Versar inc.



**MEMORANDUM**

**TO:** S. Hanley cc: 3771.101
D.Baxter

**FROM:** Mike Huang
Diane Baxter

**DATE:** April 27, 1999

**SUBJECT:** Review of *Determination of Dislodgeable Foliar Residues in Cauliflower Treated with Acephate* (MRID No. 447639-04)

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This report reviews *Determination of Dislodgeable Foliar Residues in Cauliflower Treated with Acephate*, submitted in support of the reregistration requirements for the insecticide, ORTHENE® 75 Soluble Powder. 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 Cauliflower Treated with Acephate*, 387 pages |
| Sponsor: | Joseph L. Powell<br>Valent U.S.A. Corporation<br>6560 Trinity Court<br>Dublin, CA 94568 |
| Performing Laboratory:<br>(Field Study) | Plant Sciences, Inc.<br>342 Green Valley Road<br>Watsonville, CA 95076 |
| Analytical Laboratory: | Valent U.S.A. Corporation<br>Valent Technical Center<br>6560 Trinity Court<br>Dublin, CA 94568 |
| Author & Study Director: | J.C. Lai |
| Report Date: | February 18, 1999 |
| Identifying Codes: | MRID # 447639-04, Valent Laboratory Project Ident. V10985 |

# Executive Summary

The purpose of this study was to quantify dislodgeable foliar residues (DFR) of the active ingredient acephate and its metabolite methamidophos on cauliflower following two broadcast applications of ORTHENE® 75 SP. 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) the reproducibility and the representativeness of the replicate samples collected at the sampling interval was poor; (2) the highest foliar acephate residue of 0.464 $\mu g/cm^2$ was measured on Day 3 after the second application. The average Day 0 DFR value was 0.2 $\mu g/cm^2$, which is significantly lower; (3) it is unclear whether the author corrected raw DFR data for either laboratory or field recovery losses before running the regression analysis; (4) DFR samples were collected from one location, rather than from three geographically distinct locations per formulation type as recommended in the Series 875 guidelines.

The usage scenario evaluated in this study was acephate application to a low-growing, waxy foliage crop, in a hot and dry climatic zone. The study was conducted between July 10, 1998 and August 24, 1998 in Watsonville, Santa Cruz County, California. The test plot consisted of a treated plot, divided into three replicate subplots, and a control plot. The treated plot and control plot were 100 feet apart. During the period of the DFR study, there was a total rainfall of 0.04 inches. No rain events occurred within 24 hours after the application of the pesticide. The cauliflower was maintained by normal agricultural practices, which included in-furrow irrigation. Water was not applied to plant leaves.

ORTHENE® 75 SP was applied according to label direction at the maximum application rate (i.e. 1.0 lb a.i. per acre), with a tractor-mounted boom sprayer. Two applications were made, 10 days apart. Leaf punch samples were collected 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, three sample replicates were collected from the treated plot and one sample from the control plot. At the intervals when field fortification samples were prepared, six more samples were collected from the control plot.

The samples consist of forty 1-inch (2.54 cm) diameter leaf punches collected at each interval, representing a total of 405 $cm^2$ surface area (counting both sides of leaf punches). The leaf punches were collected only from leaves which had also received the first application. Residues were dislodged from samples by extracting twice with 100 mL of 0.01% Triton X-100 solution. The extraction was performed by shaking the leaf punches in the Triton solution for ten

minutes. The dislodged samples were stored frozen until shipment. All the samples were dislodged within 4 hours of collection.

A proprietary analytical method (i.e. Method RM-12HE-2) was used to quantify acephate and methamidophos DFRs. The method was validated before the study was initiated. The limit of detection 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 for both acephate and methamidophos was 0.0025 µg /cm$^2$.

Laboratory fortification samples were prepared as follows: (1) acephate: at 1.0, 10, 20, 200, 800 µg per 100 mL 0.01% Triton solution, and (2) methamidophos: 1.0 10, 40 µg per 100 mL of the 0.01% Triton solution. These lab fortified samples were analyzed with each set of samples to evaluate the performance of the analytical method. Laboratory fortification recoveries averaged 87.0 percent ± 14% CV for acephate and 96.1 percent ± 19% CV for methamidophos. Field fortification samples were prepared in three replicates at two spiking levels at six sampling intervals. Field fortification levels were: (1) acephate - 2.0, 20, 40, and 400 µg per 200 mL of the Triton solution, and (2) methamidophos - 2.0 and 20 µg per 200 of the Triton solution. These QC samples were analyzed with the samples collected at the same interval to assure the quality of the samples. The overall average recovery was 83.4 percent ± 11% CV for acephate and 89.6 percent ± 20% CV for methamidophos. A storage stability study was also conducted by determining the residues in laboratory fortified samples at different intervals. The results suggested that the residues were stable during the period of sample storage.

Insecticide residues dissipated slowly from Day 0 to Day 7 after the second application. The highest foliar acephate residue (i.e., 0.464 µg/cm$^2$) was measured three days after the second application. Methamidophos residues were relatively low, peaking at 0.007 µg/cm$^2$ on Day 7 after the second application. The reproducibility of replicate DFR samples collected at the same interval was poor. The coefficient of variance ranged from 23.4 percent to 65 percent for acephate and from 6.91 percent to 61.7 percent for methamidophos. There were no rain and only two in-furrow irrigation events from Day 0 to Day 13 after the second application.

The author analyzed DFR data for acephate and methamidophos collected after the second application using the linear regression technique, assuming first-order dissipation kinetics. Calculated DFR half-lives were: (1) acephate - 5.98 days ($R^2$ = 0.7941); and (2) methamidophos - 11.90 days ($R^2$ = 0.7369). Versar re-analyzed the same data set. Versar calculated half-lives were: (1) acephate - 5.67 days ($R^2$ = 0.8773); and (2) methamidophos - 11.90 days ($R^2$ = 0.7249). Therefore, Versar's results differ only slightly from the author's. Versar also analyzed the combined acephate and methamidophos DFRs using the linear regression approach. The half-life for combined residues was estimated to be 5.92 days ($R^2$ = 0.8755).

## STUDY REVIEW

### Study Background

ORTHENE® 75 SP is an organophosphate insecticide used on a wide variety of crops, including: certain vegetables (e.g. head lettuce, dry and succulent beans, celery, cole crops, etc.), cranberries, cotton, mint, peanuts, tobacco, non-bearing citrus, and non-crop areas (e.g. wasteland and rights-of-way). ORTHENE® 75 SP is a water-soluble powder formulation containing 75 percent acephate as the active ingredient (a.i.). The study presents DFR data for acephate and methamidophos residues before and after two spray applications of ORTHENE® 75 SP to cauliflower. The data were submitted in response to a Data Call-in Notice issued by EPA, and are intended to assist in determination of worker re-entry intervals. The usage scenario evaluated in this study was acephate application to a low-growing, waxy foliage crop, in a hot and dry climatic zone

### Test Plot

This DFR study was conducted between July 10, 1998 and August 24, 1998 in Watsonville, Santa Cruz County, California.

The test plot consisted of a treated plot, subdivided into three replicate plots, and a control plot. Control and treated plots were at least 100 feet apart. Cauliflower (var. *White Rock*) was planted on April 21, 1998. The study was initiated at the "pre-heading" stage. The crop was maintained according to normal agricultural practices and irrigated only in the furrows (i.e., no sprinkler irrigation).

During the period of the DFR study, there were four rain events with a total precipitation of 0.04 inches and four in-furrow irrigations. No rain events occurred within 24 hours after the applications of ORTHENE® 75 SP. Rain and irrigation events related to the dates of insecticide applications are summarized below in Table 1.

### Materials and Applications

ORTHENE 75 WSP is formulated as a water soluble powder containing 75% by weight technical grade acephate as the active ingredient (a.i.). The product label was available for review in the study report (Appendix V of the study report).

As directed by the label, two foliar broadcast applications of ORTHENE® 75 SP were made at the maximum rate of 1.0 lb active ingredient per acre. Applications were made using a tractor mounted boom sprayer with nine nozzles positioned about 18 inches above the canopy. The volume of solution applied was approximately 25 gallons per acre (i.e., the minimum application volume).

The two applications were made 10 days apart. There is no minimum application interval; the label states: "repeat application as necessary to maintain control." The pre-harvest interval for cauliflower is 14 days. The crop was certified to have been destroyed on September 21, 1998.

**Table 1. Rain and Irrigation Events**

| Event Date | Event Description |
|---|---|
| July 03, 1998 | Furrow irrigation 1" |
| July 10, 1998 | **Application #1** |
| July 18, 1998 | Furrow irrigation 1.8" |
| July 20, 1998 | **Application #2** |
| July 23, 1998 | Furrow irrigation 2.8" |
| July 26, 1998 | Furrow irrigation 0.8" |
| Aug. 3, 1998 | Rainfall 0.01" |
| Aug. 7, 1998 | Rainfall 0.01" |
| Aug. 12, 1998 | Furrow irrigation 1" |
| Aug. 13, 1998 | Rainfall 0.01" |
| Aug. 23, 1998 | Rainfall 0.01" |

## Sampling/DFR Dislodging

Leaf punch samples were collected 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, three replicate samples were collected from each of the three treated sub-plots and one sample from the control plot. At the intervals when field fortification samples were prepared, six more samples were collected from the control plot.

Each sample consists of forty 2.54 cm diameter leaf punches collected at each interval (note: only from those plant leaves present at the first application). The sample represents 405 $cm^2$ of surface area of both sides of plant leaves. The residues were dislodged from samples 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. The dislodged samples were stored frozen until shipment. All the samples were dislodged within 4.0 hours of collection.

Field-fortified samples were prepared at six intervals. Triplicate leaf punch samples were collected from the control plot at the selected sampling interval, dislodged in the same way as the DFR samples, then each set was fortified with a mixed solution of acephate and methamidophos at two levels. The fortification levels were 2.0, 20, 40, and 400 µg per 200 mL of the Triton solution for acephate and 2.0 and 20 µg per 200 of the Triton solution for methamidophos. Field fortified samples were stored frozen and treated exactly in the same way as the DFR samples collected at the same intervals.

## QA/QC

*Sample Handling & Storage*

All dislodged samples were shipped by overnight delivery service on dry ice to the analytic laboratory. The samples were kept at - 20 °C until analysis.

*Sample History*

In Table 2 (see page 26) of the Study Report, the author provided a sample handling summary. Samples were analyzed from 2 to 36 days after collection.

*Analytical Methodology*

The analytical methodology used was a proprietary Method RM-12HE-2 (see Appendix II of the study). 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.

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

The LOD was 0.125 µg (0.0003 µg /cm$^2$) for acephate and 0.05 µg (0.0001 µg /cm$^2$) for methamidophos. The LOQ for both acephate and methamidophos is 0.0025 µg /cm$^2$.

*Laboratory Recovery*

Laboratory fortification samples were prepared at the following fortification levels: (1) for acephate: 1, 10, 20, 200, and 800 µg per 100 mL of detergent solution; (2) for methamidophos: for acephate and at 1, 10, and 40 µg per 100 mL of detergent solution. Samples were analyzed concurrently with each set of field DFR samples to evaluate the performance of the analytical method. The overall average recovery was 87.0 percent ± 14% CV for acephate and 96.1 percent ± 19% CV for methamidophos. The average laboratory recovery values at each fortification level are summarized below in Table 2. [The individual recovery values are provided in Table 6 of the study report.]

**Table 2. Average Laboratory Fortification Recoveries for Acephate and Methamidophos in Dislodging Solution**

| | Acephate | | | Methamidophos | | |
|---|---|---|---|---|---|---|
| **µg Fortified in 100 mL** | **n** | **Mean Recovery (%)** | **Coefficient of Variation (CV) (%)** | **n** | **Mean Recovery (%)** | **Coefficient of Variation (CV) (%)** |
| 800 | 1 | 85.5 | -- | -- | -- | -- |
| 200 | 1 | 96.3 | -- | -- | -- | -- |
| 20 (acephate) or 40 (methamidophos) | 1 | 86.8 | -- | 1 | 94.6 | -- |
| 10 | 3 | 97.7 | 9.8 | 3 | 93.9 | ± 16 |
| 1.0 | 3 | 73.9 | ± 9.1 | 5 | 97.7 | ± 23 |
| Overall Average | 9 | 87.0 | ± 14 | 9 | 96.1 | ± 19 |

*Field Fortification Recovery*

Field fortification samples for acephate and methamidophos were prepared in triplicate at six sampling intervals. The field fortified samples were analyzed with the DFR samples collected at the same sample interval to assure the quality of the DFR samples. The overall average recovery was 83.4% ± 11% CV for acephate and 89.6% ± 20% CV for methamidophos. The average recoveries for each fortification level are summarized below in Table 3. [Individual recovery values are provided in Table 5 of the study report.]

**Table 3. Average Field Fortification Recoveries for Acephate and Methamidophos in Dislodging Solution**

| | Acephate | | | Methamidophos | | |
|---|---|---|---|---|---|---|
| **µg Fortified in 200 mL** | **n** | **Mean Recovery (%)** | **Coefficient of Variation (CV) (%)** | **n** | **Mean Recovery (%)** | **Coefficient of Variation (CV) (%)** |
| 400 | 4 | 87.5 | ± 5.3 | | | |
| 40 | 1 | 85.5 | -- | | | |
| 20 | 7 | 85.8 | ± 9.9 | 8 | 96.3 | ±19 |
| 2.0 | 7 | 78.4 | ± 14 | 11 | 84.7 | ±21 |
| Overall Average | 19 | 83.4 | ± 11 | 19 | 89.6 | ±20 |

*Storage Stability Recovery*

The stability of acephate and methamidophos during sample storage was studied by periodically analyzing the laboratory fortified samples stored at two conditions--frozen or refrigerated. The results suggest that the residues of acephate and methamidophos were stable for at least 43 days whether frozen or refrigerated. Recoveries were better under refrigeration. The recoveries of acephate and methamidophos at each interval are provided in Table 7 of the study report.

## Results

Acephate and methamidophos DFR data for each sampling interval are summarized below in Table 4. After the second application, insecticide residues dissipated relatively slowly from Day 0 through Day 7. The highest foliar acephate residue (i.e., 0.464 µg/cm$^2$) was found three days after the second application. [Note: There were no rain events until Day 14 after the second application.] Methamidophos residues were much lower, peaking at 0.007 µg/cm$^2$ on Day 7 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 - 5.98 days ($R^2 = 0.794$) and (2) considering methamidophos DFR data from Day 3 (peak) to Day 35, methamidophos - 11.90 days ($R^2 = 0.737$). 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. For acephate, 50 percent dissipation was calculated to occur at 7.17 days ($R^2 = 0.76$); for methamidophos the calculated value was 17.7 days ($R^2 = 0.775$).

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 - 5.67 days ($R^2 = 0.877$) and (2) methamidophos - 11.90 days ($R^2 = 0.725$). (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 5.92 days ($R^2 = 0.876$). "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.

**Data Variability**

Versar examined data variability as part of the linear regression exercise and found that coefficients of variance for replicate samples ranged from 23.4 percent to 65 percent for acephate residues, from 13.4 percent to 61.7 percent for methamidophos residues. There are no specific requirements concerning the variability of replicate samples in the Pesticide Assessment Guidelines.

**Table 4. Dislodgeable Foliar Residues of Acephate and Methamidophos on Cauliflower Leaves after the Two Broadcast Applications of ORTHENE® 75 SP**

| | Acephate Residues on leaves ($\mu g/cm^2$) | | | | Methamidophos Residues on leaves ($\mu g/cm^2$) | | | |
|---|---|---|---|---|---|---|---|---|
| **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 | 0.445 | 0.339 | 0.236 | 0.3400 | 0.007 | 0.0059 | 0.0035 | 0.0055 |
| Pre-Application #2 | 0.056 | 0.033 | 0.045 | 0.0447 | 0.002 | 0.0012 | 0.0012 | 0.0015 |
| Post-Application #2 | 0.188 | 0.159 | 0.254 | 0.2003 | 0.0026 | 0.0023 | 0.0039 | 0.0029 |
| 1 day after App. #2 | 0.128 | 0.238 | 0.395 | 0.2537 | 0.0023 | 0.0039 | 0.0058 | 0.0040 |
| 2 days after | 0.113 | 0.174 | 0.256 | 0.1810 | 0.0023 | 0.0040 | 0.0039 | 0.0034 |
| 3 days after | 0.291 | 0.269 | 0.464 | 0.3413 | 0.0052 | 0.0047 | 0.0064 | 0.0054 |
| 5 days after | 0.0838 | 0.1325 | 0.296 | 0.1708 | 0.002 | 0.0029 | 0.0064 | 0.0038 |
| 7 days after | 0.095 | 0.251 | 0.143 | 0.1630 | 0.005 | 0.007 | 0.005 | 0.0057 |
| 10 days after | 0.0232 | 0.0343 | 0.0643 | 0.0406 | 0.0023 | 0.0028 | 0.003 | 0.0027 |
| 14 days after | 0.0204 | 0.0500 | 0.0833 | 0.0512 | 0.0046 | 0.0050 | 0.006 | 0.0052 |
| 21 days after | 0.0268 | 0.0178 | 0.0268 | 0.0238 | 0.0016 | 0.0016 | 0.0018 | 0.0017 |
| 28 days after | 0.0069 | 0.0169 | 0.0111 | 0.0116 | 0.0009 | 0.002 | 0.0015 | 0.0015 |
| 35 days after | 0.0024 | 0.0038 | 0.0029 | 0.003 | 0.0006 | 0.0007 | 0.0009 | 0.0007 |

**Table 5. Acephate and Methamidophos Half-lives as Estimated by the Registrant and Versar**

| | Acephate | | Methamidophos | | Combined Residues | |
|---|---|---|---|---|---|---|
| | **Half-life (days)** | **Correlation Coefficient ($R^2$)** | **Half-life (days)** | **Correlation Coefficient ($R^2$)** | **Half-life (days)** | **Correlation Coefficient ($R^2$)** |
| Calculated by Registrant | 5.98 | 0.7941 | 11.90 | 0.7369 | -- | -- |
| Calculated by Versar | 5.67 | 0.8773 | 11.89 | 0.7249 | 5.92 | 0.8755 |

**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 partially met. The site represents a dry hot zone where the application of pesticide is a normal agricultural practice. Whether or not reasonable "worst-case" climatic conditions were captured is unknown.

- *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.* These criteria were mostly met. The maximum application rate and minimum application volume were applied, using a label-specified method.

- *Applications occurred at time of season that the end-use product is normally to achieve intended pest control.* This criterion was met. The applications in this study were made at the "pre-heading" growth stage, which is accepted as part of the typical management season.

- *If multiple applications are made, the minimum allowable interval between applications should be used.* This criterion was met. The label does not specify a minimum treatment interval, stating only: "repeat application as necessary to maintain control." Two applications were made 10 days apart. Furthermore, the pre-harvest interval for cauliflower is 14 days. The crop was certified to have been destroyed on September 21, 1998.

- *Meteorological conditions including temperature, wind speed, daily rainfall, and humidity provided for the duration of the study.* This criterion was met. Meteorological conditions during the study are provided in Tables 1 and 3 of the study report.

- *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. Toxicity data were not provided in the study report.

- *Residue storage stability, method efficiency (residue recovery), and limit of quantitation (LOQ) provided.* This criterion was met. Storage stability recovery, laboratory recovery, and field recovery values were provided in the report. The LOQ was 0.0025 µg/cm$^2$ for both acephate and methamidophos.

- *Duplicate foliar and/or soil samples collected at each collection period.* This criterion was met. Triplicate sample replicates were collected at each sampling interval.

- *Control and baseline foliar or soil samples collected.* The criterion was met. Control foliar samples were collected from the control plot at each sampling interval. No soil samples were collected.

- *Sufficient collection times to establish dissipation curve.* This criterion was met. Samples were collected until Day 35 after the second application. By Day 35, residues were below the LOQ.

- *Foliar residue data expressed as µg/cm$^2$ leaf surface area.* This criterion was met. All residue data were provided in µg/cm$^2$.

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

- The reproducibility of replicate samples collected at the same time interval was poor. The coefficient of variance for replicate samples ranged from 23.4 percent to 65 percent for acephate and from 13.4 percent to 61.7 percent for methamidophos.

- The highest foliar acephate residue of 0.464 µg/cm$^2$ occurred at Day 3 after the second application. The Day 0 average DFR value was 0.2 µg/cm$^2$, which is significantly lower. Yet, the acephate calculated half-life was calculated from Day 0. The sampling procedure may have been flawed, in that it did not yield truly representative samples, or there may have been a climatic influence (e.g., dryness). Rainfall was extremely light during the study; the first rainfall (only 0.01 inches) after the second application was recorded 14 days afterwards. Two in-furrow irrigation events occurred on Day 3 and Day 6 after the second application.

- It is unclear whether the registrants corrected raw DFR data for either laboratory or field recovery losses before running their regression analysis. Average values for both laboratory and fortified field recoveries were often below 90%.

- OPPTS 875.2100 (an Update to Subdivision K) specifically requires that DFR data be collected from at least three geographically distinct locations for each formulation. In this study, DFR samples were collected only from one location.

# Appendix A

## Versar's Regression Analysis for DFR Acephate Data

**Regression Analysis: Summary Output for acephate**

| *Regression Statistics* | |
|---|---|
| Multiple R | 0.938699 |
| R Square | 0.881155 |
| Adjusted $R^2$ | 0.877322 |
| Standard Error | 0.520585 |
| Observations | 33 |

ANOVA

| | *df* | *SS* | *MS* | *F* | *Signif. F* |
|---|---|---|---|---|---|
| Regression | 1 | 62.28991 | 62.28991 | 229.84493 | 6.9287E-16 |
| Residual | 31 | 8.401261 | 0.271008 | | |
| Total | 32 | 70.69117 | | | |

| | *Coeff.* | *Std. Error* | *t Stat* | *P-value* | *Lower 95%* | *Upper 95%* |
|---|---|---|---|---|---|---|
| Intercept | -1.352916 | 0.129364 | -10.45821 | 1.088E-11 | -1.61675541 | -1.089075984 |
| Slope | -0.122188 | 0.00806 | -15.16064 | 6.929E-16 | -0.138625158 | -0.105750096 |

Half Life = 5.67281 Days

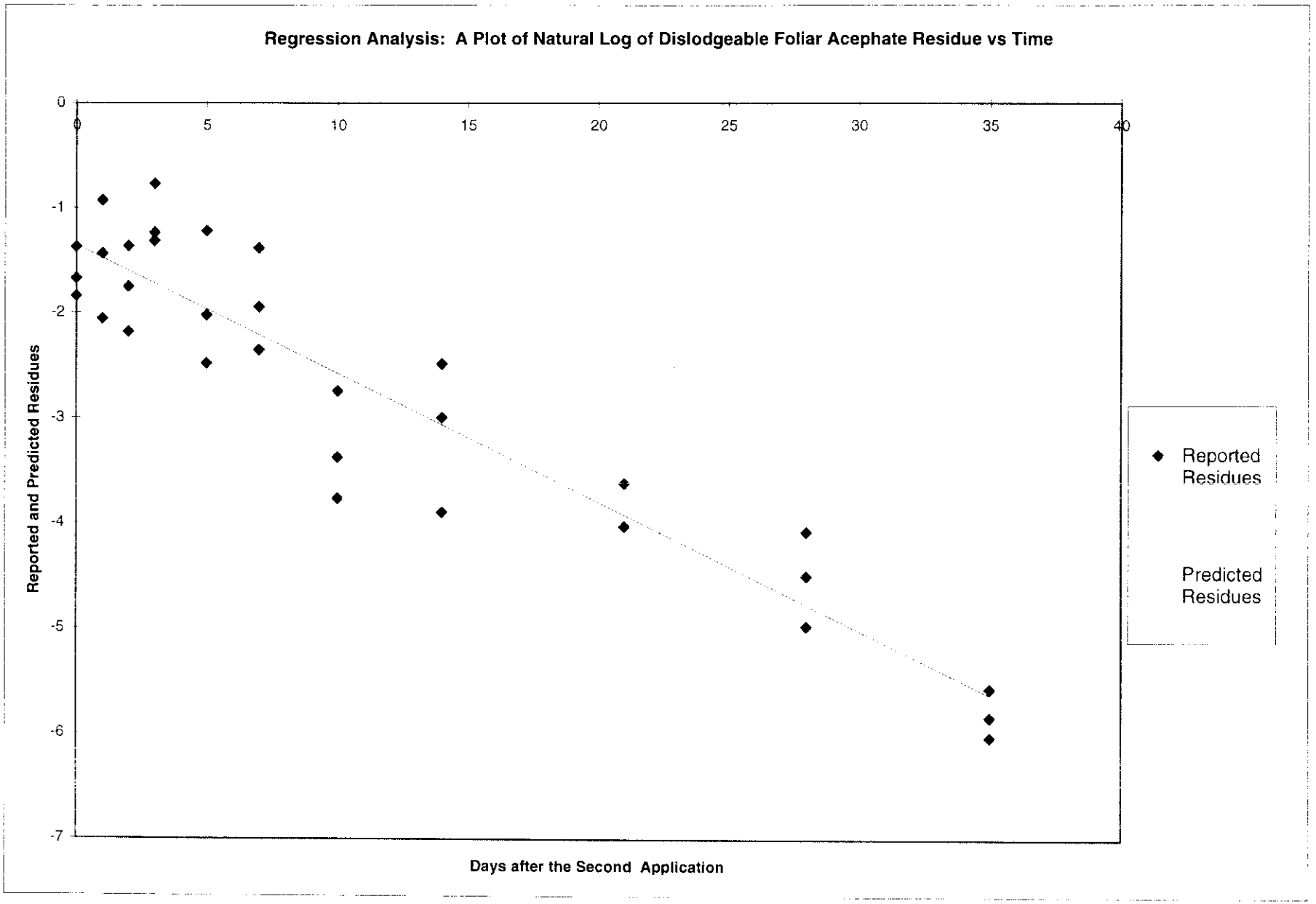
**Predicted DFR Levels**

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 0 | 0.258485 |
| 1 | 0.228755 |
| 2 | 0.202444 |
| 3 | 0.17916 |
| 4 | 0.158553 |
| 5 | 0.140317 |
| 6 | 0.124178 |
| 7 | 0.109895 |
| 8 | 0.097255 |
| 9 | 0.086069 |
| 10 | 0.07617 |
| 11 | 0.067409 |
| 12 | 0.059656 |
| 13 | 0.052794 |
| 14 | 0.046722 |
| 15 | 0.041348 |
| 16 | 0.036592 |
| 17 | 0.032384 |
| 18 | 0.028659 |
| 19 | 0.025363 |
| 20 | 0.022445 |

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 21 | 0.0198638 |
| 22 | 0.0175791 |
| 23 | 0.0155572 |
| 24 | 0.0137679 |
| 25 | 0.0121843 |
| 26 | 0.0107829 |
| 27 | 0.0095427 |
| 28 | 0.0084451 |
| 29 | 0.0074738 |
| 30 | 0.0066141 |
| 31 | 0.0058534 |
| 32 | 0.0051802 |
| 33 | 0.0045843 |
| 34 | 0.0040571 |
| 35 | 0.0035904 |

**Regression Analysis: Means and CVs for acephate**

| Days after Last Treatment | Residues (ug/cm2) | Mean (ug/cm2) | Standard Deviation (ug/cm2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 0 | 0.188 | 0.2 | 0.0487 | 24.3 |
| | 0.159 | | | |
| | 0.254 | | | |
| 1 | 0.128 | 0.254 | 0.134 | 52.8 |
| | 0.238 | | | |
| | 0.395 | | | |
| 2 | 0.113 | 0.181 | 0.0718 | 39.6 |
| | 0.174 | | | |
| | 0.256 | | | |
| 3 | 0.291 | 0.341 | 0.107 | 31.3 |
| | 0.269 | | | |
| | 0.464 | | | |
| 5 | 0.0838 | 0.171 | 0.111 | 65 |
| | 0.1325 | | | |
| | 0.296 | | | |
| 7 | 0.095 | 0.163 | 0.0799 | 49 |
| | 0.251 | | | |
| | 0.143 | | | |
| 10 | 0.0232 | 0.0406 | 0.0213 | 52.4 |
| | 0.0343 | | | |
| | 0.0643 | | | |
| 14 | 0.0204 | 0.0512 | 0.0315 | 61.5 |
| | 0.05 | | | |
| | 0.0833 | | | |
| 21 | 0.0268 | 0.0238 | 0.0052 | 21.8 |
| | 0.0178 | | | |
| | 0.0268 | | | |
| 28 | 0.0069 | 0.0116 | 0.00502 | 43.3 |
| | 0.0169 | | | |
| | 0.0111 | | | |
| 35 | 0.0024 | 0.00303 | 0.000709 | 23.4 |
| | 0.0038 | | | |
| | 0.0029 | | | |


Regression Analysis: A Plot of Natural Log of Dislodgeable Foliar Acephate Residue vs Time
Reported and Predicted Residues
0
-1
-2
-3
-4
-5
-6
-7
0
5
10
15
20
25
30
35
40
Days after the Second Application
Reported Residues
Predicted Residues

# Appendix B

## Versar's Regression Analysis for DFR Methamidophos Data

**Regression Analysis: Summary Output for methamidophos**

| *Regression Statistics* | |
|---|---|
| Multiple R | 0.858436 |
| R Square | 0.736912 |
| Adjusted $R^2$ | 0.724954 |
| Standard Error | 0.393684 |
| Observations | 24 |

ANOVA

| | *df* | *SS* | *MS* | *F* | *Signif. F* |
|---|---|---|---|---|---|
| Regression | 1 | 9.550659 | 9.550659 | 61.622315 | 8.0712E-08 |
| Residual | 22 | 3.409715 | 0.154987 | | |
| Total | 23 | 12.96037 | | | |

| | *Coeff.* | *Std. Error* | *t Stat* | *P-value* | *Lower 95%* | *Upper 95%* |
|---|---|---|---|---|---|---|
| Intercept | -5.042479 | 0.139568 | -36.12918 | 4.396E-21 | -5.331925859 | -4.753032346 |
| Slope | -0.058262 | 0.007422 | -7.849988 | 8.071E-08 | -0.073653833 | -0.04286968 |

Half Life = 11.89712 Days

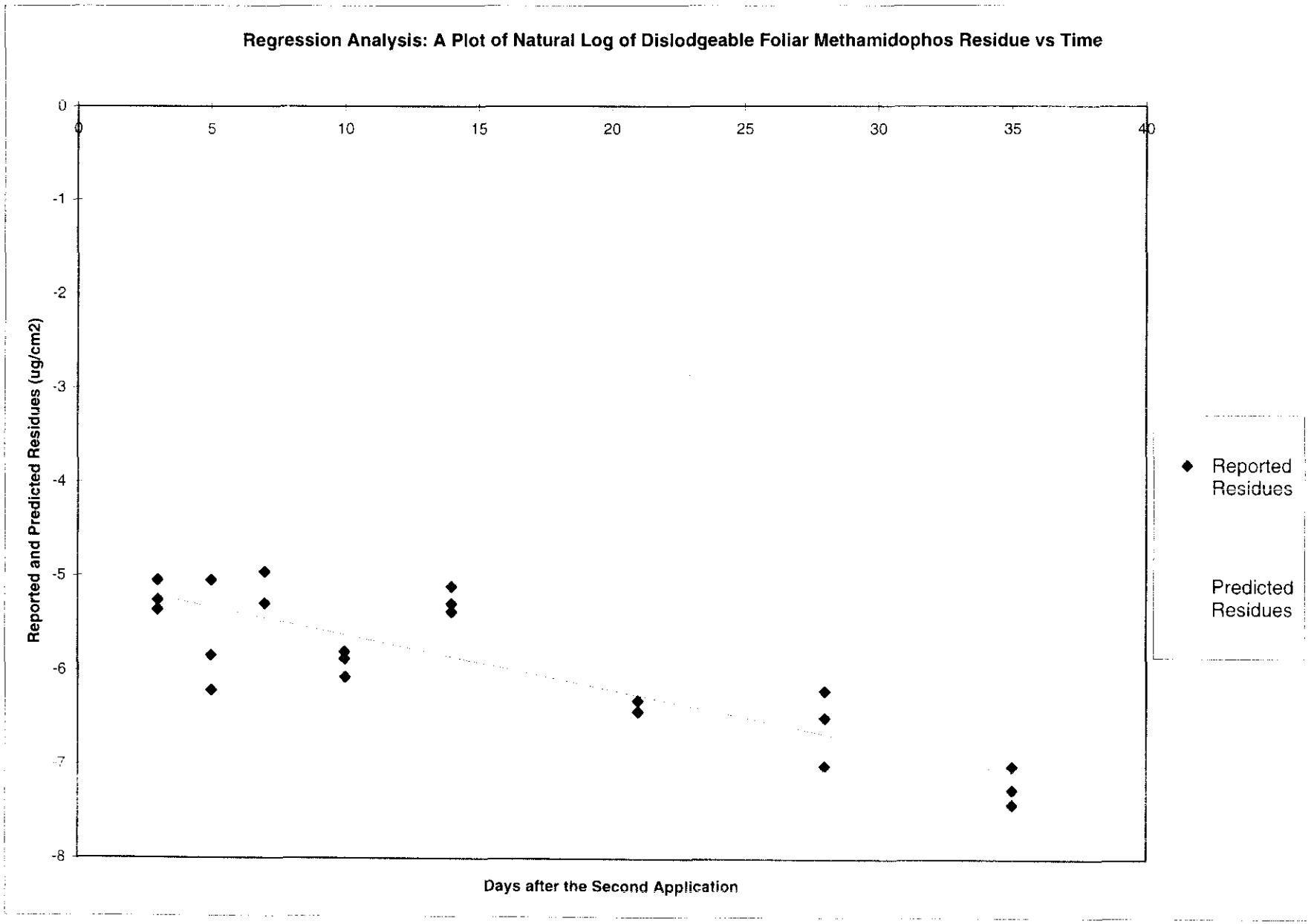
**Predicted DFR Levels**

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 0 | 0.006458 |
| 1 | 0.006092 |
| 2 | 0.005747 |
| 3 | 0.005422 |
| 4 | 0.005115 |
| 5 | 0.004826 |
| 6 | 0.004553 |
| 7 | 0.004295 |
| 8 | 0.004052 |
| 9 | 0.003823 |
| 10 | 0.003606 |
| 11 | 0.003402 |
| 12 | 0.00321 |
| 13 | 0.003028 |
| 14 | 0.002857 |
| 15 | 0.002695 |
| 16 | 0.002542 |
| 17 | 0.002398 |
| 18 | 0.002263 |
| 19 | 0.002135 |
| 20 | 0.002014 |

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 21 | 0.0018999 |
| 22 | 0.0017923 |
| 23 | 0.0016909 |
| 24 | 0.0015952 |
| 25 | 0.0015049 |
| 26 | 0.0014197 |
| 27 | 0.0013394 |
| 28 | 0.0012636 |
| 29 | 0.0011921 |
| 30 | 0.0011246 |
| 31 | 0.0010609 |
| 32 | 0.0010009 |
| 33 | 0.0009443 |
| 34 | 0.0008908 |
| 35 | 0.0008404 |

**Regression Analysis: Means and CVs for methamidophos**

| Days after Last Treatment | Residues (ug/cm2) | Mean (ug/cm2) | Standard Deviation (ug/cm2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 3 | 0.0052 | 0.00543 | 0.000874 | 16.1 |
| | 0.0047 | | | |
| | 0.0064 | | | |
| 5 | 0.002 | 0.00377 | 0.00232 | 61.7 |
| | 0.0029 | | | |
| | 0.0064 | | | |
| 7 | 0.005 | 0.00567 | 0.00115 | 20.4 |
| | 0.007 | | | |
| | 0.005 | | | |
| 10 | 0.0023 | 0.0027 | 0.000361 | 13.4 |
| | 0.0028 | | | |
| | 0.003 | | | |
| 14 | 0.0046 | 0.0052 | 0.000721 | 13.9 |
| | 0.005 | | | |
| | 0.006 | | | |
| 21 | 0.0016 | 0.00167 | 0.000115 | 6.91 |
| | 0.0016 | | | |
| | 0.0018 | | | |
| 28 | 0.0009 | 0.00147 | 0.000551 | 37.5 |
| | 0.002 | | | |
| | 0.0015 | | | |
| 35 | 0.0006 | 0.000733 | 0.000153 | 20.8 |
| | 0.0007 | | | |
| | 0.0009 | | | |


Regression Analysis: A Plot of Natural Log of Dislodgeable Foliar Methamidophos Residue vs Time
Reported and Predicted Residues (ug/cm2)
0
-1
-2
-3
-4
-5
-6
-7
-8
0
5
10
15
20
25
30
35
40
Days after the Second Application
Reported Residues
Predicted Residues

## Appendix C

## Versar's Regression Analysis for Combined residues of Acephate and Methamidophos

**Regression Analysis: Summary Output for Combined Residues**

| *Regression Statistics* | |
|---|---|
| Multiple R | 0.937763 |
| R Square | 0.879399 |
| Adjusted $R^2$ | 0.875509 |
| Standard Error | 0.503017 |
| Observations | 33 |

ANOVA

| | *df* | *SS* | *MS* | *F* | *Signif. F* |
|---|---|---|---|---|---|
| Regression | 1 | 57.19559 | 57.19559 | 226.04582 | 8.70602E-16 |
| Residual | 31 | 7.843822 | 0.253027 | | |
| Total | 32 | 65.03941 | | | |

| | *Coeff.* | *Std. Error* | *t Stat* | *P-value* | *Lower 95%* | *Upper 95%* |
|---|---|---|---|---|---|---|
| Intercept | -1.34476 | 0.124999 | -10.75821 | 5.438E-12 | -1.599696806 | -1.089824054 |
| Slope | -0.117085 | 0.007788 | -15.03482 | 8.706E-16 | -0.13296741 | -0.101201725 |

Half Life = 5.920056 Days

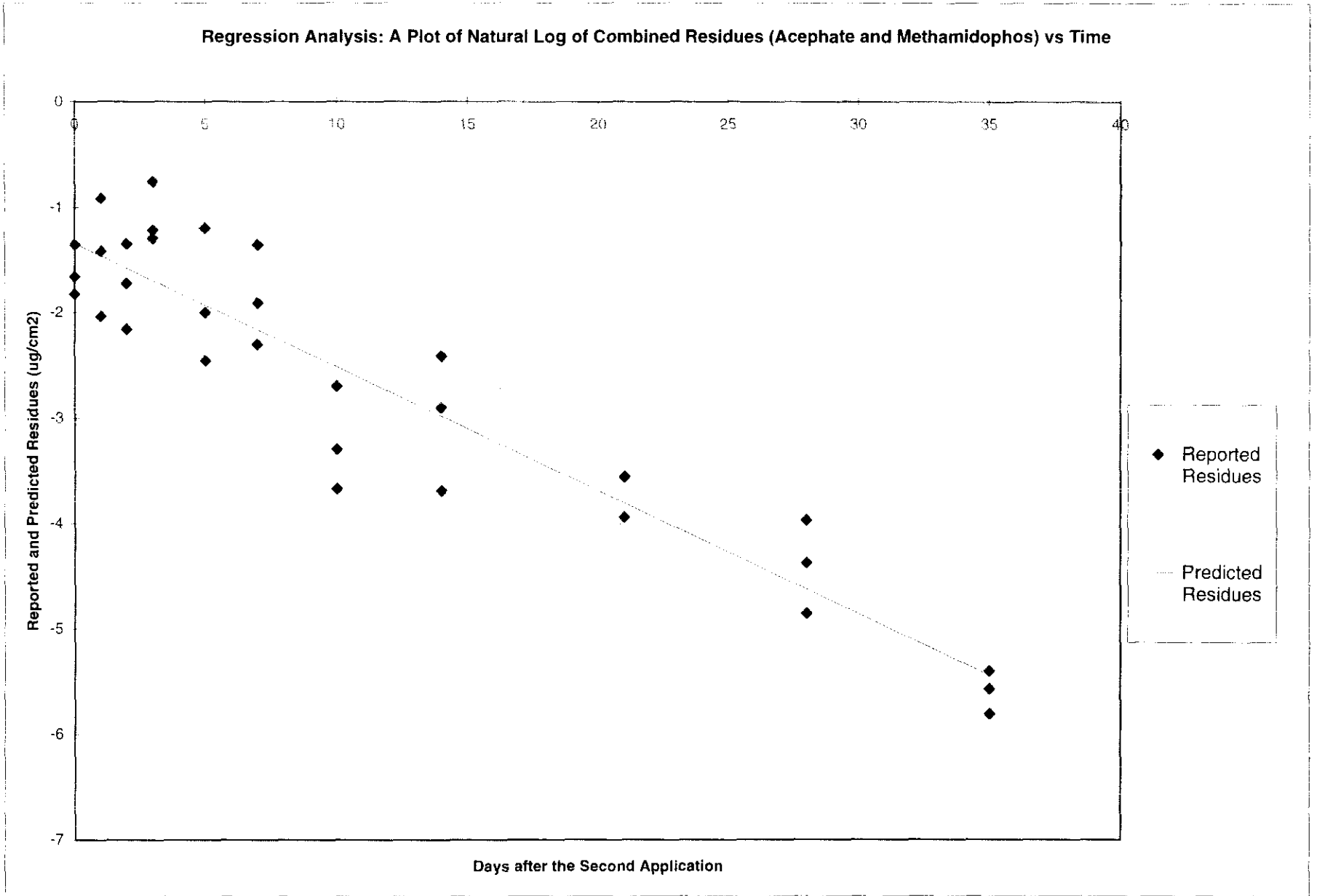
**Predicted DFR Levels**

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 0 | 0.260602 |
| 1 | 0.231808 |
| 2 | 0.206196 |
| 3 | 0.183413 |
| 4 | 0.163148 |
| 5 | 0.145122 |
| 6 | 0.129087 |
| 7 | 0.114824 |
| 8 | 0.102137 |
| 9 | 0.090852 |
| 10 | 0.080814 |
| 11 | 0.071885 |
| 12 | 0.063942 |
| 13 | 0.056877 |
| 14 | 0.050593 |
| 15 | 0.045003 |
| 16 | 0.040031 |
| 17 | 0.035608 |
| 18 | 0.031673 |
| 19 | 0.028174 |
| 20 | 0.025061 |

| Time (Days) | Residue (ug/cm2) |
|---|---|
| 21 | 0.0222918 |
| 22 | 0.0198288 |
| 23 | 0.0176379 |
| 24 | 0.0156891 |
| 25 | 0.0139556 |
| 26 | 0.0124136 |
| 27 | 0.0110421 |
| 28 | 0.009822 |
| 29 | 0.0087368 |
| 30 | 0.0077715 |
| 31 | 0.0069128 |
| 32 | 0.006149 |
| 33 | 0.0054696 |
| 34 | 0.0048653 |
| 35 | 0.0043277 |

**Regression Analysis: Means and CVs for Combined Residue**

| Days after Last Treatment | Residues (ug/cm2) | Mean (ug/cm2) | Standard Deviation (ug/cm2) | Coefficient of Variation (%) |
|---|---|---|---|---|
| 0 | 0.1906 | 0.203 | 0.0495 | 24.4 |
| | 0.1613 | | | |
| | 0.2579 | | | |
| 1 | 0.1303 | 0.258 | 0.136 | 52.7 |
| | 0.2419 | | | |
| | 0.4008 | | | |
| 2 | 0.1153 | 0.184 | 0.0725 | 39.4 |
| | 0.178 | | | |
| | 0.2599 | | | |
| 3 | 0.2962 | 0.347 | 0.108 | 31 |
| | 0.2737 | | | |
| | 0.4704 | | | |
| 5 | 0.0858 | 0.175 | 0.113 | 64.8 |
| | 0.1354 | | | |
| | 0.3024 | | | |
| 7 | 0.1 | 0.169 | 0.081 | 47.9 |
| | 0.258 | | | |
| | 0.148 | | | |
| 10 | 0.0255 | 0.0433 | 0.0216 | 49.8 |
| | 0.0371 | | | |
| | 0.0673 | | | |
| 14 | 0.025 | 0.0564 | 0.0322 | 57 |
| | 0.055 | | | |
| | 0.0893 | | | |
| 21 | 0.0284 | 0.0255 | 0.00525 | 20.6 |
| | 0.0194 | | | |
| | 0.0286 | | | |
| 28 | 0.0078 | 0.0131 | 0.00557 | 42.5 |
| | 0.0189 | | | |
| | 0.0126 | | | |
| 35 | 0.003 | 0.00377 | 0.000751 | 19.9 |
| | 0.0045 | | | |
| | 0.0038 | | | |


Regression Analysis: A Plot of Natural Log of Combined Residues (Acephate and Methamidophos) vs Time
Reported and Predicted Residues (ug/cm2)
0
-1
-2
-3
-4
-5
-6
-7
0
5
10
15
20
25
30
35
40
Days after the Second Application
Reported Residues
Predicted Residues

13544

# R132858

Chemical: Acephate

PC Code:
103301

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

Memo Date: 4/27/1999

File ID: 00000000

Accession #: 412-07-0024

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11/9/2006