

California Environmental Protection Agency



**Ambient Air Monitoring for Acephate and Methamidophos
In Fresno County – Summer 2002**

Prepared by
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Quality Management Branch
Monitoring and Laboratory Division

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Executive Summary

Ambient Air Monitoring for Acephate and Methamidophos In Fresno County - Summer 2002

In January 2002 the California Department of Pesticide Regulation (DPR) requested that the Air Resources Board (ARB) conduct ambient air monitoring for the pesticides acephate and methamidophos in Fresno County during the summer of 2002. Monitoring was conducted in Fresno County from July 8 through August 23, 2002, to coincide with the use of acephate and methamidophos as insecticides. California growers primarily use acephate and methamidophos to control a variety of plant and soil insects. According to the DPR monitoring recommendation, acephate converts to methamidophos in the environment.

Five sampling sites were selected in relatively high-population areas or in areas frequented by people (e.g., schools or school district offices, fire stations, or other public buildings). Background samples were collected at the ARB's regular air monitoring site in Fresno. At each site, 28 discrete 24-hour samples were collected, Monday through Friday (4 samples/week), during the 7-week sampling period. Collocated (replicate) samples were collected for seven dates (each Wednesday) at each sampling location.

The sites were selected by ARB personnel from areas in Fresno County where acephate and methamidophos was used in the past. Sites were selected for their proximity to the prior use areas with considerations for both accessibility and security of the sampling equipment. The ARB understands that DPR staff will verify and quantify the actual use of acephate and methamidophos that took place during the study when the information becomes available.

Acephate Results

Of the 168 ambient samples collected, one contained a concentration of acephate above the reported estimated quantitation limit (EQL) of 10 ng/m^3 . This concentration of 15 nanograms per cubic meter of sampled air (ng/m^3) (2.0 parts per trillion by volume (pptv)) was measured at the Tranquility High School (THS) site. Four samples were found to have results of "detected," and 163 were below the method detection limit (MDL).

Seven-week average concentrations ranged from 1.1 ng/m^3 (0.15 pptv) to 1.6 ng/m^3 (0.21 pptv). The highest average was measured at the THS site.

Methamidophos Results

Of the 168 ambient samples collected, 10 contained concentrations of methamidophos above the reported EQL of 3.5 ng/m^3 , 7 were found to have results of "detected," and 151 were below the MDL.

Daily concentrations of methamidophos ranged from <MDL to 16 ng/m^3 (2.8 pptv). The

highest concentration was measured at the San Joaquin Elementary School (SJS) site.

Seven-week average concentrations ranged from 0.55 ng/m³ (0.095 pptv) to 1.4 ng/m³ (0.24 pptv). The highest average was measured at the SJS site.

Acknowledgments

Assistance in sampling site selection was provided by Mr. Cliff Francone and Mr. Chuck Francone of the Fresno County Agricultural Commissioner's Office. Staff of the Air Resources Board (ARB) Air Quality Surveillance Branch (AQSB) collected the ambient samples. Mr. Steve Rider of the AQSB coordinated the fieldwork. Ms. Theresa Houston, Mr. Mike Orbanosky, and Mr. Jim Omand of the ARB Special Analysis Section laboratory performed the method development and chemical analyses. Ms. Yun Pan-Huang of the Operations Planning & Assessment Section prepared the monitoring report. Mr. Lynn Baker of the ARB Stationary Source Division provided comments on the monitoring protocol and report.

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Ambient Air Monitoring for Acephate and Methamidophos
In Fresno County - Summer 2002

I. Introduction

At the request (January 2, 2002, Memorandum, Helliker to Lloyd) of the California Department of Pesticide Regulation (DPR), the Air Resources Board (ARB) staff determined airborne concentrations of the pesticides acephate and methamidophos in Fresno County over a seven week ambient monitoring program. This monitoring was done to fulfill the requirements of Assembly Bill 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions ... of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. Monitoring was conducted in Fresno County from July 8 through August 23, 2002, to coincide with the use of acephate and methamidophos as insecticides. California growers use acephate and methamidophos to control a variety of plant and soil insects. According to the DPR monitoring recommendation, acephate converts to methamidophos in the environment. The application site monitoring conducted during summer 2002 for methamidophos will be described in a separate report.

The ARB Special Analysis Section of the Northern Laboratory Branch conducted the method development and sample analyses. The ARB Air Quality Surveillance Branch staff conducted sample collections for the ambient study.

The protocol for the ambient air monitoring for acephate and methamidophos is enclosed separately as Appendix I (page 1 of the Appendices to this report). The protocol Attachments I, II, and V have not been included in Appendix I, but are available upon request. The laboratory report, "Air Sampling Cartridge Method Development and Analytical Results of Ambient Monitoring in Fresno County for Methamidophos and Acephate" is enclosed separately as Appendix II (pages 19 of the Appendices to this report). The Standard Operating Procedure (SOP) for acephate and methamidophos are also enclosed as Appendix II (page 35 of the Appendices to this report). The field data sheets for acephate and methamidophos are enclosed separately as Appendix III (page 41 of the Appendices to this report).

II. Sampling

A. Sampling Method

Air samples were collected by passing a measured volume of ambient air through XAD-2 resin. The exposed XAD-2 resin tubes (SKC #226-30-06) were stored in an ice chest (on dry ice) or in a freezer until desorbed with 10% of acetone in ethyl acetate. The tubes are 8 mm x 110 mm with 400 mg XAD-2 in the primary section and 200 mg in the secondary section. The flow rate of 3.0 standard liters per minute (slpm) was accurately measured and the sampling system operated continuously for 24 hours with the exact operating interval recorded in the logbook. The tubes were protected from direct sunlight and positioned 1.5 meters above roof tops for the ambient monitoring. At

the end of each sampling period, the tubes were placed in culture tubes with an identification label affixed. Subsequent to sampling, the sample tubes were transported on dry ice, as quickly as possible, to the ARB Monitoring and Laboratory Division laboratory for analysis. The samples were stored at or below 4 °C or extracted/analyzed immediately.

Each sample train consisted of an adsorbent tube, Teflon fittings and tubing, rain/sun shield, rotameter (or needle valve), train support, and either a 12-volt DC or a 115-volt AC vacuum pump. Tubes were prepared for use by breaking off the sealed glass ends and immediately inserting the tube into the Teflon fitting. The tubes were oriented in the sample train according to a small arrow printed on the side indicating the direction of flow. A needle valve with a range of 0-5 slpm was used to control sample flow rate. The flow rates were set using a calibrated digital mass flow meter (MFM), scaled from 0-5 slpm, before the start of each sampling period. The flow rate was also checked and recorded, using the MFM, at the end of each sampling period. Samplers were leak checked prior to each sampling period, with the sampling tubes installed. Any change in flow rates was recorded on the field log sheet. The pesticide sampling procedures for adsorbent tubes are included in Appendix I (page 15 of the Appendices to this report).

The ambient monitoring study included 168 individual sampling periods (6 sites x 28 sampling days). Collocated (duplicate) samples were collected for one day/week (each Wednesday) at each sampling location. Trip blanks were submitted once per week.

B. Sampling Site Selection

The DPR recommendations for acephate and methamidophos requested that ambient monitoring occur in Fresno County for 7 week consecutive weeks between July 8 and August 23, 2002. Monitoring was conducted in Fresno County from July 8 through August 23, 2002. Five sampling sites were selected by ARB personnel in populated areas or in areas frequented by people. Site selection was based upon considerations for accessibility, security of the sampling equipment, and compliance with technical siting requirements. Urban background samples were collected at the ARB's Fresno Ambient Air Monitoring Station. The six sites are listed in Table 1. Although the sampling sites are near areas of prior use of acephate and methamidophos it is understood that DPR staff will verify and quantify the actual use of acephate and methamidophos that occurred during the study when the information becomes available.

Table 1
Ambient Sampling Sites

| | | |
|-----|--|----------------|
| FRS | ARB Fresno - First Air Monitoring Station | (559) 228-1825 |
| | 3425 North First Street | Pat Seames, |
| | Fresno CA 93726 | Site Operator |
| | Section/Township/Range: SE.22/T.13S/R.20E | |
| | GPS Coordinates: N. 36° 46.906' W. 119°46.328' | |

- HES Helm Elementary School (559) 866-5683
 13883 South Lassen Avenue Sylvia Grider
 Helm, CA 93627 Principal
 Section/Township/Range: SE.15/T.16S/R.17E
 GPS Coordinates: N. 36° 31.977' W. 120°05.903'
- SJS San Joaquin Elementary School (559) 693-4321
 West Nevada Avenue John Crider
 San Joaquin, CA 93660 Principal
 Section/Township/Range: SW.24/T.15S/R.16E
 GPS Coordinates: N. 36° 36.580' W. 120°11.201
- THS Tranquility High School (559) 698-7205
 6052 Juanche Avenue Jo Ann R. Minnite
 Tranquility, CA 93668 School Secretary
 Section/Township/Range: NW.8/T.15S/R.16E
 GPS Coordinates: N. 36° 38.735' W. 120°15.194
- CES Cantua Creek Elementary School (559) 829-3331
 19288 West Clarkson Avenue Rubin V. Castillo
 Cantua Creek, CA 93608 Principal
 Section/Township/Range: SE.27/T.16S/R.15E
 GPS Coordinates: N. 36° 30.0935' W. 120°19.192'
- WRS West Side Research and Extension (559) 884-2412
 17353 W. Oakland Avenue Jimmie H. Ross
 Five Points, CA 93624 Superintendent
 Section/Township/Range: NE.27/T.18S/R.19E
 GPS Coordinates: N. 36° 20.494' W. 120°06.515'

FRS

The urban background site was located at ARB's ambient air monitoring station in the city of Fresno. This station monitors concentrations and collects samples of most criteria gas and particulate pollutants as well as meteorological data. The site is located relatively close to the center of the metropolitan Fresno city limits in a mix of business offices, parks, and residences. Fresno has a population of approximately 450,000. The pesticide samplers were operated on top of the two-story building housing ARB's air monitoring station. The sample inlets were 34.5 ±0.5 feet above ground level. The site met all technical siting requirements. Elevation of the site is 350 ±10 feet above mean sea level (MSL). No agricultural fields were noted within a 3-mile radius.

HES

The Helm Elementary School was located in a rural agricultural/residential mixed area in the town of Helm, which has a population of approximately 200. The pesticide samplers were

operated on the roof of one of the school buildings and their inlets were 18 ± 0.5 feet above ground level. The site met all technical siting requirements. Elevation of the site is 200 ± 10 feet above MSL. Cotton and bean fields were located to the northeast and north within a 1.5-mile radius.

SJS

The San Joaquin Elementary School site was located in a rural, residential/agricultural mixed area near the center of the town of San Joaquin, which has a population of approximately 3,300. The pesticide samplers were operated on the roof of a shipping container and their inlets were 9.5 ± 0.5 feet above ground level. The site met all technical siting requirements. Elevation of the site is 170 ± 10 feet above MSL. Cotton fields surround this site within a 2-mile radius.

THS

The Tranquility High School site was located in a rural, residential/agricultural mixed area in the town of Tranquility, which has a population of approximately 200. The pesticide samplers were operated on the roof of the south wing of the gymnasium and their inlets were 10.5 ± 0.5 feet above ground level. The site met all technical siting requirements as the roof peak was more than twice the height away. Elevation of the site is 165 ± 10 feet above MSL. Cotton fields surround this site within a 2-mile radius.

CES

The Cantua Creek Elementary School site was located in a rural, agricultural/residential mixed area in the town of Cantua Creek, which has a population of approximately 300. The pesticide samplers were operated on the roof of one of the school buildings and their inlets were 17 ± 0.5 feet above ground level. The site met all technical siting requirements. Elevation of the site is 310 ± 10 feet above MSL. Multiple cotton and bean fields were located within a 1.5-mile radius.

WRS

The West Side Research & Extension site was located in a rural, agricultural/residential mixed area at the west end of the community of Calflax, which has a population of approximately 50. The pesticide samplers were operated on the roof of one of the complex's buildings, which at one time was a PM10 site, and their inlets were 16.5 ± 0.5 feet above ground level. The site met all technical siting requirements. Elevation of the site is 325 ± 10 feet above MSL. Multiple cotton fields surround this site within a 1.5-mile radius.

III. Analytical Methodology

The standard operating procedures for sampling and analysis of acephate and methamidophos are enclosed in Appendix II.

Per 40 CFR, Part 136, Appendix B, the method detection limit (MDL) was determined by analysis of 7 replicate cartridge spikes (near the estimated detection limit) for acephate and methamidophos. The $MDL = (3.14) \times \text{standard deviation}$, calculated from the 7 replicate results. The analytical $EQL = (5) \times MDL$. Based on a 3-ml extraction volume and a sample collected for 24 hours at a flow rate of 3.0 slpm, the

MDLs achieved by the laboratory were 2.2 ng/m³ and 0.86 ng/m³ for acephate and methamidophos, respectively. This corresponds to analytical EQLs of 11 ng/m³ for acephate and 4.3 ng/m³ for methamidophos. The DPR requested target 24-hour EQLs of 5.0 ng/m³ and 1.0 ng/m³ for acephate and methamidophos, respectively. However, the reported EQLs in the lab report were based on the lowest calibration standards of 45 ng/sample (10 ng/m³) and 15 ng/sample (3.5 ng/m³) for acephate and methamidophos, respectively.

The sampling and analytical method used for this study specifies that the ambient air is collected on XAD-2 cartridges for 24 hours at 3.0 slpm flow rate. The samples are stored in an ice chest on dry ice or in a refrigerator until extracted with 3 milliliters (ml) of 10% acetone in ethyl acetate. Sample extracts are analyzed on a gas chromatograph (GC) with a flame photometric detector (FPD) using internal standard method.

IV. Monitoring Results

All samples were extracted and analyzed within 21 days of collection, within the period samples were shown to be stable. Samples were stored at or below 4°C before extraction. All samples were analyzed the day of extraction and not kept in extract storage longer than a few hours.

For acephate and methamidophos, results below the MDL are reported as <MDL, results equal to or above the MDL, but below the reported EQL, are reported as "detected" (DET). Laboratory results equal to or above the reported EQL are reported to 3 significant figures in units of ng/sample, final concentrations in sampled air are reported to 2 significant figures. No sample results have been adjusted or corrected for recoveries of quality assurance spike samples.

Acephate Results

Table 2 presents the results of ambient air monitoring for acephate in units of ng/m³ and pptv. A summary of the ambient results for acephate is presented in Table 4.

The equation used to convert acephate air concentration results from units of ng/m³ to units of pptv at 1 atmosphere and 25°C is shown below:

$$\text{pptv} = (\text{ng/m}^3) \times \frac{(0.0820575 \text{ liter-atm/mole-}^\circ\text{K})(298^\circ\text{K})}{(1 \text{ atm})(183.16 \text{ gram/mole})} = (0.1335) \times (\text{ng/m}^3)$$

Of the 168 ambient samples collected (spikes, blanks, and the lower value of each collocated pair excluded), one contained a concentration of acephate above the reported EQL of 10 ng/m³. This concentration was 15 ng/m³ (2.0 pptv), measured at the THS site on August 20, 2002. Four samples were found to have results of detected, and 163 were below the MDL.

Seven-week average concentrations ranged from 1.1 ng/m³ (0.15 pptv) to 1.6 ng/m³ (0.21 pptv). The highest average was measured at the THS site.

As discussed in Section VI, Part C of this report, an increase in response is observed for acephate in the solvent extracts with time (i.e., "Re-analysis of an extract within 24 hours results in acephate values up to 2 times higher than the original value"). The laboratory report did not provide information regarding the time elapsed between extraction and analysis for the samples. The impact of this problem on the results is difficult to quantitatively assess. However, the results reported may be considered as maximum possible concentrations.

Methamidophos Results

Table 3 presents the results of ambient air monitoring for methamidophos in units of ng/m³ and pptv. A summary of the ambient results for methamidophos is presented in Table 5.

The equation used to convert methamidophos air concentration results from units of ng/m³ to units of pptv at 1 atmosphere and 25°C is shown below:

$$\text{pptv} = (\text{ng/m}^3) \times \frac{(0.0820575 \text{ liter-atm/mole-}^\circ\text{K})(298^\circ\text{K})}{(1 \text{ atm})(141.13 \text{ gram/mole})} = (0.1733) \times (\text{ng/m}^3)$$

Of the 168 ambient samples collected (spikes, blanks, and the lower value of each collocated pair excluded), 10 contained concentrations of methamidophos above the reported EQL of 3.5 ng/m³, 7 were found to have results of detected, and 151 were below the MDL.

Daily concentrations of methamidophos ranged from <MDL to 16 ng/m³ (2.8 pptv). The highest concentration was measured at the SJS site on July 31, 2002.

Seven-week average concentrations ranged from 0.55 ng/m³ (0.095 pptv) to 1.4 ng/m³ (0.24 pptv). The highest average was measured at the SJS site.

V. Quality Control (QC)

Field QC for the ambient monitoring included the following:

- 1) Seven field spikes (same environmental and experimental conditions as those occurring at the time of ambient sampling) prepared by the Special Analysis Section staff. The field spikes were obtained by sampling ambient air at the background monitoring site for 24-hour periods (collocated with an ambient sample);
- 2) Seven trip spikes;
- 3) Collocated (duplicate) samples taken once per week at each sampling location; and
- 4) 1 trip blank submitted per week;
- 5) The battery operated mass flow meters used to set and check the sampling flow rate were calibrated by the ARB's Program Evaluation and Standards Section.
- 6) A flow audit of each sampler was performed by the Quality Assurance

Section (QAS) on August 30, 2002, at the MLD's 5th Street warehouse facility. All pesticide sampler flow rates were within the QAS's $\pm 10\%$ control limit.

For each sampler using cartridges, the flow rate was set and recorded at the start of every sampling period for every sample using a calibrated, battery operated, digital mass flow meter. The flow rates were also checked and recorded at the end of each sampling period using a calibrated mass flow meter.

VI. Quality Control Results

A. Trip Blank Results

Referring to Table 3, Appendix II (page 34 of the Appendices to this report), seven trip blanks were analyzed for acephate and methamidophos and all trip blanks results were <MDL.

B. Collocated Sample Results

The relative percent difference (RPD) of the collocated results provides an indication of the precision of the monitoring method (i.e., the lower the RPD the better the precision). RPD is calculated as follows: $RPD = (| \text{difference} | / \text{average}) \times 100$.

Referring to Table 6, only one sample had an acephate result above the EQL. The corresponding collocated sample result was <MDL.

Referring to Table 7, 2 collocated pairs of samples had both methamidophos results above the EQL. The RPDs were 4.1% and 15.7%. The results indicate acceptable precision for the method.

C. Laboratory, Trip, and Field Spikes

The purpose of collecting spiked samples is to assess the accuracy (% recovery) of the sampling and analytical methods. The field spikes are collected by sampling ambient air through the previously spiked cartridges at one of the sampling sites. Thus, the field spikes provide an assessment of the accuracy of the entire method and are collected under the same environmental and experimental conditions as those occurring at the time of ambient sampling. The lab and trip spikes are used to confirm the field spike results or to help identify the source of losses (problems) when they occur in the field spikes.

Laboratory, trip, and field spikes were prepared by spiking a known amount of the target compound onto the appropriate cartridges. The spikes were made and collected in seven separate sets, one every week for the seven-week sampling period.

The laboratory spikes were placed immediately in a freezer and kept there until extraction and analysis. The trip and field spikes were kept in the lab freezer until transported to the field. The trip spikes were kept on dry ice in an ice chest (the same one used for samples) during transport to and from the field and at all times while in the

field except log-in and labeling. 300 ng of acephate and 210 ng of methamidophos were spiked onto a cartridge for all laboratory, trip, and field spikes. The extraction and analysis of each set of laboratory, trip and field spikes normally occurs at the same time. The collocated (unspiked) sample result, if above the EQL, was subtracted from the field spike sample result before calculation of percent recovery of the analytes. The lab, trip and field spikes for acephate and methamidophos are reported in Tables 8, 9 and 10 respectively.

The percent recoveries of the acephate lab, trip and field spikes ranged from 81% to 128%, 93% to 118%, and 48% to 163%, respectively.

For acephate, the lab and trip spike results are acceptable. Five of seven field spike recoveries are acceptable (86% to 126%), one is relatively low (48%) and one is high (163%). The laboratory report indicates that the high recovery field spike result may be caused by the interaction between the XAD resin and the target compound in the extraction solution and/or that the high and low results may be "outliers". The laboratory report states:

"Acephate is very unstable after extraction and there is no explanation to account for the wide variability of recovery efficiencies. While the acephate is stable on the XAD based on the storage studies, once in solution, even at low temperature it is not. The data from the laboratory and trip spike indicate that the extraction method is acceptable. The higher percentage recovery of acephate indicates that some interaction may be occurring between the XAD resin and the target compound. Re-analysis of an extract within 24 hours results in acephate values up to 2 times higher than the original value. Due to interfering peaks on the nitrogen phosphorous detector, use of this detector for confirmation was not available. The 48% and 163% recoveries of the field spikes appear to be outliers compared to the remainder of the field spike recovery data. The field spike recoveries during the method development were acceptable, although "high" for the lower spiked samples. Since the samples were predominantly <MDL, the actual values may be lower based on the "high" recoveries of the field spikes. All the samples were analyzed the day of extraction and not kept in extract storage longer than a few hours."

The percent recoveries of the methamidophos lab, trip and field spikes ranged from 80% to 96%, 86% to 96%, and 56% to 76%, respectively.

For methamidophos, the lab and trip spike results are acceptable, but the field spike recoveries are relatively low. According to lab report, random analysis of the back bed of the cartridges did not detect any methamidophos. There is no explanation for the low recoveries of methamidophos so far. During the collection and extraction efficiency study, the recoveries of methamidophos at low concentration (15 ng/sample) were good ($116.5\% \pm 12.4$), but at high concentration (210 ng/sample) were low ($46.4\% \pm 2.1$), which are consistent with the field spike recoveries (56% to 76%). The sample results for this project range from <MDL to 69 ng/sample. The consistently low recoveries of field spikes may indicate that ambient sample results reported are lower than actual.

VII. Method Development

Refer to Attachment III in Appendix I and Appendix II and IV for discussion and results of method development studies.

A. Collection and Extraction Efficiency

For acephate, the average recoveries for low (45 ng/sample) and high (300 ng/sample) spike levels were 193% and 111%, respectively. The method validation memo did not provide an explanation or discussion in regard to the high recoveries with the low-level spike. Based on the method development results, the actual concentrations of acephate in the samples may be lower than the values reported. All sample results for acephate are below the EQL of 45ng/sample except one sample with a result of 61.3 ng/sample.

For methamidophos, the average recoveries for low (15 ng/sample) and high (210 ng/sample) spike levels were 117% and 46%, respectively. The method validation memo did not provide an explanation or discussion in regard to the low recoveries with the high-level spike.

B. Storage Stability

The storage stability study results show that acephate and methamidophos are stable for up to 21 days on the XAD-2 cartridge when stored in a freezer at -20 °C. All samples were extracted and analyzed within 21 days of collection for the current project. Note however that the laboratory report states that: "Sample cartridges are stored at or below four (4) degrees centigrade (°C) before extraction."

C. Breakthrough

XAD-2 cartridges spiked with 1500 ng of acephate and methamidophos in the front tubes were sampled at a flow rate of 3 slpm for 24 hours. The analytical results show that no acephate and methamidophos were detected in the back tubes of cartridge.

Figure 1. Ambient Monitoring Area
(use map provided by DPR)

Attachment C(4): 2000 Methamidophos Use In Fresno, Madera, and Kings Counties (June 1- August 31, 2000)

