion of the study involved a treated plot, divided into three replicate subplots, and a control plot situated at least 100 feet away. Two applications of ORTHENE® Turf, Tree and Ornamental spray were made with a backpack sprayer with a handheld boom, seven days apart. The application rate was 2.15 lb a.i. per acre (maximum label rate) in 215 to 214 gallons of water per acre. Leaf punch samples were collected at the following intervals: just prior to application #1, just after application #1 when the spray had dried, 1 day before application #2, just after application #2, and on Day 1, 2, 3, 5, 7, 10, 14, 21, 28, 35 after the second application. At each interval, three replicate samples were collected from the treated plot and one sample was collected from the control plot.

Sample replicates each consisted of forty 1- inch (2.54 cm) diameter leaf punches collected at each interval, representing a total of 405 cm^2 surface area. (Leaf punches were collected only from leaves which had also been present at the first application). Insecticide residues were dislodged by extracting twice with 100 mL of 0.01% Triton X-100 solution. The extraction was performed by mechanically shaking the leaf punches in the Triton solution for ten minutes. All the samples were dislodged within 4 hours of collection. The dislodged samples were stored frozen until shipment.

Validation of the analytical method was not mentioned in the Study. Laboratory fortification recoveries averaged: (1) for acephate - 87.5 ± 12 percent (n=7) and (2) for methamidophos - 91.7 ± 23 percent (n=7). The limit of detection (LOD) was 0.125 µg (0.0003 µg /cm^2) for acephate and 0.05 µg (0.0001 µg /cm^2) for methamidophos. The limit of quantitation (LOQ) for both acephate and methamidophos was 0.0025 µg /cm^2.

Field fortification samples were prepared in triplicate at two spiking levels. Field spikes were analyzed with field DFR samples collected at the same interval to assure the quality of the samples. The overall average (all fortification levels) recovery was 91.3 ± 12 percent for acephate and 93.4 ± 23 percent for methamidophos. A storage stability study was also conducted and results suggested that the residues were stable during the period of sample storage.

STUDY REVIEW

Study Background

ORTHENE® Turf, Tree and Ornamental Spray (OTTO) is an insecticide used on non-crop areas such as azaleas, camelias, rhododendron, roses, trees, shrubs and turf grass. The active ingredient (a.i.) is technical grade acephate, present at 75 percent by weight.

Chevron Chemical Company has conducted acephate metabolism studies in a variety of plants. The major extractable residue found was the parent compound, acephate (chemical name: O,S-dimethyl acetylphosphoramidothioate; CAS No. 30560-19-1), with minor amounts of methamidophos (chemical name: O,S-dimethyl phosphoramidothioate; CAS 10265-92-6). Therefore, dislodgeable residues of acephate and methamidophos were measured in this study.

This study was submitted in response to a March 1, 1993, Data Call-In notice, and was completed in cooperation with the Agricultural Reentry Task Force. Details on the study design were provided in the protocol in Appendix A of the Study Report.

Test Plots

DFR samples were collected from one greenhouse test plot located in Pajaro (Monterey County), California, Region X. The trial was conducted in a single glass commercial greenhouse between June 15, 1998 (planting date) and September 17, 1998 (last DFR sampling date). The analytical phase of the study was completed on September 28, 1998. The test-site was reportedly representative of a reasonable worst-case scenario for potential exposure to individuals for all of the registered indoor uses of the test product OTTO. The test-site is also said to be representative of the spectrum of "climatic conditions" and rose varieties expected in the intended use areas.

The test-site consisted of one untreated control plot and one treated plot, the latter subdivided into three replicate subplots. Each plot consisted of hydroponically grown roses (var. *Concord*). The *Concord* rose is a commercially grown hybrid tea rose.

The control plot was 40 feet long, consisting of six pot beds, with 3 plants per pot on 4.5 foot centers. The treated plot was 38 feet long, consisting of the same number of pot beds and plants on 4.5 foot centers. [Diagrams depicting the location of the greenhouse and the plots within the greenhouse may be reviewed on pgs. 154-156 of the Study Report.]

The rose crop used in this study was established specifically for this trial and due to the nature of the hydroponic growing method, no previous crop or pesticide history exists. Table 1 lists the maintenance pesticides applied to the rose crop during the year of the trial. According to the registrant the plots were maintained according to normal agricultural practices throughout the growing season.

Table 1. Maintenance Pesticides

Pesticide Used	Active Ingredient(s)	Dates Used
M-Pede® 2 EC	Fatty acid soap	6-17-98
Thiodan® 3 EC	Endosulfan	6-19-98; 7-29-98; 8-8-98; 9-11-98
Avid® 0.15 EC	Abamectin (avermectin)	6-25-98; 8-27-98
Pipron® 84% a.i.	Piperalin (fungicide)	6-25-98; 7-9-98; 7-16-98; 7-21-98; 7-30-98; 8-6-98; 8-10-98; 8-16-98; 8-24-98; 8-28-98; 8-31-98; 9-4-98; 9-9-98; 9-18-98
Systhane® 40% a.i.	Myclobutanil	6-26-98
Mesurol® 75 W	Methiocarb	6-27-98; 7-1-98; 8-6-98; 8-10-98; 9-4-98; 9-18-98
Pentac® 38% a.i.	Dienochlor	7-9-98; 8-3-98; 8-19-98
Dimethoate 2.67 EC	Dimethoate	7-16-98; 8-25-98; 8-31-98
Kaligreen® 82 SP	Potassium bicarbonate	7-30-98
Orthene® OTTO	Acephate	8-6-98; 8-13-98

Materials and Application

OTTO (Batch # VIB009SP-1) was applied on August 6 and August 13, 1998. A Certificate of Product Quality and three Certificates of Analytical Reference Standard forms were provided in the Study Report (pages 316-319).

OTTO was applied with a backpack sprayer equipped with a handheld boom having two nozzles. Sprayers were calibrated on the day of application. The broadcast (wand) was 8 to 16 inches above the canopy. The label specifies a maximum application rate of 1 lb a.i. per 100 gallons for container grown rose nursery stock. The target application rate was 2.15 lbs a.i. per acre in 215 gallons of water. The application rate was within $\pm$ 1 percent of the target rate for the first application and $\pm$ 2 percent of the target rate for the second application.

The label does not specify a minimum application interval. Two foliar applications were made seven days apart. The first application occurred once the roses entered the mature flowering stage. The crop height was between 12 and 14 inches.

Greenhouse Conditions/Meteorology

The relative humidity within the greenhouse ranged from 52% to 97% and the temperature inside the greenhouse ranged from approximately 50°F to 85°F. The extrapolated

light intensity ranged from 64.1 to 303 Langleys/day. Irrigation was conducted daily between the first application and the last sampling interval. The registrant reported that due to the direction of the plant growth, the irrigation fluids never came in contact with the foliage. The registrant did not provide any historical greenhouse meteorological data.

Because the trial took place in a commercial, climate-controlled greenhouse, outdoor meteorological data are not expected to be relevant. Nevertheless, wind speed, wind direction, percent cloud cover and rainfall were recorded (see pgs. 117-137).

Sampling/ DFR Dislodging

Leaf punch samples were collected using a leaf punch sampler at the following intervals: prior to application #1, just after application #1 when the spray had dried, just before application #2, after application #2, and on Day 1, 2, 3, 5, 7, 10, 14, 21, 28, 35 after the second application. At each interval, one sample of untreated leaf punches was collected before triplicate samples were collected from treated plots. Each sample consisted of 40 (~ 2.54 cm diameter) randomly collected leaf disks, from leaves present at the first application. Therefore, a total 405 cm^2 leaf surface area (taking both sides of the leaf surface into account) was collected per sample replicate. At the intervals when field fortification samples were prepared, six more samples were collected from the control plot.

Leaf punch samples were then placed in coolers on blue ice and transported to the field laboratory to be dislodged. Samples were dislodged within 4 hours of collection. The residues were dislodged from the leaf punch samples in 100 mL of a 0.01 percent Triton X-100 aqueous solution. The Triton X-100 solution was decanted into containers after having been mechanically shaken for 10 minutes. This process was then repeated and the rinsates were combined (~ 200 mL). At several sampling intervals, additional untreated leaf punches were dislodged, and the detergent solutions were fortified with mixed solution of acephate and methamidophos.

Sample Storage and Handling

After the samples were dislodged, they were stored frozen until shipment to the analytical laboratory. Once the samples arrived at the analytical laboratory, the samples were logged in and the condition of the samples were noted. The samples were then either defrosted for extraction or placed in the freezer until time to be extracted. Receipt logs were kept and freezer temperatures were monitored and recorded. Copies of these logs were placed in the Study Report.

QA/QC

Sample History

Sample collection dates, shipping, handling, and storage data may be found in Table 2 of the Study Report (see pp. 22-24). Time from the day of sampling to the day of extraction and analysis ranged between 6 and 15 days.

Analytical Methodology

A proprietary analytical method, Method RM-12HE-2, was used. Reportedly, it was validated prior to initiation of the DFR study. The method involved salting the samples with anhydrous sodium sulfate, extraction with ethyl acetate, and analysis via gas chromatography with flame photometric detection. The protocol was provided in Appendix II of the study report.

Calibration curves were generated using a minimum of 4 concentrations of the reference standards. The coefficient of variation (CV) for the response factors for the standards used was ±10 percent or less. [One CV of 10.3% was accepted upon review by the Study Director.] Response factors with the corresponding CVs for the linearity of the data sets were provided in Appendix IV of the study report. The reproducibility of the gas chromatographic system was verified by determining the reproducibility of the standard measurement for each set of samples. The CV was ±10 percent or less.

Limit of Detection (LOD) & Limit of Quantitation (LOQ)

The LOD was 0.125 μg for acephate and 0.05 μg for methamidophos. The LOQ was 1 μg (0.0025 μg/cm^2) for acephate and 1 μg (0.0025 μg/cm^2) for methamidophos.

Laboratory Recovery

Laboratory fortified spikes were analyzed with each set of samples. Fortification levels ranged from 1 μg (LOQ) to 400 μg for acephate and 1 μg (LOQ) to 20 μg for methamidophos. Table 2 shows the recovery data from fortified detergent solutions. The mean percent recovery for acephate was 87.5 ± 12 percent (n=7). The mean percent recovery for methamidophos was 91.7 ± 23 percent (n=7). The majority of the individual recovery values were within EPA's tolerance range (i.e., 70 percent - 120 percent). Four recovery values were just outside the acceptable range: one acephate recovery at 69 percent and three methamidophos recovery values at 122 percent, 68.2 percent, and 69.4 percent). Sample calculations were provided in Appendix II of the Study Report.

Fortified Field Sample Recovery

Acephate and methamidophos field fortifications (field spikes) were prepared in triplicate at two concentrations (2 μg/mL to 400 μg/mL for acephate and 2 μg/mL to 20 μg/mL for methamidophos). The Study Report stated that these field fortified samples were collected at

several sampling intervals but did not provide details on which specific intervals were used. The field fortified samples were analyzed concurrently with the DFR samples. The overall average (all fortification levels) recovery was 91.3 ± 12 percent for acephate and 93.4 ± 23 percent for methamidophos. Individual recovery values are provided in Table 5 of the study report (pp 27-28).

Table 2. Recovery of Acephate & Methamidophos in Laboratory Fortified Samples

Extraction Date	Analyte	µg Fortified in 100 mL*	µg Found in 100 mL*	% Recovery*
14-Aug-98	Acephate	200	174.9	87.5
	Methamidophos	10.0	11.87	119
17-Aug-98	Acephate	200	206.7	103
	Methamidophos	10.0	12.18	122
20-Aug-98	Acephate	10.0	9.07	90.65
	Methamidophos	1.00	0.88	87.55
25-Aug-98	Acephate	10.0	5.98	89.75
	Methamidophos	1.00	0.86	86.25
31-Aug-98	Acephate	20.0	18.04	90.2
	Methamidophos	10.0	8.7	87
9-Sep-98	Acephate	1.00	0.82	82.3
	Methamidophos	1.00	0.71	70.85
25-Sep-98	Acephate	1.00	0.69	69
	Methamidophos	1.00	0.69	69.4

* Duplicate results have been averaged.

Storage Stability

A storage stability study (see page 15) was performed on acephate and methamidophos residues stored in 0.01% Triton X-100 in water, either refrigerated or frozen. The storage intervals ranged from 0 to 43 days. This was appropriate because the longest reported field sample storage interval was 15 days. The results for the stability study were tabulated in Tables 7A and 7B of the Study Report and the analytical data can be found in Appendix VI of the Study Report. Due to low freshly fortified sample recoveries the results for Day 7 are questionable. The results after 43 days of refrigerator storage and freezer storage are summarized in Table 3,

below. The results show that both analytes are stable in detergent solutions stored at approximately -20° C. and at 5° C. for 43 days.

Table 3. Freezer and Refrigerator Storage Stability Recoveries after 43 Days.

Storage Location	Extraction Date	Analyte-Sample #	Residue (µg)	Storage Interval (days)	% Fresh Fortification Recovery	% Recovery	% Corrected Recovery
Freezer	4-21-98	Acep.-		43	84.2		
		SS5B	11.4			91.2	108
		SS6B	12.0			96.0	114
		Meth.-		43	118		
		SS5B	5.09			102	86.3
		SS6B	5.45			109	92.4
Refrigerator	4-21-98	Acep.-		43	84.2		
		SS5B	12.9			103	123
		SS6B	11.9			95.0	113
		Meth.-		43	118		
		SS5B	5.91			118	100
		SS6B	6.06			121	103

Results

The DFR data for each sampling interval are summarized below in Table 4. The highest foliar acephate residue level (i.e., 3.56 µg/cm^2) was found immediately after the second application. The highest methamidophos residue level (i.e., 0.123 µg/cm^2) was also found immediately after the second application. Acephate levels dropped below LOQ 28 days after the second application. The methamidophos levels dropped below LOQ 14 days after the second application.

The study author calculated dissipation half-life values for acephate and methamidophos using two methods. The first method, log linear least squares regression analysis, assumed first order dissipation kinetics. Considering acephate DFR data from Day 0 to Day 35 after the second application, the calculated half-lives were: (1) acephate - 2.74 days ($R^2 = 0.924$) and (2) considering methamidophos DFR data from Day 0 to Day 28, methamidophos - 4.63 ($R^2 = 0.728$).

The second method used employed a curve-fitting program (CurveExpert® v. 1.3) to generate an empirical exponential equation [i.e., $y = ae^{bx}$], from which was calculated the time at which 50 percent of the residues dissipated. The report (see pp. 18-19) may contain a typographical error, since two results are given. For acephate, 50 percent dissipation was

calculated to occur at either 1.60 days (R^2 = unknown) or 2.03 days (R^2 = 0.961); for methamidophos the calculated value was either 1.03 days (R^2 = unknown) or 1.38 (R^2 = 0.924).

Table 4. Dislodgeable Foliar Residues of Acephate and Methamidophos on Roses

	Acephate Residues on leaves (µg/cm²)				Methamidophos Residues on leaves (µg/cm²)			
Sampling interval	Repli. 1	Repli. 2	Repli. 3	Average	Repli. 1	Repli. 2	Repli. 3	Average
Pre-Application 1	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025
Post-Application 1	2.13	2.60	2.09	2.27	0.015	0.024	0.024	0.021
Pre-Application 2	0.226	0.202	0.171	0.20	0.011	0.011	0.008	0.01
Post-Application 2	2.69	3.08	3.56	3.11	0.095	0.118	0.123	0.112
1 day after appln 2	2.93	2.67	2.61	8.21	0.137	0.085	0.082	0.304
2	2.05	2.36	1.58	2.00	0.024	0.024	0.015	0.021
3	0.443	0.424	0.599	0.489	0.010	0.008	0.011	0.010
5	0.242	0.138	0.518	0.299	0.006	0.005	0.008	0.006
7	0.255	0.125	0.278	0.219	0.009	0.005	0.008	0.007
10	0.121	0.043	0.165	0.069	0.005	0.003	0.004	0.012
14	0.022	0.018	0.032	0.024	0.002	0.002	0.002	0.002
21	0.008	0.014	0.016	0.013	0.001	0.002	0.002	0.002
28	0.003	0.002	0.002	0.002	0.001	0.001	0.001	0.001
35	0.001	0.001	0.001	0.001				

Versar re-analyzed the same data-set using the Microsoft EXCEL 97® linear regression function, considering Day 0 to Day 35 data, and calculated very similar half-life values: (1) acephate - 3.02 days (R^2 = 0.93) and (2) methamidophos - 4.63 days (R^2 = 0.73). (See Appendices A and B of this review). Versar also calculated a half-life value for the combined residues of acephate and methamidophos. The half-life for combined residues was estimated to be 3.076 days (R^2 = 0.93). (See Appendix C). "Predicted" residues were found to deviate significantly from actual DFR values measured. An alternative approach might be needed to provide a better description of the DFR dissipation data. See Table 5, below, for a summary of the author's and Versar's calculated half-lives.

Table 5. Half-life for Acephate and Methamidophos as Estimated by the Registrant and Versar

	Acephate		Methamidophos		Combined Residues	
	Half-life (days)	Correlation Coeffi. (R^2)	Half-life (days)	Correlation Coeffi. (R^2)	Half-life (days)	Correlation Coeffi. (R^2)
Calculated by Valent U.S.A	2.74	0.924	4.63	0.728	--	--
Calculated by Versar	3.02	0.935	4.63	0.728	3.08	0.935

Data Variability

Versar examined data variability as part of the linear regression exercise and found that coefficients of variance for replicate samples ranged from 0 to 65.7% percent for acephate residues, from 0% to 34.6% percent for methamidophos residues. There are no specific requirements concerning the variability of replicate samples in the Pesticide Assessment Guidelines.

Compliance Checklist

Compliance with OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Dislodgeable Foliar Residue Dissipation: Agricultural, [formerly, EPA Assessment Guidelines Subpart K, Reentry Exposure Series 132-1] is critical. The itemized checklist below describes compliance with the major technical aspects of OPPTS 875.2100, and is based on the "Checklist for Residue Dissipation Data" used for study review by the U.S. EPA/OPP/HED. Additional data gaps identified in the study (not covered by the checklist) are also presented below:

- *Typical end use product of the active ingredient used.* This criterion was met. The product label was provided with the study report.

- *Site(s) treated representative of reasonable worst-case climatic conditions expected in intended use areas.* This criterion was probably met. The study was performed in an indoor, climate-controlled greenhouse. A detailed analysis of historic greenhouse climatic data for hydroponically-grown roses was not presented. Therefore, whether conditions at this site were "representative of reasonable worst-case .... conditions" either at this site or across the U.S. cannot be determined.

- *End use product applied by application method recommended for the crop. Application rate given and should be at the least dilution and highest, label permitted, application rate.* This criterion was met.

- *Applications occurred at time of season that the end-use product is normally to achieve intended pest control.* This criterion was met.

- *If multiple applications are made, the minimum allowable interval between applications should be used.* This criterion was probably met. The label does not specify a minimum application interval for indoor container grown nursery-stock roses. Two applications were made 7 days apart.

- *Meteorological conditions including temperature, wind speed, daily rainfall, and humidity provided for the duration of the study.* This criterion was met. Greenhouse temperature and relative humidity values were provided. In addition, outdoor temperature, humidity, wind speed, wind direction and rainfall data were reported.

- *Reported residue dissipation data in conjunction with toxicity data must be sufficient to support the determination of a reentry interval.* This criterion was partially met. Residue dissipation data were provided. Toxicity data were not provided.

- *Residue storage stability, method efficiency (residue recovery), and limit of quantitation provided.* This criterion was met. Storage stability recovery, fortified field recovery, laboratory method recovery were reported. The limit of quantitation was 0.0025 $\mu g/cm^2$ for both acephate and methamidophos.

- *Duplicate foliar and/or soil samples collected at each collection period.* This criterion was met. Triplicate foliar samples were collected at each sampling interval. The roses were not grown in soil, therefore, soil samples were not collected.

- *Control and baseline foliar or soil samples collected.* The criterion was met. Control samples were collected from the control plot at each sampling interval. Blank detergent solution samples were also analyzed.

- *Sufficient collection times to establish dissipation curve.* This criterion was met. Samples were collected just before and just after both applications, and 1, 2, 3, 5, 7, 10, 14, 21, 28, and 35 days after the second application. By Day 35, residues were below the LOQ.

- *Foliar residue data expressed as $\mu g/cm^2$ leaf surface area.* This criterion was met. All residue data were reported in $\mu g/cm^2$.

- *A minimum of 400 cm^2 foliar material was collected per DFR sample.* The criterion was met.

Pertinent data gaps and other issues critical to the scientific validity and regulatory acceptability of the study (i.e., Subdivision K compliance), not already addressed, are presented below. The following issues were identified:

- The report (see pp. 18-19) may contain a typographical error, since two values are given. For acephate, 50 percent dissipation was calculated using a curve-fitting software to occur at either 1.60 days (R^2 = unknown) or 2.03 days (R^2 = 0.961); for methamidophos the calculated value was either 1.03 days (R^2 = unknown) or 1.38 (R^2 = 0.924).

- It is unclear whether the registrants corrected raw DFR data for laboratory or field recovery losses before running their regression analysis.

- The Study Report stated that field fortified recovery samples were collected at several sampling intervals, however no details were provided on which specific intervals were used.

- "Predicted" residues calculated based on first-order kinetics deviated significantly from the actual DFR data. An alternative approach might be needed to provide a better description of the residue dissipation data.

- OPPTS 875.2100 (an Update to Subdivision K) specifically requires that the DFR samples be typically collected from at least three geographically distinct locations for each crop. In this study, DFR samples were collected only from one location. There are no specific guidelines governing greenhouse studies concerning the acceptable number of trial locations even though greenhouse environments are controlled and should not differ from one to the next significantly.

Appendix A

Versar's Regression Analysis for DFR Acephate Data

Regression Analysis: Summary Output for Acephate in CA

Regression Statistics	
Multiple R	0.966961
R Square	0.935014
Adjusted R^2	0.932918
Standard Error	0.70246
Observations	33

ANOVA

	df	*SS*	*MS*	*F*	*Signif. F*
Regression	1	220.0922	220.0922	446.0273	5.8227E-20
Residual	31	15.29695	0.49345		
Total	32	235.3892			

	Coeff.	*Std. Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	0.416819	0.17456	2.387832	0.0232275	0.06080213	0.772835587
Slope	-0.22968	0.010875	-21.1194	5.823E-20	-0.251858973	-0.207498434

Half Life = 3.017899 Days

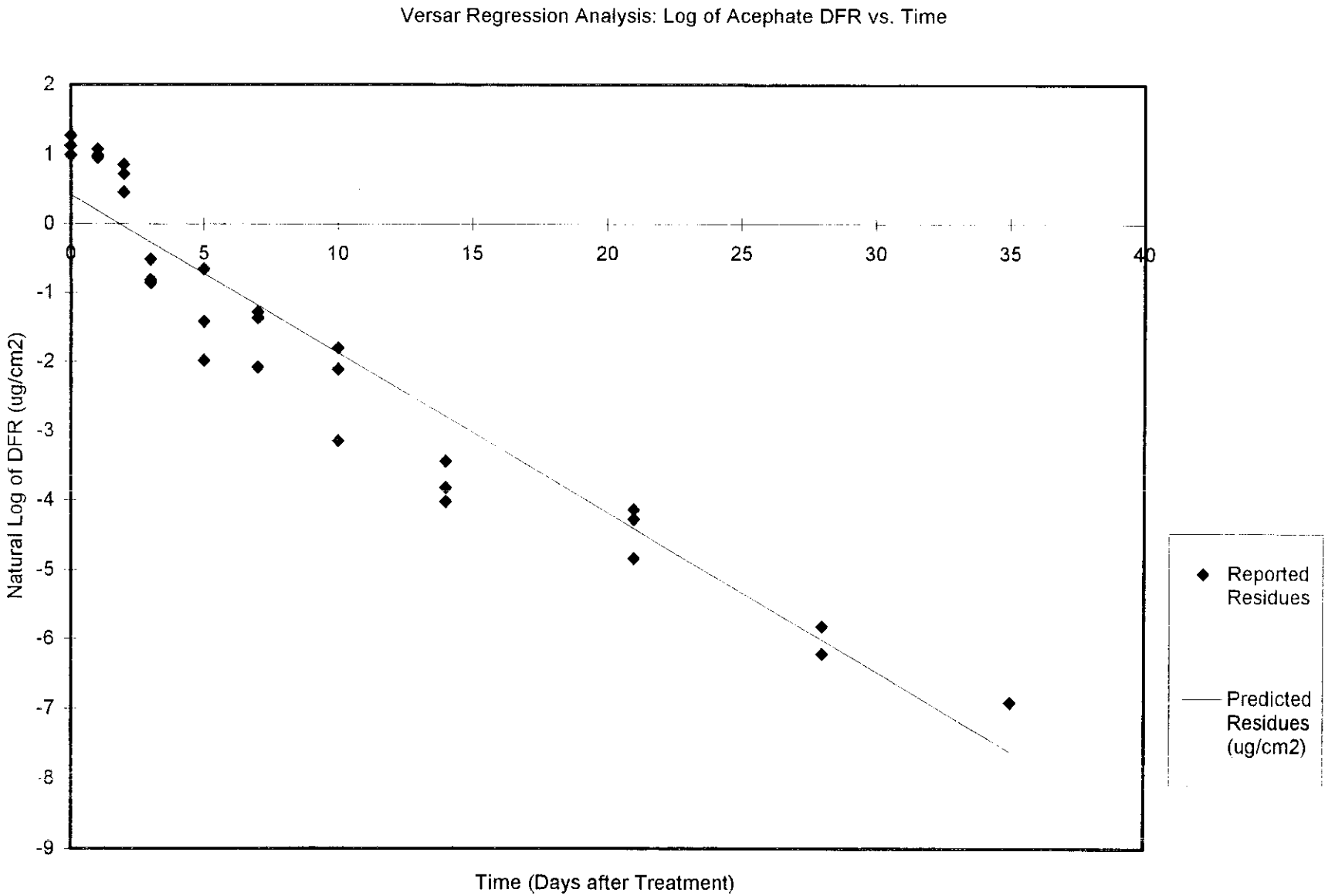
Predicted DFR Levels

Time (Days)	Residue (ug/cm2)
0	1.517128
1	1.205796
2	0.958354
3	0.761689
4	0.605382
5	0.481151
6	0.382413
7	0.303938
8	0.241566
9	0.191994
10	0.152595
11	0.121281
12	0.096393
13	0.076612
14	0.06089
15	0.048395
16	0.038464
17	0.030571
18	0.024297
19	0.019311
20	0.015348

Time (Days)	Residue (ug/cm2)
21	0.0121986
22	0.0096953
23	0.0077057
24	0.0061244
25	0.0048676
26	0.0038687
27	0.0030748
28	0.0024438
29	0.0019423
30	0.0015437
31	0.001227
32	0.0009752
33	0.0007751
34	0.000616
35	0.0004896

Regression Analysis: Means and CVs for Acephate in CA

Days after Last Treatment	Residues (ug/cm2)	Mean (ug/cm2)	Standard Deviation (ug/cm2)	Coefficient of Variation (%)
0	2.69	3.11	0.436	14
	3.08			
	3.56			
1	2.93	2.74	0.17	6.21
	2.67			
	2.61			
2	2.05	2	0.393	19.6
	2.36			
	1.58			
3	0.443	0.489	0.096	19.6
	0.424			
	0.599			
5	0.242	0.299	0.196	65.7
	0.138			
	0.518			
7	0.255	0.219	0.0825	37.7
	0.125			
	0.278			
10	0.121	0.11	0.0618	56.2
	0.043			
	0.165			
14	0.022	0.024	0.00721	30
	0.018			
	0.032			
21	0.008	0.0127	0.00416	32.8
	0.014			
	0.016			
28	0.003	0.00233	0.000577	24.8
	0.002			
	0.002			
35	0.001	0.001	0	0
	0.001			
	0.001			


Versar Regression Analysis: Log of Acephate DFR vs. Time
Natural Log of DFR (ug/cm2)
2
1
0
-1
-2
-3
-4
-5
-6
-7
-8
-9
0
5
10
15
20
25
30
35
40
Time (Days after Treatment)
Reported Residues
Predicted Residues (ug/cm2)

Appendix B

Versar's Regression Analysis for DFR Methamidophos Data

Regression Analysis: Summary Output for Methamidophos in CA

Regression Statistics	
Multiple R	0.853246
R Square	0.728029
Adjusted R^2	0.718316
Standard Error	0.836086
Observations	30

ANOVA

	df	*SS*	*MS*	*F*	*Signif. F*
Regression	1	52.39459	52.39459	74.952167	2.09636E-09
Residual	28	19.57313	0.69904		
Total	29	71.96772			

	Coeff.	*Std. Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	-3.45762	0.219114	-15.78	1.825E-15	-3.906457646	-3.008785885
Slope	-0.14955	0.017274	-8.65749	2.096E-09	-0.184933682	-0.114165255

Half Life = 4.634902 Days

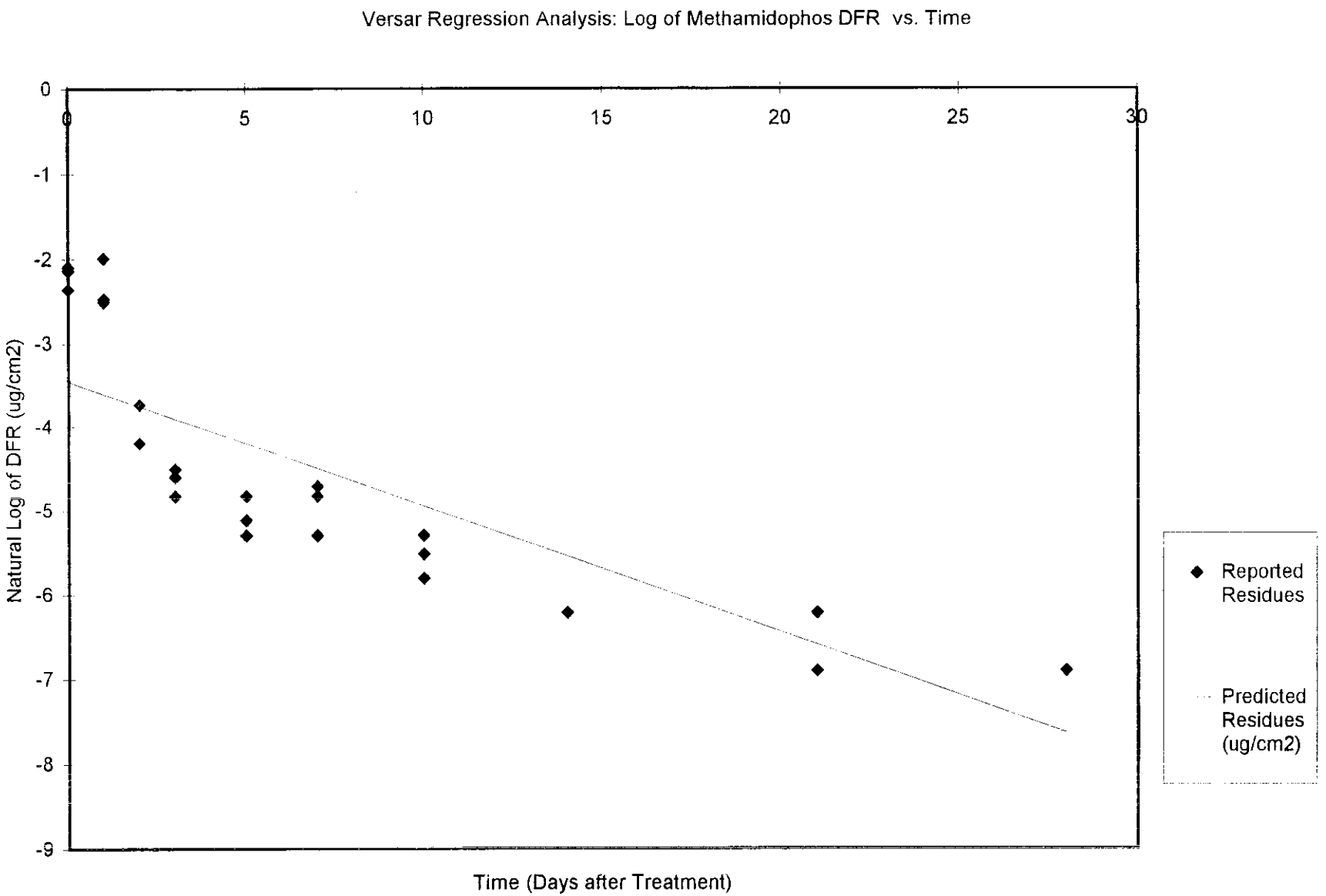
Predicted DFR Levels

Time (Days)	Residue (ug/cm2)
0	0.031505
1	0.027128
2	0.02336
3	0.020115
4	0.017321
5	0.014915
6	0.012843
7	0.011059
8	0.009523
9	0.0082
10	0.007061
11	0.006081
12	0.005236
13	0.004509
14	0.003882
15	0.003343
16	0.002879
17	0.002479
18	0.002135
19	0.001838
20	0.001583

Time (Days)	Residue (ug/cm2)
21	0.0013629
22	0.0011736
23	0.0010106
24	0.0008702
25	0.0007493
26	0.0006452
27	0.0005556
28	0.0004784
29	0.000412
30	0.0003547
31	0.0003055
32	0.000263
33	0.0002265
34	0.000195
35	0.0001679

Regression Analysis: Means and CVs for Methamidophos in CA

Days after Last Treatment	Residues (ug/cm2)	Mean (ug/cm2)	Standard Deviation (ug/cm2)	Coefficient of Variation (%)
0	0.095	0.112	0.0149	13.3
	0.118			
	0.123			
1	0.137	0.101	0.0309	30.6
	0.085			
	0.082			
2	0.024	0.021	0.0052	24.7
	0.024			
	0.015			
3	0.01	0.00967	0.00153	15.8
	0.008			
	0.011			
5	0.006	0.00633	0.00153	24.1
	0.005			
	0.008			
7	0.009	0.00733	0.00208	28.4
	0.005			
	0.008			
10	0.005	0.004	0.001	25
	0.003			
	0.004			
14	0.002	0.002	0	0
	0.002			
	0.002			
21	0.001	0.00167	0.000577	34.6
	0.002			
	0.002			
28	0.001	0.001	0	0
	0.001			
	0.001			


Versar Regression Analysis: Log of Methamidophos DFR vs. Time
Natural Log of DFR (ug/cm2)
0
-1
-2
-3
-4
-5
-6
-7
-8
-9
0
5
10
15
20
25
30
Time (Days after Treatment)
Reported Residues
Predicted Residues (ug/cm2)

Appendix C

Versar's Regression Analysis for Combined Residues Acephate and Methamidophos

Regression Analysis: Summary Output for Combined in CA

Regression Statistics	
Multiple R	0.966879
R Square	0.934855
Adjusted R^2	0.932754
Standard Error	0.690116
Observations	33

ANOVA

	df	*SS*	*MS*	*F*	*Signif. F*
Regression	1	211.8706	211.8706	444.86335	6.04789E-20
Residual	31	14.76406	0.47626		
Total	32	226.6347			

	Coeff.	*Std. Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	0.437969	0.171492	2.553871	0.0157893	0.088207983	0.787729113
Slope	-0.22535	0.010684	-21.0918	6.048E-20	-0.24713853	-0.203557523

Half Life = 3.075896 Days

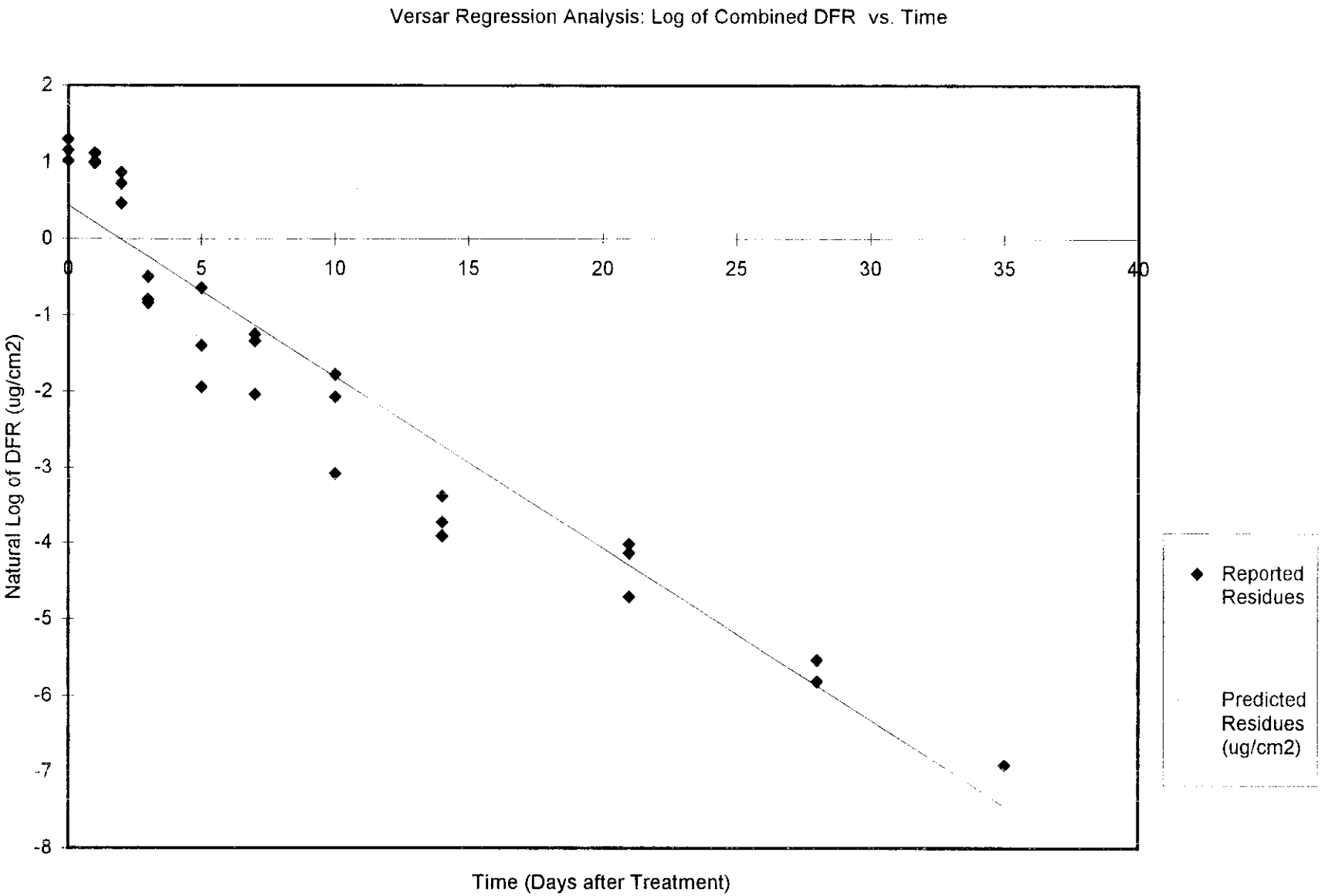
Predicted DFR Levels

Time (Days)	Residue (ug/cm2)
0	1.549556
1	1.236915
2	0.987353
3	0.788143
4	0.629126
5	0.502193
6	0.400869
7	0.319989
8	0.255428
9	0.203892
10	0.162755
11	0.129917
12	0.103705
13	0.082781
14	0.066079
15	0.052747
16	0.042105
17	0.033609
18	0.026828
19	0.021415
20	0.017095

Time (Days)	Residue (ug/cm2)
21	0.0136456
22	0.0108924
23	0.0086947
24	0.0069405
25	0.0055402
26	0.0044224
27	0.0035301
28	0.0028179
29	0.0022493
30	0.0017955
31	0.0014332
32	0.0011441
33	0.0009132
34	0.000729
35	0.0005819

Regression Analysis: Means and CVs for Combined in CA

Days after Last Treatment	Residues (ug/cm2)	Mean (ug/cm2)	Standard Deviation (ug/cm2)	Coefficient of Variation (%)
0	2.785	3.22	0.449	14
	3.198			
	3.683			
1	3.067	2.84	0.201	7.07
	2.755			
	2.692			
2	2.074	2.02	0.398	19.7
	2.384			
	1.595			
3	0.453	0.498	0.0973	19.5
	0.432			
	0.61			
5	0.248	0.306	0.198	64.7
	0.143			
	0.526			
7	0.264	0.227	0.0844	37.2
	0.13			
	0.286			
10	0.126	0.114	0.0624	54.8
	0.046			
	0.169			
14	0.024	0.026	0.00721	27.7
	0.02			
	0.034			
21	0.009	0.0143	0.00473	33
	0.016			
	0.018			
28	0.004	0.00333	0.000577	17.3
	0.003			
	0.003			
35	0.001	0.001	0	0
	0.001			
	0.001			


Versar Regression Analysis: Log of Combined DFR vs. Time
Natural Log of DFR (ug/cm2)
2
1
0
-1
-2
-3
-4
-5
-6
-7
-8
0
5
10
15
20
25
30
35
40
Time (Days after Treatment)
Reported Residues
Predicted Residues (ug/cm2)

13544

R132652

Chemical: Acephate

PC Code:
103301

HED File Code: 19050 Versar DER Warning: May not have been QAed by EPA- - CONTRACTOR DRAFT DOCUMENT

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