# METHOD VALIDATION REPORT

**STUDY TITLE**
Validation of Methods for the Analysis of Exposure Matrices for Acephate

Study Number AHE06
Morse Laboratories, Inc. Study Number ML03-1058-AHE

**DATA REQUIREMENT**

Series 875: Occupational and Residential Exposure Test Guidelines
40 CFR Part 136 or Part 141

**SPONSOR**
Agricultural Handlers Exposure Task Force
c/o Stewart Ag. Research Services, Inc.
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**REPORT DATE**
August 27, 2004

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## STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claims of confidentiality are made for any information contained in this study on the basis of its falling within the scope of FIFRA 10(d)(1)(A),(B) or (C).

Elliot Gordon, Ph.D.
Submitter
Administrative Committee Chair
Agricultural Handlers Exposure Task Force

11/22/04
Date

# COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

The Agricultural Handlers Exposure Task Force (AHETF) Study AHE06, "Validation of Methods for the Analysis of Exposure Matrices for Acephate" was conducted according to the Good Laboratory Practice Standards (GLPs), 40 CFR Part 160 as described in Federal Register, 54:34052-34074, August 17, 1989.

Elliot Gordon, Ph.D.
Submitter
Administrative Committee Chair
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Date 11/22/04

Susan Artz
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Date 8/27/04

Vicky Standart
Sponsor Representative
Technical Committee Chair
Agricultural Handlers Exposure Task Force

Date 11/10/04

# REPORT RE-ISSUE STATEMENT

This report is being re-issued for the following reasons:

1. The AHETF will be submiting all reports electronically. The original report was not written in the specific format required for electronic submissions.

2. The Statement of No Data Confidentiality Claims (page 2) of the original report incorrectly listed Vicky Standart instead of Elliot Gordon as the submitter.

3. The GLP Compliance page (Page 3) of the original report incorrectly listed Vicky Standard (misspelled) instead of Elliot Gordon as the Administrative Committee chair and submitter; and incorrectly listed David Johnson instead of Vicky Standart as the Sponsor.

4. The Study Identification and Approval page (page 5 of the original report) incorrectly listed Dave Johnson as the Sponsor Representative and Susan Artz as the Study Director. This page should have been signed by Richard Reed II as the Principal Investigator/Author and by Thomas Schreier as the AHETF Analytical Monitor.

This re-issued report corrects the errors noted above.

# AHETF QUALITY ASSURANCE STATEMENT

The Agricultural Handlers Exposure Task Force (AHETF) report for study AHE06 was audited by the AHETF Quality Assurance Unit (QAU) according to the applicable EPA Good Laboratory Practice Standards (40 CFR Part 160). All findings were reported to AHETF management and to the Study Director.

| Phase Inspected | Inspection Dates | Date Reported |
|---|---|---|
| Final Report Review | September 16-17, 2003 | September 18, 2003 |
| Post Audit Review | June 9-10, 2004<br>June 17-18, 2004 | June 10, 2004<br>June 18, 2004 |

Randy Fuller
AHETF Quality Assurance Unit
Compliance Assessment and Training Services, Inc.

Aug 26, 2004
Date

# MORSE LABORATORIES, INC. QUALITY ASSURANCE UNIT STATEMENT

Quality Assurance inspections were carried out during the execution of the AHETF Study AHE06 by Quality Assurance personnel according to §40 CFR 160.35 of the EPA Good Laboratory Practice Standards to ensure the integrity of the data. The final analytical report, as submitted to the Study Director, reflects the raw data.

| Phase Inspected | Inspection Dates | Date Findings Reported to the Study Director/Management |
|---|---|---|
| Protocol Review | May 6, 2003 | June 13, 2003 |
| In Process | May 12, 2003<br>May 14, 2003<br>June 4, 2003 | June 13, 2003 |
| Raw Data | May 27, 2003<br>May 28, 2003<br>June 19, 2003<br>August 5, 2003 | August 7, 2003 |
| Draft Report | July 24, 2003<br>August 4, 2003<br>August 5, 2003<br>August 14, 2003 | August 25, 2003 |
| Final Report | August 25, 2003 | August 25, 2003 |
| | July 24, 2004<br>August 16, 2004 | August 16, 2004 |

Sherita Normington
Sherita Normington
Morse Laboratories, Inc.
Quality Assurance Coordinator

Aug 16, 2004
Date

# STUDY IDENTIFICATION AND APPROVAL

| | |
|---|---|
| Sponsor: | Agricultural Handlers Exposure Task Force<br>c/o Stewart Ag. Research Services, Inc.<br>1893 Highway K<br>Clarence, MO 63437<br>660-762-4240<br>David R. Johnson<br>davejohn@marktwain.net |
| Sponsor Representative: | David R. Johnson, Ph.D. |
| Testing Facility: | Stewart Ag. Research Services, Inc.<br>1893 Highway K<br>Clarence, MO 63437 |
| Study Director: | Susan C. Artz<br>Artz Analytical Consulting Services, Inc.<br>3 Lansgate Ct.<br>Durham, NC 27713 |
| Study Initiation Date: | May 5, 2003 |
| Experimental Completion Date: | August 25, 2003 |
| Analytical Laboratory: | Morse Laboratories, Inc.<br>1525 Fulton Avenue<br>Sacramento, CA 95825 |
| Study Personnel: | Richard L. Reed II, Analytical Project Coordinator<br>Kevin Clark, Chief GC Chemist<br>Mike Percoski, GC/HLPLC Chemist<br>Yuk Wong, Laboratory Technician II<br>Phua Thao, Laboratory Technician II |

Data that are specific to the contracting laboratory will be archived at their respective facility. All other raw data, protocol and final report are stored in the Agricultural Handlers Exposure Task Force archives at Quality Associates, Inc., 9017 Red Branch Road, Suite 102, Columbia, MD 21045.

This report is approved and issued by:

Susan C. Artz

Susan Artz
Study Director

8/27/04
Date

Richard L. Reed II

Richard L. Reed II
Principal Investigator/Author

Aug 16, 2004
Date

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# 1.0 SUMMARY

This study was conducted to validate the analytical methodology for the determination of a pesticide, acephate, on inner dosimeter cloth matrix, face/neck wipe gauze matrix, hand wash matrix and OVS tubes. These are matrices that may be used in a study to be conducted to assess mixer, loader and applicator exposure to workers mixing, loading and applying pesticides. The four methods, AHETF-AM-001, "Determination of Acephate on Cotton Inner Dosimeters," dated February 13, 2003; AHETF-AM-002, "Determination of Acephate in Face/Neck Wipe Samples," dated February 18, 2003; AHETF-AM-003, "Determination of Acephate in Handwash Exposure Samples," dated February 19, 2003 and AHETF-AM-004, "Determination of Acephate in OVS Air Sampling Tubes," dated February 26, 2003, were all validated at Morse Laboratories, Inc., 1525 Fulton Ave., Sacramento, CA 95825. This report contains information on the experimental details, a summary of the analytical methods, calculations and a discussion of the results.

The following is a table of the matrices validated in this study and the results of the validations. All results were acceptable, meeting both EPA's guidelines with recoveries between 70 and 120% and standard deviations (SDs) of <20% and the AHETF's requirements with the mean recovery minus one standard deviation being ≥70% and the mean plus one standard deviation being ≤120% for each spike level for each matrix.

| Validated Matrix/Spike Level | Mean Recovery ±SD |
|---|---|
| Cotton Inner Dosimeters | |
| Low Level (0.5 µg/section) | 78.5% ± 3.4% |
| 2 x Low Level (1.0 µg/section) | 104% ± 14% |
| High Level (10,000 µg/section) | 97.3% ± 2.6% |
| Overall ( All Fortification Levels) | 93.2% ± 14% |
| Face/Neck Wipe Samples | |
| Low Level (0.5 µg/sample) | 93.5% ± 15% |
| 2 x Low Level (1.0 µg/sample) | 85.8% ± 4.1% |
| High Level (100 µg/sample) | 85.3% ± 5.6% |
| Overall ( All Fortification Levels) | 88.2% ± 9.5 |
| Hand Wash Samples | |
| Low Level (0.2 µg/sample) | 82.0% ± 5.5% |
| 2 x Low Level (1.0 µg/sample) | 93.5% ± 6.7% |
| High Level (1000 µg/sample) | 97.7% ± 7.3% |
| Overall ( All Fortification Levels) | 91.1% ± 9.2% |
| OVS Air Sampling Tubes | |
| Low Level (0.01 µg/tube) | 82.1% ± 7.7% |
| High Level (50 µg/tube) | 84.8% ± 6.4% |
| Overall ( All Fortification Levels) | 83.5% ± 6.9% |

## 2.0 INTRODUCTION AND BACKGROUND

This study is part of a series of studies being conducted by the Agricultural Handlers Exposure Task Force (AHETF). The AHETF was formed to develop a generic database for use by its member companies in assessing exposure to workers while mixing/loading and applying pesticide products. This study was designed to fulfill the requirements of Series 875: Occupational and Residential Exposure Test Guidelines and was conducted in accordance with EPA, FIFRA, Good Laboratory Practice Standards (GLP); 40 CFR, Part 160 (October, 1989).

Four methods were validated in this study:

AHETF-AM-001, "Determination of Acephate on Cotton Inner Dosimeters," dated February 13, 2003.

AHETF-AM-002, "Determination of Acephate in Face/Neck Wipe Samples," dated February 18, 2003.

AHETF-AM-003, "Determination of Acephate in Handwash Exposure Samples," dated February 19, 2003.

AHETF-AM-004, "Determination of Acephate in OVS Air Sampling Tubes," dated February 26, 2003.

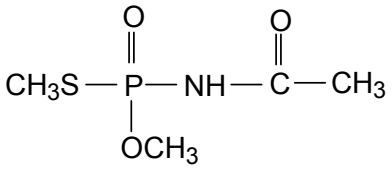
This study was conducted by Morse Laboratories, Inc. according to AHETF Study Protocol No. AHE06, entitled "Validation of Methods for the Analysis of Exposure Matrices for Acephate" (Appendix A).

# 3.0 MATERIALS

## 3.1 Reference Substance

| | |
|---|---|
| Common name: | acephate |
| Chemical formula: | $C_4H_{10}NO_3PS$ |
| Lot number: | AS 40r |
| CAS number: | 30560-19-1 |
| Purity: | 100% |
| Source: | Valent |
| Storage conditions: | Freezer, approximately -8 to -22°C |

Structure:




Acephate

The AHETF has retained a reserve sample of this chemical, and has documentation specifying the location of the synthesis and characterization information. The physical characteristics of the reference substance is the responsibility of the sponsor.

## 3.2 Test Matrices

The test matrices used in this study were cotton inner dosimeter material, face/neck wipe gauze, hand wash matrix and OVS air sampling tube matrix.

## 3.3 Reagents

The reagents used in these validations are all listed in the applicable methods noted in Section 1.0 of this report. The actual methods can be found in Appendix B.

# 4.0 METHODS

The methods described in the section can be found in Appendix B.

## 4.1 Description of AHETF-AM-001 "Determination of Acephate on Cotton Inner Dosimeters," dated February 13, 2003 by Gary Westberg, Morse Laboratories, Inc.

Acephate was extracted from cotton inner dosimeter sections with acetone. A combination of platform shaking and overnight soaking was employed to insure the efficient extraction of weathered (aged) residues. An aliquot of the extract was filtered and a measured volume of the filtrate was evaporated to dryness, then redissolved in acetone, at a suitable volume for GC analysis. Analysis was performed using gas chromatography employing flame photometric detection (FPD) in the phosphorous-specific mode. The method provides for a silica SPE cleanup if extract cleanup is deemed necessary (as determined by unacceptable chromatography resulting from co-elution of interfering compounds). The Limit of Quantification (LOQ) is 0.5 µg/section.

## 4.2 Description of AHETF-AM-002 "Determination of Acephate in Face/Neck Wipe Samples," dated February 18, 2003 by Gary Westberg, Morse Laboratories, Inc.

Acephate was extracted from cotton face/neck wipes with acetone. An aliquot of the extract was filtered and a measured volume of the filtrate was concentrated for GC analysis. Analysis was performed using gas chromatography employing flame photometric detection (FPD) in the phosphorous-specific mode. The method provides for a silica SPE cleanup if extract cleanup is deemed necessary (as determined by unacceptable chromatography resulting from co-elution of interfering compounds). The Limit of Quantification (LOQ) is 0.5 µg/sample.

## 4.3 Description of AHETF-AM-003 "Determination of Acephate in Handwash Exposure Samples," dated February 19, 2003 by Gary Westberg, Morse Laboratories, Inc.

Acephate was extracted from hand wash exposure samples using solid phase extraction (SPE). Acephate was retained on an activated charcoal SPE cartridge (AC-2 Sep-Pak® Plus) when passed through the cartridge in aqueous solution. It was eluted from the SPE cartridge with acetone. The acetone eluate was evaporated to dryness, then reconstituted in a volume of acetone suitable to attain the desired LOQ. The concentrate was submitted to gas chromatographic analysis employing flame photometric detection (FPD) in the phosphorous-specific mode. The Limit of Quantification (LOQ) is 0.2 µg/sample.

## 4.4 Description of AHETF-AM-004 "Determination of Acephate in OVS Air Sampling Tubes," dated February 26, 2003 by Gary Westberg, Morse Laboratories, Inc.

For this study, the entire OVS tube was analyzed as a whole tube. The method also provides for the analysis of a specific section of the tube (front or rear).

Acephate was extracted from the OVS air sampling tube with acetone using sonication. An aliquot of the extract was evaporated to dryness, reconstituted in acetone, then submitted to gas chromatographic analysis employing flame photometric detection (FPD). The method provides for an optional Silica SPE purification step which can be used on samples requiring additional cleanup. The limit of quantitation (LOQ), is 0.01 µg/tube.

## 4.5 Chromatographic Conditions

Chromatographic conditions are listed in the methods which can be found in Appendix B.

## 4.6 Statistic and Quantification

Calculations for instrumental analysis were conducted using a GLP validated software application to create a standard curve based on linear regression. The regression functions were used to calculate a best fit line (from a set of standard concentrations in µg/mL versus peak response) and to determine concentrations of the analyte found during sample analysis from the calculated best fit line. All standards injected were used to construct the standard curve.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

| | | |
|---|---|---|
| y | = | peak response |
| x | = | µg/mL found for peak of interest |
| m | = | slope |
| b | = | y-intercept |

Calculated recovery values from the spreadsheets were entered into a SYSTAT version 8.0 software program for calculation of the means, standard deviations and ranges.

## 4.7 Example Calculations

Example calculations are shown in the following sections for the determination of the amount of acephate and percent recovery for laboratory-fortified controls for each matrix.

### 4.7.a Calculations for Inner Dosimeter Samples

- **Equations**

$$\mu g/\text{section} = \mu g/\text{mL found} \times \frac{\text{final vol. (mL)}}{\text{one (1) section}} \times \frac{\text{mL solv.}}{\text{mL aliq.}} \times \text{GC dilution factor}$$

where:

| | |
|---|---|
| µg/section = | total µg of analyte present per section of sample analyzed |
| µg/mL found = | µg/mL of analyte found as determined by the analysis |
| one (1) section = | amount of sample taken through the extraction process |
| mL solv. = | extraction solvent volume (2000 mL) |
| mL aliq. = | volume of sample extract taken from initial extraction (80 mL) |
| mL final volume = | volume of final extract submitted to instrumentation (2.0 mL) |
| GC dil. factor = | dilution of sample extract required to produce an analyte response bracketed by standards |

$$\%\ \text{Recovery} = \frac{\mu g/\text{section found in fortified control} - \mu g/\text{section found in control}}{\mu g/\text{section added}} \times 100$$

- **Example Calculations**

**Lab-fortified Sample (upper legs)**

The following example calculation is from inner dosimeter set #3.

ML ticket #80987, set #3, acephate, inner dosimeter, 1055G-UL fortified control 19 at 0.5 µg/section, 1 mL = 0.02 section:

$$92.5 \text{ peak response} \rightarrow 0.0081\,\mu\text{g/mL}$$

$$\mu\text{g/section} = 0.0081\,\mu\text{g/mL} \times \frac{2.0 \text{ mL}}{1 \text{ section}} \times \frac{2000 \text{ mL}}{80 \text{ mL}} \times 1$$

$$= 0.405 \ \mu\text{g/section (reported as 0.4 } \mu\text{g/section)}$$

$$\%\ \text{Recovery} = \frac{0.405 \ \mu\text{g/section} - 0.000\ \mu\text{g/section}}{0.5\ \mu\text{g/section}} \times 100$$

$$= 81.0\%$$

### 4.7.b Calculations for Face/Neck Wipes

- **Equations**

$$\mu\text{g/sample} = \mu\text{g/mL found} \times \frac{\text{final vol. (mL)}}{\text{one (1) sample}} \times \frac{\text{mL solv.}}{\text{mL aliq.}} \times \text{GC dilution factor}$$

where:

| | |
|---|---|
| µg/sample = | total µg of analyte present per sample (2 wipes) analyzed |
| µg/mL found = | µg/mL of analyte found as determined by the analysis |
| one sample = | amount of sample taken through the extraction process (equivalent to 2 wipes combined) |
| mL solv. = | extraction solvent volume (100 mL) |
| mL aliq. = | volume of sample extract taken from initial extraction (2.0 mL) |
| mL final volume = | volume of final extract submitted to instrumentation (1.0 mL) |
| GC dilution factor = | dilution of sample extract required to produce an analyte response bracketed by standards |

$$\% \text{ Recovery} = \frac{\mu\text{g/sample found in fortified control - } \mu\text{g/sample found in control}}{\mu\text{g/sample added}} \text{ x } 100$$

- **Example Calculations**

**Lab-fortified Face/Neck Wipe Sample**

The following example calculation is from face/neck wipe set #2.

ML ticket #80986, set #2, acephate, face/neck wipe sample, 1055C fortified control 12 at 0.5 µg/sample, 1 mL = 0.02 sample:

$$90.6 \text{ peak response} \rightarrow 0.0105\ \mu\text{g/mL}$$

$$\mu\text{g/sample} = 0.0105\ \mu\text{g/mL} \times \frac{1.0\ \text{mL}}{1.0\ \text{sample}} \times \frac{100\ \text{mL}}{2.0\ \text{mL}} \times 1$$

$$= 0.525\ \mu\text{g/sample (reported as } 0.5\ \mu\text{g/sample)}$$

$$\% \text{ Recovery} = \frac{0.525\ \mu\text{g/sample} - 0.000\ \mu\text{g/sample}}{0.5\ \mu\text{g/sample}} \times 100$$

$$= 105\%$$

### 4.7.c Calculations for Hand Wash Samples

- **Equations**

$$\mu\text{g/sample} = \mu\text{g/mL found x } \frac{\text{final vol. (mL)}}{0.1\ \text{sample}} \text{ x GC dilution factor}$$

where:

| | |
|---|---|
| µg/sample = | total µg of analyte present per hand wash sample |
| µg/mL found = | µg/mL of analyte found as determined by the analysis |

| | |
|---|---|
| 0.1 sample = | amount of sample taken through the extraction process (equivalent to 50 mL of the 500 total mL of wash solution generated) |
| mL final volume = | volume of final extract submitted to instrumentation (2.0 mL) |
| GC dilution factor = | dilution of sample extract required to produce an analyte response bracketed by standards |

$$\% \text{ Recovery} = \frac{\mu\text{g/sample found in fortified control} - \mu\text{g/sample found in control}}{\mu\text{g/sample added}} \times 100$$

- **Example Calculations**

**Lab-fortified Hand Wash Sample**

The following example calculation is from hand wash set #9.

ML ticket #80985, set #9, acephate, hand wash sample, 1058A fortified control 67 at 0.2 µg/sample, 1 mL = 0.05 sample:

$$13125 \text{ peak response} \rightarrow 0.0089\ \mu\text{g/mL}$$

$$\mu\text{g/sample} = 0.0089\ \mu\text{g/mL} \times \frac{2.0\ \text{mL}}{0.1\ \text{sample}} \times 1$$

$$= 0.178\ \mu\text{g/sample (reported as } 0.2\ \mu\text{g/sample)}$$

$$\% \text{ Recovery} = \frac{0.178\ \mu\text{g/sample} - 0.000\ \mu\text{g/sample}}{0.2\ \mu\text{g/sample}} \times 100$$

$$= 89.0\%$$

### 4.7.d Calculations for OVS Air Sampling Tubes

- **Equations**

$$\mu\text{g/tube} = \mu\text{g/mL found} \times \frac{\text{final vol. (mL)}}{\text{one (1) sorbent tube}} \times \frac{\text{mL solv.}}{\text{mL aliq.}} \times \text{GC dilution factor}$$

where:

| | |
|---|---|
| µg/tube = | total µg of analyte present in the whole sorbent tube |
| µg/mL found = | µg/mL of analyte found as determined by the analysis |
| one (1) sorbent tube = | amount of sample taken through the extraction process |
| mL solv. = | extraction solvent volume (10.0 mL) |
| mL aliq. = | volume of sample extract taken from initial extraction (4.0 mL) |
| final vol. (mL) = | volume of final extract submitted to instrumentation (0.50 mL) |
| GC dil. factor = | dilution of sample extract required to produce an analyte response bracketed by standard |

$$\% \text{ Recovery} = \frac{\mu\text{g/tube found in fortified control - } \mu\text{g/tube found in control}}{\mu\text{g/tube added}} \text{ x } 100$$

- **Example Calculations**

**Lab-fortified OVS Air Sampling Tube**

The following example calculation is from OVS tubes set #7.

ML ticket #80990, set #7, acephate, OVS tube, 1058B fortified control 56 at 0.01 µg/tube, 1 mL = 0.8 tube:

$$72.9 \text{ peak response} \rightarrow 0.0070\ \mu\text{g/mL}$$

$$\mu\text{g/tube} = 0.0070\ \mu\text{g/mL} \times \frac{0.50\ \text{mL}}{1\ \text{tube}} \times \frac{10.0\ \text{mL}}{4.0\ \text{mL}} \times 1$$

$$= 0.00875\ \mu\text{g/tube (reported as } 0.009\ \mu\text{g/tube)}$$

$$\% \text{ Recovery} = \frac{0.00875\ \mu\text{g/tube} - 0.000\,\mu\text{g/tube}}{0.01\ \mu\text{g/tube}} \times 100$$

$$= 87.5\%$$

# 5.0 RESULTS AND CONCLUSIONS

All methods were performed as written. No modifications were made.

Methods were validated for cotton inner dosimeter, face/neck wipe, hand wash and OVS tube matrices. The LOQs for the methods were:

| Matrix | LOQ |
|---|---|
| Cotton Inner Dosimeters | 0.5 µg/section |
| Face/Neck Wipe Samples | 0.5 µg/sample |
| Hand Wash Samples | 0.2 µg/sample |
| OVS Air Sampling Tubes | 0.01 µg/tube |

## 5.1 Recovery of Acephate from Inner Dosimeters

The validation of the inner dosimeter method, AHETF-AM-001, "Determination of Acephate on Cotton Inner Dosimeters," dated February 13, 2003, was performed on two different days in two different analytical sample sets by two different analysts. Each set consisted of running a standard curve of five standard concentrations ranging from 0.005 µg/mL to 0.10 µg/mL (with all standards injected used to construct the standard curve), a control and three fortifications at each of three fortification levels (0.5 µg/section, the LOQ, 1.0 µg/section and 10,000 µg/section). The standard curve $R^2$ values were 0.999 and 0.999 for the first and second sample sets, respectively. Results from the validation of this method showed the following:

| Fortification Identification | Mean ± SD |
|---|---|
| Low Level (0.5 µg/section) | 78.5% ± 3.4% |
| 2 x Low Level (1.0 µg/section) | 104% ± 14% |
| High Level (10,000 µg/section) | 97.3% ± 2.6% |
| Overall (All Fortification Levels) | 93.2% ± 14% |

As can be seen, the results indicate that at all three fortification levels the method performed within the EPA requirements of 70 to 120% with standard deviations <20% and the AHETF's requirements with the mean recovery minus one standard deviation being ≥70% and the mean plus one standard deviation being ≤120% for each spike level. Individual sample analytical results can be found in Table 1 in the Table section of this report.

There were no residues (no detectable responses >30% of the LOQ) found in the control samples.

Back calculation of the standards against the standard curve showed all values to be within the AHETF Guideline requirements of ±15% for all standard concentrations except the lowest standard (typically 50% of the LOQ) which is ±20%. Details of these results are provided in Table 5 in the Table section of this report.

Details of the extraction of the samples can be found in Appendix C on the sample spreadsheets. Example chromatograms can be found in Appendix D.

## 5.2 Recovery of Acephate from Face/Neck Wipes

The validation of the face/neck wipe method, AHETF-AM-002, "Determination of Acephate in Face/Neck Wipe Samples," dated February 18, 2003, was performed on two different days in two different analytical sample sets by two different analysts. Each set consisted of running a standard curve of five standard concentrations ranging from 0.005 µg/mL to 0.10 µg/mL (with all standards injected used to construct the standard curve), a control and three fortifications at each of three fortification levels (0.5 µg/sample, the LOQ, 1.0 µg/sample and 100 µg/sample). The standard curve $R^2$ values were 0.994 and 0.999 for the first and second sample sets, respectively. Results from the validation of this method showed the following:

| Fortification Identification | Mean ± SD |
|---|---|
| Low Level (0.5 µg/sample) | 93.5% ± 15% |
| 2 × Low Level (1.0 µg/sample) | 85.8% ± 4.1% |
| High Level (100 µg/sample) | 85.3% ± 5.6% |
| Overall ( All Fortification Levels) | 88.2% ± 9.5% |

As can be seen, the results indicate that at all three fortification levels the method performed within the EPA requirements of 70 to 120% with standard deviations <20% and the AHETF's requirements with the mean recovery minus one standard deviation being ≥70% and the mean plus one standard deviation being ≤120% for each spike level.

Individual sample analytical results can be found in Table 2 in the Table section of this report.

There were no residues (no detectable responses >30% of the LOQ) found in the control samples.

Except for one "lowest standard" back calculated value at -20.7%, back calculation of all remaining standards against the standard curve showed values to be within the AHETF Guideline requirements of ±15% for all standard concentrations except the lowest standard (typically 50% of the LOQ) which is ±20%. Details of these results are provided in Table 6 in the Table section of this report.

Details of the extraction of the samples can be found in Appendix C on the sample spreadsheets. Example chromatograms can be found in Appendix D.

### 5.3 Recovery of Acephate from Hand Washes

The validation of the hand wash method, AHETF-AM-003, "Determination of Acephate in Handwash Exposure Samples," dated February 19, 2003, was performed on two different days in two different analytical sample sets by two different analysts. Each set consisted of running a standard curve of five standard concentrations ranging from 0.005 µg/mL to 0.10 µg/mL (with all standards injected used to construct the standard curve), a control and three fortifications at each of three fortification levels (0.2 µg/sample, the LOQ, 1.0 µg/sample and 1000 µg/sample). The standard curve $R^2$ values were 1.000 and 0.999 for the first and second sample sets, respectively. Results from the validation of this method showed the following:

| Fortification Identification | Mean ± SD |
|---|---|
| Low Level (0.2 µg/sample) | 82.0% ± 5.5% |
| 5 × Low Level (1.0 µg/sample) | 93.5% ± 6.7% |
| High Level (1000 µg/sample) | 97.7% ± 7.3% |
| Overall (All Fortification Levels) | 91.1% ± 9.2% |

As can be seen, the results indicate that at all three fortification levels the method performed within the EPA requirements of 70 to 120% with standard deviations <20% and the AHETF's requirements with the mean recovery minus one standard deviation being ≥70% and the mean plus one standard deviation being ≤120% for each spike level. Individual sample analytical results can be found in Table 3 in the Table section of this report.

There were no residues (no detectable responses >30% of the LOQ) found in the control samples.

Back calculation of the standards against the standard curve showed all values to be within the AHETF Guideline requirements of ±15% for all standard concentrations except the lowest standard (typically 50% of the LOQ) which is ±20%. Details of these results are provided in Table 7 in the Table section of this report.

Details of the extraction of the samples can be found in Appendix C on the sample spreadsheets. Example chromatograms can be found in Appendix D.

### 5.4 Recovery of Acephate from OVS Air Sampling Tubes

The validation of the OVS tube method, AHETF-AM-004, "Determination of Acephate in OVS Air Sampling Tubes," dated February 26, 2003, was performed on two different days in two different analytical sample sets by two different analysts. Each set consisted of running a standard curve of five standard concentrations ranging from 0.004 µg/mL to 0.10 µg/mL (with all standards injected used to construct the standard curve), a control and three fortifications at each of two fortification levels (0.01 µg/tube, the LOQ, and 50 µg/tube). The standard curve $R^2$ values were 1.000 and 0.999 for the first and second sample sets, respectively. Results from the validation of this method showed the following:

| Fortification Identification | Mean ± SD |
|---|---|
| Low Level (0.01 µg/tube) | 82.1% ± 7.7% |
| High Level (50 µg/tube) | 84.8% ± 6.4% |
| Overall (All Fortification Levels) | 83.5% ± 6.9% |

As can be seen, the results indicate that at both fortification levels the method performed within the EPA requirements of 70 to 120% with standard deviations <20% and the AHETF's requirements with the mean recovery minus one standard deviation being ≥70% and the mean plus one standard deviation being ≤120% for each spike level. Individual sample analytical results can be found in Table 4 in the Table section of this report.

There were no residues (no detectable responses >30% of the LOQ) found in the control samples.

Back calculation of the standards against the standard curve showed all values to be within the AHETF Guideline requirements of ±15% for all standard concentrations except the lowest standard (typically 50% of the LOQ) which is ±20%. Details of these results are provided in Table 7 in the Table section of this report.

Details of the extraction of the samples can be found in Appendix C on the sample spreadsheets. Example chromatograms can be found in Appendix D.

## 6.0 PROTOCOL /SOP CHANGES

There were no protocol amendments or deviations made to the protocol. Also, there were no AHETF or laboratory SOP changes issued.

## 7.0 CONCLUSION

The methods were shown to be satisfactory for the analyte and matrices specified. The overall recovery ranges and mean and standard deviations for the validation samples analyzed and described in this report show that the validations of the inner dosimeter, face/neck wipe, hand wash and OVS tube methods were successfully performed.

## 8.0 DISPOSITION OF RAW DATA

All original study-related raw data with the exception of the laboratories' facility documents will be transferred to the AHETF archives at Quality Associates, Inc., 9017 Red Branch Road, Suite 102, Columbia, MD 21045. Exact copies of the laboratories' facility documents will be sent to the AHETF for archiving. Authentic copies of the raw data will be maintained in the archive facilities of Morse Laboratories, Inc.

# TABLES

**Table 1. Validation Results for AHETF Method AHETF-AM-001, "Determination of Acephate on Cotton Inner Dosimeters"**

| Sample Identification | Set # | Sample Description | Extraction Date | Analysis Date | Fort. Level (µg/sect) | µg/sect Found | % Rec[1] |
|---|---|---|---|---|---|---|---|
| 1055G cont.1 | 1a[2] | control | 7-May-03 | 21-May-03 | N/A | <0.5 | N/A |
| 1055G fort.cont.1 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 0.5 | 0.4 | 80.0 |
| 1055G fort.cont.2 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 0.5 | 0.4 | 76.0 |
| 1055G fort.cont.3 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 0.5 | 0.4 | 83.0 |
| 1055G fort.cont.4 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 1.0 | 1.2 | 120 |
| 1055G fort.cont.5 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 1.0 | 1.1 | 114 |
| 1055G fort.cont.6 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 1.0 | 1.2 | 116 |
| 1055G fort.cont.7 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 10000 | 9880 | 98.8 |
| 1055G fort.cont.8 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 10000 | 10000 | 100 |
| 1055G fort.cont.9 | 1a[2] | fortification | 7-May-03 | 21-May-03 | 10000 | 9980 | 99.8 |
| 1055G cont.3 | 3 | control | 8-May-03 | 9-May-03 | N/A | <0.5 | N/A |
| 1055G fort.cont.19 | 3 | fortification | 8-May-03 | 9-May-03 | 0.5 | 0.4 | 81.0 |
| 1055G fort.cont.20 | 3 | fortification | 8-May-03 | 9-May-03 | 0.5 | 0.4 | 74.0 |
| 1055G fort.cont.21 | 3 | fortification | 8-May-03 | 9-May-03 | 0.5 | 0.4 | 77.0 |
| 1055G fort.cont.22 | 3 | fortification | 8-May-03 | 9-May-03 | 1.0 | 0.9 | 88.5 |
| 1055G fort.cont.23 | 3 | fortification | 8-May-03 | 9-May-03 | 1.0 | 0.9 | 94.5 |
| 1055G fort.cont.24 | 3 | fortification | 8-May-03 | 9-May-03 | 1.0 | 0.9 | 90.0 |
| 1055G fort.cont.25 | 3 | fortification | 8-May-03 | 9-May-03 | 10000 | 9460 | 94.6 |
| 1055G fort.cont.26 | 3 | fortification | 8-May-03 | 9-May-03 | 10000 | 9420 | 94.2 |
| 1055G fort.cont.27 | 3 | fortification | 8-May-03 | 9-May-03 | 10000 | 9620 | 96.2 |

[1] Recovery values are corrected for the concurrent control sample contribution.
[2] Set #1a is a retain extract analysis.

**0.5 µg/section fortification statistics:**

| | |
|---|---|
| Mean: | 78.5% |
| Standard deviation: | ±3.4 |
| Coefficient of variation: | 0.043 |
| (n=6) | |

**1.0 µg/ section fortification statistics:**

| | |
|---|---|
| Mean: | 104% |
| Standard deviation: | ±14 |
| Coefficient of variation: | 0.14 |
| (n=6) | |

**10,000 µg/ section fortification statistics:**

| | |
|---|---|
| Mean: | 97.3% |
| Standard deviation: | ±2.6 |
| Coefficient of variation: | 0.027 |
| (n=6) | |

**Overall fortification statistics:**

| | |
|---|---|
| Mean: | 93.2% |
| Standard deviation: | ±14 |
| Coefficient of variation: | 0.15 |
| (n=18) | |

**Table 2. Validation Results for AHETF Method AHETF-AM-002, "Determination of Acephate in Face/Neck Wipe Samples"**

| Sample Identification | Set # | Sample Description | Extraction Date | Analysis Date | Fort. Level (µg/samp) | µg/samp Found | % Rec[1] |
|---|---|---|---|---|---|---|---|
| 1055C cont.2 | 2 | control | 7-May-03 | 22-May-03 | N/A | <0.5 | N/A |
| 1055C fort.cont.10 | 2 | fortification | 7-May-03 | 22-May-03 | 0.5 | 0.5 | 106 |
| 1055C fort.cont.11 | 2 | fortification | 7-May-03 | 22-May-03 | 0.5 | 0.5 | 106 |
| 1055C fort.cont.12 | 2 | fortification | 7-May-03 | 22-May-03 | 0.5 | 0.5 | 105 |
| 1055C fort.cont.13 | 2 | fortification | 7-May-03 | 22-May-03 | 1.0 | 0.8 | 83.0 |
| 1055C fort.cont.14 | 2 | fortification | 7-May-03 | 22-May-03 | 1.0 | 0.9 | 87.0 |
| 1055C fort.cont.15 | 2 | fortification | 7-May-03 | 22-May-03 | 1.0 | 0.8 | 83.0 |
| 1055C fort.cont.16 | 2 | fortification | 7-May-03 | 22-May-03 | 100 | 83.0 | 83.0 |
| 1055C fort.cont.17 | 2 | fortification | 7-May-03 | 22-May-03 | 100 | 79.4 | 79.4 |
| 1055C fort.cont.18 | 2 | fortification | 7-May-03 | 22-May-03 | 100 | 79.8 | 79.8 |
| 1055C cont.4 | 4 | control | 9-May-03 | 13-May-03 | N/A | <0.5 | N/A |
| 1055C fort.cont.28 | 4 | fortification | 9-May-03 | 13-May-03 | 0.5 | 0.4 | 80.0 |
| 1055C fort.cont.29 | 4 | fortification | 9-May-03 | 13-May-03 | 0.5 | 0.4 | 73.0 |
| 1055C fort.cont.30 | 4 | fortification | 9-May-03 | 13-May-03 | 0.5 | 0.5 | 91.0 |
| 1055C fort.cont.31 | 4 | fortification | 9-May-03 | 13-May-03 | 1.0 | 0.8 | 81.5 |
| 1055C fort.cont.32 | 4 | fortification | 9-May-03 | 13-May-03 | 1.0 | 0.9 | 92.5 |
| 1055C fort.cont.33 | 4 | fortification | 9-May-03 | 13-May-03 | 1.0 | 0.9 | 87.5 |
| 1055C fort.cont.34 | 4 | fortification | 9-May-03 | 13-May-03 | 100 | 87.8 | 87.8 |
| 1055C fort.cont.35 | 4 | fortification | 9-May-03 | 13-May-03 | 100 | 88.4 | 88.4 |
| 1055C fort.cont.36 | 4 | fortification | 9-May-03 | 13-May-03 | 100 | 93.6 | 93.6 |

[1] Recovery values are corrected for the concurrent control sample contribution.

**0.5 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 93.5% |
| Standard deviation: | ±15 |
| Coefficient of variation: | 0.16 |
| (n=6) | |

**1.0 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 85.8% |
| Standard deviation: | ±4.1 |
| Coefficient of variation: | 0.048 |
| (n=6) | |

**100 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 85.3% |
| Standard deviation: | ±5.6 |
| Coefficient of variation: | 0.065 |
| (n=6) | |

**Overall fortification statistics:**

| | |
|---|---|
| Mean: | 88.2% |
| Standard deviation: | ±9.5 |
| Coefficient of variation: | 0.11 |
| (n=18) | |

**Table 3. Validation Results for AHETF Method AHETF-AM-003, "Determination of Acephate in Handwash Exposure Samples"**

| Sample Identification | Set # | Sample Description | Extraction Date | Analysis Date | Fort. Level (µg/samp) | µg/samp Found | % Rec[1] |
|---|---|---|---|---|---|---|---|
| 1058A cont.5 | 5 | control | 12-May-03 | 15-May-03 | N/A | <0.2 | N/A |
| 1058A fort.cont.37 | 5 | fortification | 12-May-03 | 15-May-03 | 0.2 | 0.2 | 81.0 |
| 1058A fort.cont.38 | 5 | fortification | 12-May-03 | 15-May-03 | 0.2 | 0.1 | 74.0 |
| 1058A fort.cont.39 | 5 | fortification | 12-May-03 | 15-May-03 | 0.2 | 0.2 | 78.0 |
| 1058A fort.cont.40 | 5 | fortification | 12-May-03 | 15-May-03 | 1.0 | 0.9 | 91.8 |
| 1058A fort.cont.41 | 5 | fortification | 12-May-03 | 15-May-03 | 1.0 | 0.9 | 88.4 |
| 1058A fort.cont.42 | 5 | fortification | 12-May-03 | 15-May-03 | 1.0 | 0.9 | 85.0 |
| 1058A fort.cont.43 | 5 | fortification | 12-May-03 | 15-May-03 | 1000 | 898 | 89.8 |
| 1058A fort.cont.44 | 5 | fortification | 12-May-03 | 15-May-03 | 1000 | 978 | 97.8 |
| 1058A fort.cont.45 | 5 | fortification | 12-May-03 | 15-May-03 | 1000 | 1110 | 111 |
| 1058A cont.9 | 9 | control | 19-May-03 | 20-May-03 | N/A | <0.2 | N/A |
| 1058A fort.cont.67 | 9 | fortification | 19-May-03 | 20-May-03 | 0.2 | 0.2 | 89.0 |
| 1058A fort.cont.68 | 9 | fortification | 19-May-03 | 20-May-03 | 0.2 | 0.2 | 84.0 |
| 1058A fort.cont.69 | 9 | fortification | 19-May-03 | 20-May-03 | 0.2 | 0.2 | 86.0 |
| 1058A fort.cont.70 | 9 | fortification | 19-May-03 | 20-May-03 | 1.0 | 1.0 | 104 |
| 1058A fort.cont.71 | 9 | fortification | 19-May-03 | 20-May-03 | 1.0 | 1.0 | 95.8 |
| 1058A fort.cont.72 | 9 | fortification | 19-May-03 | 20-May-03 | 1.0 | 1.0 | 96.2 |
| 1058A fort.cont.73 | 9 | fortification | 19-May-03 | 20-May-03 | 1000 | 990 | 99.0 |
| 1058A fort.cont.74 | 9 | fortification | 19-May-03 | 20-May-03 | 1000 | 940 | 94.0 |
| 1058A fort.cont.75 | 9 | fortification | 19-May-03 | 20-May-03 | 1000 | 944 | 94.4 |

[1] Recovery values are corrected for the concurrent control sample contribution.

**0.2 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 82.0% |
| Standard deviation: | ±5.5 |
| Coefficient of variation: | 0.067 |
| (n=6) | |

**1.0 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 93.5% |
| Standard deviation: | ±6.7 |
| Coefficient of variation: | 0.072 |
| (n=6) | |

**1000 µg/sample fortification statistics:**

| | |
|---|---|
| Mean: | 97.7% |
| Standard deviation: | ±7.3 |
| Coefficient of variation: | 0.075 |
| (n=6) | |

**Overall fortification statistics:**

| | |
|---|---|
| Mean: | 91.1% |
| Standard deviation: | ±9.2 |
| Coefficient of variation: | 0.10 |
| (n=18) | |

**Table 4. Validation Results for AHETF Method AHETF-AM-004, "Determination of Acephate in OVS Air Sampling Tubes"**

| Sample Identification | Set # | Sample Description | Extraction Date | Analysis Date | Fort. Level (µg/tube) | µg/tube Found | % Rec[1] |
|---|---|---|---|---|---|---|---|
| 1058B cont.7 | 7 | control | 13-May-03 | 14-May-03 | N/A | <0.01 | N/A |
| 1058B fort.cont.55 | 7 | fortification | 13-May-03 | 14-May-03 | 0.01 | 0.007 | 73.8 |
| 1058B fort.cont.56 | 7 | fortification | 13-May-03 | 14-May-03 | 0.01 | 0.009 | 87.5 |
| 1058B fort.cont.57 | 7 | fortification | 13-May-03 | 14-May-03 | 0.01 | 0.010 | 95.0 |
| 1058B fort.cont.58 | 7 | fortification | 13-May-03 | 14-May-03 | 50 | 45.6 | 91.2 |
| 1058B fort.cont.59 | 7 | fortification | 13-May-03 | 14-May-03 | 50 | 36.5 | 73.0 |
| 1058B fort.cont.60 | 7 | fortification | 13-May-03 | 14-May-03 | 50 | 44.8 | 89.6 |
| 1058B cont.8 | 8a[3] | control | 14-May-03 | 19-May-03 | N/A | <0.01[2] | N/A |
| 1058B fort.cont.61 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 0.01 | 0.008 | 78.8 |
| 1058B fort.cont.62 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 0.01 | 0.008 | 78.8 |
| 1058B fort.cont.63 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 0.01 | 0.008 | 78.8 |
| 1058B fort.cont.64 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 50 | 42.9 | 85.8 |
| 1058B fort.cont.65 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 50 | 41.9 | 83.8 |
| 1058B fort.cont.66 | 8a[3] | fortification | 14-May-03 | 19-May-03 | 50 | 42.8 | 85.6 |

[1] Recovery values are corrected for the concurrent control sample contribution.
[2] Residue found in control: 0.00212 µg/tube.
[3] Set #8a is a retain extract sample.

**0.01 µg/tube fortification statistics:**

| | |
|---|---|
| Mean: | 82.1% |
| Standard deviation: | ±7.7 |
| Coefficient of variation: | 0.094 |
| (n=6) | |

**50 µg/tube fortification statistics:**

| | |
|---|---|
| Mean: | 84.8% |
| Standard deviation: | ±6.4 |
| Coefficient of variation: | 0.076 |
| (n=6) | |

**Overall fortification statistics:**

| | |
|---|---|
| Mean: | 83.5% |
| Standard deviation: | ±6.9 |
| Coefficient of variation: | 0.083 |
| (n=12) | |

**Table 5. Standard Back Calculation Data for Samples Analyzed With AHETF Method AHETF-AM-001, "Determination of Acephate on Cotton Inner Dosimeters"**

**Set 1a.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 233.3 | 0.025191304 | 0.77 |
| 0.10 | 890.6 | 0.102565032 | 2.57 |
| 0.05 | 457.4 | 0.051571119 | 3.14 |
| 0.025 | 228.5 | 0.024626275 | -1.49 |
| 0.01 | 102.6 | 0.009806022 | -1.94 |
| 0.005 | 53.3 | 0.004002698 | -19.95 |
| 0.05 | 451.9 | 0.050923689 | 1.85 |
| 0.10 | 852.4 | 0.098068339 | -1.93 |
| 0.10 | 853.9 | 0.09824491 | -1.76 |

Standard Curve: y = mx + b, where: m = 8495.13161, b = 19.296556

**Set 3.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 240.9 | 0.024607976 | -1.57 |
| 0.10 | 935.0 | 0.101887802 | 1.89 |
| 0.05 | 484.5 | 0.051729955 | 3.46 |
| 0.01 | 112.4 | 0.01030102 | 3.01 |
| 0.005 | 58.0 | 0.004244224 | -15.11 |
| 0.025 | 239.0 | 0.024396433 | -2.41 |
| 0.05 | 474.8 | 0.050649975 | 1.30 |
| 0.10 | 892.7 | 0.097178198 | -2.82 |

Standard Curve: y = mx + b, where: m = 8981.64542, b = 19.879887

**Table 6. Standard Back Calculation Data for Samples Analyzed With AHETF Method AHETF-AM-002, "Determination of Acephate in Face/Neck Wipe Samples"**

**Set 2.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 219.4 | 0.028376914 | 13.51 |
| 0.10 | 780.2 | 0.106191058 | 6.19 |
| 0.05 | 383.6 | 0.051160585 | 2.32 |
| 0.01 | 89.0 | 0.010283183 | 2.83 |
| 0.005 | 47.7 | 0.004552577 | -8.95 |
| 0.025 | 201.7 | 0.02592094 | 3.68 |
| 0.05 | 366.6 | 0.04880174 | -2.40 |
| 0.05 | 331.4 | 0.043917542 | -12.16 |
| 0.10 | 705.2 | 0.095784388 | -4.22 |

Standard Curve: y = mx + b, where: m = 7206.91599, b = 14.889963

**Set 4.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.10 | 874.8 | 0.097127244 | -2.87 |
| 0.10 | 896.6 | 0.099593734 | -0.41 |
| 0.10 | 914.9 | 0.101664229 | 1.66 |
| 0.05 | 475.6 | 0.051961057 | 3.92 |
| 0.01 | 106.1 | 0.010155179 | 1.55 |
| 0.005 | 51.4 | 0.003966325 | -20.70 |
| 0.025 | 237.1 | 0.024976749 | -0.09 |
| 0.025 | 240.4 | 0.025350116 | 1.40 |
| 0.01 | 104.0 | 0.009917581 | -0.82 |
| 0.005 | 51.7 | 0.004000268 | -20.00 |
| 0.05 | 469.7 | 0.05129352 | 2.59 |

Standard Curve: y = mx + b, where: m = 8838.47012, b = 16.343754

**Table 7. Standard Back Calculation Data for Samples Analyzed With AHETF Method AHETF-AM-003, "Determination of Acephate in Handwash Exposure Samples"**

**Set 5.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 234.1 | 0.024570864 | -1.72 |
| 0.025 | 230.4 | 0.024160927 | -3.36 |
| 0.10 | 908.2 | 0.0992569 | -0.74 |
| 0.05 | 477.0 | 0.051482655 | 2.97 |
| 0.025 | 242.0 | 0.025446134 | 1.78 |
| 0.01 | 104.2 | 0.010178762 | 1.79 |
| 0.005 | 51.3 | 0.004317775 | -13.64 |
| 0.025 | 240.7 | 0.025302102 | 1.21 |
| 0.01 | 104.6 | 0.010223079 | 2.23 |
| 0.10 | 916.1 | 0.10013217 | 0.13 |
| 0.005 | 57.0 | 0.004949299 | -1.01 |

Standard Curve: $y = mx + b$, where: $m = 9025.78363$, $b = 12.328699$

**Set 9.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 44586 | 0.025418544 | 1.67 |
| 0.10 | 189983 | 0.101869057 | 1.87 |
| 0.05 | 91888 | 0.050290186 | 0.58 |
| 0.025 | 41622 | 0.023860057 | -4.56 |
| 0.01 | 17218 | 0.011028305 | 10.28 |
| 0.005 | 7281 | 0.005803378 | 16.10 |
| 0.05 | 89986 | 0.049290105 | -1.42 |
| 0.025 | 41115 | 0.023593474 | -5.63 |
| 0.10 | 183789 | 0.098612219 | -1.39 |
| 0.05 | 91784 | 0.050235503 | 0.47 |

Standard Curve: $y = mx + b$, where: $m = 1901844.67$, $b = -3756.1227$

**Table 8. Standard Back Calculation Data for Samples Analyzed With AHETF Method AHETF-AM-004, "Determination of Acephate in OVS Air Sampling Tubes"**

**Set 7.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.025 | 236.1 | 0.023619536 | -5.52 |
| 0.025 | 248.5 | 0.02488207 | -0.47 |
| 0.01 | 109.1 | 0.010688744 | 6.89 |
| 0.10 | 977.1 | 0.099066123 | -0.93 |
| 0.05 | 509.7 | 0.051476737 | 2.95 |
| 0.025 | 245.9 | 0.024617345 | -1.53 |
| 0.01 | 111.7 | 0.010953469 | 9.53 |
| 0.004 | 38.1 | 0.003459719 | -13.51 |
| 0.10 | 991.5 | 0.100532292 | 0.53 |
| 0.004 | 40.5 | 0.003704081 | -7.40 |

Standard Curve: y = mx + b, where: m = 9821.51782, b = 4.1203061

**Set 8a.**

| Nominal Value (µg/mL) | Peak Area | Back Calc. (µg/mL) | % Difference from Nominal Value |
|---|---|---|---|
| 0.01 | 19365 | 0.010837244 | 8.37 |
| 0.025 | 46735 | 0.02507674 | 0.31 |
| 0.10 | 192883 | 0.101111593 | 1.11 |
| 0.05 | 96219 | 0.05082125 | 1.64 |
| 0.025 | 44857 | 0.024099693 | -3.60 |
| 0.01 | 19132 | 0.010716024 | 7.16 |
| 0.004 | 6810 | 0.004305389 | 7.63 |
| 0.05 | 88935 | 0.047031682 | -5.94 |

Standard Curve: y = mx + b, where: m =1922118.54, b = -1465.4675

# APPENDICES

## Appendix A. Study Protocol

# AGRICULTURAL HANDLERS EXPOSURE TASK FORCE (AHETF)

## Analytical Method Validation Protocol

## STUDY No. AHE06

Study Title: Validation of Methods for the Analysis of Exposure Matrices for Acephate

## PROTOCOL AUTHORIZATION

**Read and Approved by:**

AHETF Sponsor Representative:

Signature David Johnson

David Johnson

Date May 05, 2003

Study Director:

Signature Susan C. Artz

Susan Artz

Date 5/5/03

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## 1. GENERAL INFORMATION

Study Title: Validation of Methods for the Analysis of Exposure Matrices For Acephate

Study No: AHE06

Objective of the Study:
The objective of this study is to validate analytical methods for the determination of acephate in exposure matrices to establish their accuracy and precision.

Proposed Experimental Start Date: May 1, 2003

Proposed Experimental Termination Date: May 23, 2003

Proposed Study Completion Date: August 1, 2003

Good Laboratory Practice:
This study will be conducted in compliance with the US EPA FIFRA Good Laboratory Practice (GLP) Standards (40 CFR 160) and will adhere to applicable AHETF and/or laboratory standard operating procedures (SOPs).

Pesticide Assessment Guideline:
This study is based upon EPA's guidance documents for dermal and inhalation exposure measurement under Series 875: Occupational and Residential Exposure Test Guidelines. The method write-up will be patterned after the 40 CFR Part 136 or Part 141 guideline for pesticide methodology.

Quality Assurance:
In-progress inspections and raw data/report audits will be conducted by the Quality Assurance Unit of the analytical laboratory. In addition, in-progress inspections and raw data and/or final report audits may be conducted by the AHETF Quality Assurance Unit.

Sponsor/Testing Facility: Agricultural Handlers Exposure Task Force
c/o David Johnson, Ph.D.
Stewart Agricultural Research Services, Inc.
1893 Highway K
Clarence, MO 63437
(660)762-4240

| | |
|---|---|
| Analytical Facility: | The analytical work will be carried out by: |
| | Morse Laboratories, Inc.<br>1525 Fulton Ave.<br>Sacramento, CA 95825 |
| | with Principal Analytical Investigator:<br>Richard Reed |
| Study Director: | Susan C. Artz<br>Artz Analytical Consulting Services, Inc.<br>3 Lansgate Ct.<br>Durham, NC 27713 |

## 2. INTRODUCTION

This study is part of a series of studies being conducted by the Agricultural Handlers Exposure Task Force (AHETF). The AHETF was formed to develop a generic database for use by its member companies in assessing exposure to workers mixing/loading and applying pesticide products.

This study will be concerned with validating the analytical methodology for the determination of a pesticide, acephate, on inner dosimeter cloth matrix, face/neck wipe gauze matrix, hand wash matrix and OVS tubes. These are matrices that will be used in a study to be conducted to assess mixer, loader and applicator exposure to workers mixing, loading and applying pesticides. This study is designed to fulfill the requirements of Series 875: Occupational and Residential Exposure Test Guidelines and will be conducted in accordance with EPA, FIFRA, Good Laboratory Practice Standards (GLP); 40 CFR, Part 160 (October, 1989).

## 3. TEST SYSTEM JUSTIFICATION AND IDENTIFICATION

The test system will consist of inner dosimeter cloth matrix, face/neck wipe gauze matrix, hand wash matrix and OVS tubes that will be used in mixer/loader and applicator exposure monitoring using acephate.

## 4. TEST/REFERENCE SUBSTANCE

| | |
|---|---|
| Common Name: | acephate |
| Chemical Formula: | $C_4H_{10}NO_3PS$ |
| CAS Number of a.i.: | 30560-19-1 |
| Percent Active Ingredient: | To be recorded |
| Storage Conditions: | To be recorded |

Information regarding the stability and concentration of the test substance is on file with the AHETF. A retention sample from the batch of the test substance was archived at Quality Associates, Inc., Columbia, MD.

## 5. ANALYTICAL METHODS TO BE VALIDATED

1. Method AHETF-AM-001, "Determination of Acephate On Cotton Inner Dosimeters," February 13, 2003.

2. Method AHETF-AM-002, "Determination of Acephate In Face/Neck Wipe Samples," February 18, 2003.

3. Method AHETF-AM-003, "Determination of Acephate In Hand Wash Exposure Samples," February 19, 2003.

4. Method AHETF-AM-004, "Determination of Acephate In OVS Air Sampling Tubes," February 26, 2003.

Equivalent instrumentation and reagents may be substituted for those specified in the methods. All substitutions must be clearly documented in the raw data along with the reasons for the changes.

## 6. STUDY PROCEDURES

A. All analytical methods and matrices except for the hand wash matrix (0.01% Aerosol® OT 75, AOT) will be provided by the AHETF. Procedures and techniques described in the methods will be followed as rigidly as possible. No changes are permitted without the prior approval of the AHETF.

B. The following procedures will be followed for validation of each method:

For each method to be validated, two sets of samples will be run. Each set will consist of 1 control, 3 LOQ fortifications, (0.5 µg/sample for inner dosimeters and face/neck wipes, and 0.2 µg/sample for hand washes) 3 fortifications at the 1 µg/sample level for all matrices but OVS tubes (for which the validation at the 0.01 µg/tube has been accounted for with the first validation level) and 3 fortifications to be made at a specified higher rate. The higher rate for the matrices to be tested are:

Inner Dosimeters: 10,000 µg/section
Face/neck wipe matrix: 100 µg/sample

Hand wash matrix: 1000 µg/sample
OVS tubes: 50 µg/tube

Each set of validation samples will be run on a different day by a different analyst.

C. Recoveries of acephate from spiked samples will be measured against a standard curve (five-point minimum) that will bracket the levels of the matrix spikes.

The validation study will be acceptable to the AHETF provided that the mean recovery minus one standard deviation is greater than or equal to 70% recovery and the mean plus one standard deviation is less than or equal to 120% recovery for each spike level for each matrix. The laboratory may, at its discretion, replicate any sample set upon approval from the AHETF Study Director.

Estimated number of analyses: 20 per matrix except OVS which is 14, total 74

## 7. SAMPLE DESCRIPTION

Samples will be identified by the study number, the matrix being tested, the fortification level and the fortification number. The laboratory may add their own identification as long as the AHETF identification is cross referenced and included in the documentation of the sample.

## 8. STATISTICAL EVALUATION

Chromatographic quantification will be achieved using a standard curve obtained from peak heights or areas of injections of several concentrations of standards. The standard curve will be a least squares fit unless otherwise approved by the AHETF. Mean, standard deviation and coefficient of variation will be calculated for each fortification level of each matrix. All data will be supplied in a columnar format showing actual results versus the mean, the relative standard deviation of the mean and the coefficient of variation.

## 9. RECORDS TO BE MAINTAINED

Along with the study final report, all study-specific original data generated in the course of this study, including but not limited to the following, shall be maintained and turned over to the AHETF at the completion of the study.

1. A copy of the study protocol as an appendix to the final report
2. Laboratory notebooks or bench sheets used to record details of the analyses
3. Chromatograms and/or machine-generated analysis reports and data
4. Spreadsheets and other calculated data
5. Chain-of-custody records

In addition to the above study-specific raw data, the following records must also be kept and original or true copies submitted with the raw data:

a. Storage conditions for test and reference substances and samples
b. Test and Reference substance use log
c. Communications logs or records

All data generated during this study will be kept in files bearing the study number. Following completion of the study, the laboratory will prepare the final report. All QA'd original raw data will be transferred directly to the AHETF GLP study archive at Quality Associates, Inc., 9017 Red Branch Road, Suite 102, Columbia, MD 21045 and stored for an indefinite period.

## 10. REPORT

At the completion of the study, the laboratory will submit a report to the AHETF. The report is required to be in the PR Notice 86-5 format. The report must contain but is not limited to containing:

a. A GLP compliance statement (40 CFR 160) signed by the Study Director and sponsor representative

b. A laboratory QAU statement giving dates of inspections and dates that findings were reported to the Study Director and Management

c. A detailed description of each method validation

d. Example calculations

e. A summary of each method validation procedure and a summary of the recovery data in the format described in the STATISTICAL EVALUATION SECTION

f. Representative chromatograms of control samples, fortified control samples and calibration standards

g. A representative standard curve

h. All elements of the EPA Data Reporting Guidelines on Analytical Methods

Specific details will be worked out by the Study Director and the AHETF when the report is in draft form.

## 11. AMENDMENTS AND DEVIATIONS

Amendments:

Amendments to this protocol must be approved by the Study Director and be acknowledged by the Sponsor. Protocol amendments are to be documented using a laboratory-supplied Protocol Amendment Form. The original change form will be retained with the protocol by the AHETF QAU. A copy of all amendments will be attached to the report.

Deviations:

Any deviations to the protocol, lab SOPs or GLPs, or situations that may affect the integrity of the study must be communicated to the Study Director in a timely manner. Protocol deviations are to be documented using a laboratory-supplied Protocol Deviation Form. The dated signatures from the Study Director and Sponsor representative of the AHETF must be obtained. The original change form will be retained with the protocol by the AHETF QAU. A copy of all deviations will be attached to the report.

SOP deviations are to be documented by the PI, approved by the SD and documented in the raw data. GLP deviations are to be documented on the "Statement of GLP Compliance" in the report. A description of any and all changes to the protocol must appear in the final report.

## 12. QUALITY ASSURANCE

The laboratory's QAU will inspect the study at appropriate intervals for consistency with the protocol, internal SOPs and raw data. All findings obtained during QAU inspections and the corrective actions taken must be documented and reported promptly to the Study Director and to the performing laboratory's management.

The AHETF's QAU will review the final report for compliance with the protocol and GLP regulations.

# Appendix B. Analytical Methods

**Determination of Acephate on Cotton Inner Dosimeters**

# DETERMINATION OF

# ACEPHATE ON COTTON INNER DOSIMETERS

**Analytical Method No.: AHETF-AM-001**

Agricultural Handlers Exposure Task Force
(AHETF)

Morse Laboratories, Inc.
1525 Fulton Avenue
Sacramento, CA 95825
TE 916/481-3141

Method Prepared by:
Gary L. Westberg

Date Issued:
February 13, 2003

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**DETERMINATION OF ACEPHATE ON COTTON INNER DOSIMETERS**

## 1.0 PRINCIPLE

Acephate is extracted from cotton inner dosimeter sections with acetone. A combination of platform shaking and overnight soaking is employed to insure the efficient extraction of weathered (aged) residues. An aliquot of the extract is filtered and a measured volume of the filtrate is evaporated to dryness, then redissolved in acetone, at a suitable volume for GC analysis. Analysis is performed using gas chromatography employing flame photometric detection (FPD) in the phosphorous-specific mode. The method provides for a silica SPE cleanup if extract cleanup is deemed necessary (as determined by unacceptable chromatography resulting from co-elution of interfering compounds). The Limit of Quantification (LOQ) is 0.5 µg/section.

## 2.0 EQUIVALENCE STATEMENT

During the conduct of this analysis, comparable apparatus, solvents, glassware, and techniques (such as sample extract evaporation) may be substituted for those described in this method, except where specifically stated. In the event a substituted piece of equipment or technique is used, its use will be documented in the study records.

## 3.0 APPARATUS AND EQUIPMENT

Assorted laboratory glassware:

| | |
|---|---|
| Balances: | Analytical balance capable of weighing to ±0.1 mg |
| | Top-loading balance capable of weighing to ±0.1 g |
| Evaporator: | Rotary evaporator equipped with a Dewar condenser (Buchler Instruments, Labconco, Lenexa, KS) |
| | N-Evap Laboratory Sample Evaporator, Model 115, attached to a nitrogen source (Organomation Associates, South Berlin, MA) |
| Evaporation flasks: | Glass, round or flat bottom, 250 and 125 mL |
| Funnels: | Glass, 100 mm diameter |

| | |
|---|---|
| Gas chromatograph: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |
| GC column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |
| GC guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica (Manufacturer: Restek) |
| GC inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. |
| Graduated cylinders: | Glass; 1000, 500, 250, 100, and 50 mL |
| Extraction jars: | Wide-mouth glass jars with Teflon®-lined lids: 130 oz. (California Glass Co., Oakland, CA) |
| Microliter syringes: | Various sizes (Hamilton Co., Reno, NV) |
| Pasteur pipets: | Glass, 9 inch and 5¾ inch, disposable |
| Pipets: | Glass, graduated, serological; various sizes |
| Pipets: | Glass, volumetric, class A; various sizes |
| Platform shaker: | Eberbach Model 6000 (Eberbach Corp., Ann Arbor, MI) |
| Solid Phase Extraction Apparatus: | Vac Elut SPS 24 (Varian Analytical Instruments, Sunnyvale, CA) |
| Storage jars: | Wide-mouth glass jars with Teflon®-lined lids: 4 oz. (Qorpak, Pittsburgh, PA) |
| Test (culture) tubes: | Glass, 13×100 mm and 16×150 mm |
| Ultrasonic bath: | Branson Model 2210 ultrasonic bath (VWR Scientific, Bridgeport, NJ) |
| Volumetric flasks: | Glass; 250, 100, 50 and 25 mL |
| Vortex mixer: | Vortexer 2 (VWR Scientific, Bridgeport, NJ) |

## 4.0 REAGENTS AND MATERIALS

| | |
|---|---|
| Acetone: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| 1-Decanol: | "Baker" (J.T. Baker Chemical Company, Phillipsburg, NJ) |
| Ethyl ether: | AR®, analytical reagent (Mallinckrodt Baker, Inc., Paris, Kentucky) |
| Filters: | Nylon, 25 mm, 0.45 µm (Alltech Assoc., Inc. Deerfield, IL) |
| Filter paper: | Whatman #4, 185 mm |
| Methanol: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Solid phase extraction tubes: | Silica Bond Elut LRC®, 10cc/500mg (Varian Sample Preparation Products, Harbor City, CA) |

4.1 Reagents and Materials to be Prepared (including typical preparation instructions)

4.1.1 *1% (w/v) keeper solution:* Place 1.0 g 1-decanol in a 100 mL volumetric flask. Fill to mark with acetone. Mix well.

4.1.2 *Silica SPE elution mixture [ethyl ether:methanol (9:1, v/v)]:* To a 250 mL volumetric flask, add 25 mL of methanol. Fill to mark with ethyl ether. Mix well. Prepare daily. Sufficient for approximately 20 samples.

## 5.0 STANDARD PREPARATION

5.1 Stock Standard Solution

Typically, 250 mg (corrected for purity) of acephate analytical standard is accurately weighed, quantitatively transferred to a 25 mL volumetric flask, and brought to volume with acetone to make a stock standard solution having a concentration of 10,000 µg/mL. This solution is to be stored at 1 to 8°C when not in use.

5.2 Fortification Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions are stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

1000 µg/mL: Transfer 2.5 mL of 10,000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

100 µg/mL: Transfer 2.5 mL of 1000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

10 µg/mL: Transfer 2.5 mL of 100 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

1.0 µg/mL: Transfer 2.5 mL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.1 µg/mL: Transfer 2.5 mL of 1.0 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

5.3 GC (Calibration) Standard Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions prepared in this section are to be stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

0.005 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 100 mL volumetric flask. Bring to volume in acetone. Mix well.

0.01 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.025 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.05 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.10 µg/mL: Transfer 250 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

## 6.0 SAMPLE DESCRIPTION/PREPARATION

A dosimeter section is defined as a portion of the whole body dosimeter that is taken through the analysis. For inner dosimeters, each whole body dosimeter (consisting of a one-piece, 100% cotton long underwear garment) is divided into six sections: 1) right and left upper arms, 2) right and left lower arms, 3) front torso, 4) rear torso, 5) right and left upper legs and 6) right and left lower legs.

## 7.0 SAMPLE FORTIFICATION

Fortify the sample by applying the appropriate amount of compound to a control dosimeter section using a graduated pipet or syringe. While applying the solution, make sure to distribute it over as much of the exposed sample as possible. Allow solvents to evaporate for approximately 10 minutes prior to extraction.

## 8.0 SAMPLE EXTRACTION

1. Place one dosimeter section into a 130 oz. wide-mouth glass jar. Fortify, as applicable, at this point. (Example: Prepare a 1.0 µg/section fortification by adding 1.0 mL of 1.0 µg/mL fortification solution to the sample). Add 2000 mL acetone.

2. Cap the jar tightly and shake for 20 minutes on a platform shaker (placing the jar on its side). Set shaker speed so that the extraction solvent is reaching all areas of the dosimeter sample (approximately 100 to 150 cycles/minute).

3. Remove the sample jars from the shaker and allow them to stand overnight at ambient temperature. This permits the sample to maintain a lengthy exposure to the extraction solvent which is crucial for maximum extraction efficiency of weathered residues.

4. Following overnight extraction, place the sample jars on the platform shaker for a second time and shake for 20 minutes.

5. Decant approximately 200 mL of extract through Whatman #4 filter paper. Discard the first ~20 mL through the paper and collect the next ~180 mL into a glass container of suitable size (preferably a 250 or 400 mL beaker).

6. Using a 100 mL graduated cylinder to measure, remove an 80 mL aliquot (equivalent to 0.04 section) and transfer to a 250 mL evaporation flask. Add 0.2 mL of 1% keeper solution.

   Transfer the remaining extract (~100 mL) to a 4 oz. Qorpak jar and store at <10 °C (refrigerator or freezer).

7. Concentrate to ~0.2 mL using a rotary evaporator set at ≤35 °C. Evaporate to dryness, with care, using manual nitrogen blowdown.

8. Redissolve residue in 2.0 mL of acetone. Sonicate, if necessary to dissolve/dislodge all residue. Quantitatively transfer extract, using small amounts of acetone to aid in the transfer, to a 13 × 100 mm screw-cap culture tube calibrated at 2.0 mL.

If no SPE cleanup is required, proceed to Step 9. If SPE cleanup is required, proceed to Step 11.1.

9 Concentrate to ~1.0-1.8 mL using an N-Evap evaporator set at ≤35°C. Bring to a final volume of 2.0 mL with acetone.

10. Filter the extract through a 0.45 µm nylon filter into a 13 × 100 mm culture tube and mark the meniscus. Submit to GC analysis without any further cleanup. Resulting concentration is 1 mL = 0.02 section. This concentration provides for an LOQ of 0.5 µg/section.

11. If the extract appears to be too "dirty" or the resulting chromatogram indicates the need for extract cleanup, proceed to the silica SPE cartridge cleanup which follows. If required, aliquots of the retain extract (Step 6) are used to process through SPE cleanup.

11.1 Concentrate the acetone extract from Step 8 to ~0.1-0.2 mL using an N-Evap evaporator set at ≤35°C, then evaporate to dryness with manual nitrogen blowdown.

11.2 Redissolve the residue in 3.0 mL ethyl ether. Sonicate to dissolve/dislodge all residue.

11.3 Proceed to silica SPE cartridge cleanup (Section 9.0).

## 9.0 SILICA SPE CARTRIDGE CLEANUP

**Note:** Check or calibrate the SPE cartridges prior to use in order to ensure optimum method performance. In general, check one cartridge per lot number per box. This assessment should be conducted well in advance of needing the cartridges for sample analysis. Recovery of >90% is desired to ensure that a box of cartridges is suitable for use. The analyses are conducted on a reagent spike basis.

See Appendix II for detailed instructions on assessment of the SPE cartridges.

**Procedure:**

1. Set up Vac Elut system and support apparatus and proceed with the silica SPE cleanup. In general, set the vacuum to produce a flow rate of approximately 2-3 distinct drops/second (not continuous flow) for all elutions.

2. Condition a 500 mg silica SPE cartridge by passing 5 mL of ethyl ether through the cartridge. Do not let the cartridge go to dryness after conditioning. (Stop elution when conditioning solvent reaches top of frit.) Discard eluate.

3. Place a 16 × 150 mm test tube under the SPE cartridge. Collect all eluates from the following load and elution steps.

4. Pass the ethyl ether extract (3 mL) from Step 8.0.11.3 through the SPE cartridge. Stop elution when the solvent reaches the top of the frit. Collect eluate.

5. Wash the test tube from Step 4 one time with 3 mL of ethyl ether. Swirl to rinse the walls of the tube and pass the rinse through the sample-laden cartridge. Stop elution when solvent reaches top of frit. Collect eluate.

6. Elute the acephate with 3 × 5 mL portions of ethyl ether:methanol (9:1, v/v) solution. Each 5 mL portion is used to rinse the test tube from Step 4 (using sonication, if necessary) prior to passing through the cartridge. Collect all eluates. Quantitatively transfer the combined eluates to a 125 mL evaporation flask, using 2 mL of ethyl ether:methanol (9:1, v/v) solution to aid in the transfer.

7. Add 0.2 mL of 1% keeper solution to the eluate and concentrate to ~0.1-0.2 mL using a rotary evaporator set at ≤30°C, then on to dryness, with care, using manual nitrogen blow down.

8. Redissolve the residue in 2.0 mL of acetone. Quantitatively transfer extract, using small amounts of acetone to aid in the transfer, to a 13 × 100 mm screw-cap culture tube calibrated at 2.0 mL.

9. Concentrate to ~1.0-1.8 mL using an N-Evap evaporator set at ≤35°C. Bring to a final volume of 2.0 mL with acetone. Sonicate to dissolve/dislodge all residue.

10. Filter the extract through a 0.45 µm nylon filter into a 13 × 100 mm culture tube and mark the meniscus. Submit to GC analysis. Resulting concentration is 1 mL = 0.02 section. This concentration provides for an LOQ of 0.5 µg/section.

## 10.0 GAS CHROMATOGRAPHIC ANALYSIS

**Note:** The column and conditions stated in the method have proved to have been satisfactory for the matrix being analyzed. The specific column packing/coating, carrier gas, column temperature and flow rate listed are typical conditions for this analysis. Specific conditions used will be noted on each chromatographic run and will not otherwise be documented.

10.1 Operating Conditions

| | | |
|---|---|---|
| Instrument: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. | |
| Inlet design: | "Purged/packed" recommended. "Capillary split-splitless" **is not** recommended. When set up to simulate a purged packed inlet, a capillary split-splitless inlet is often operated with the purge valve off over the course of the injection which, with megabore columns, tends to cause non-reproducible injections as well as non-linearity. | |
| Column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 | |
| Guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica | |
| Inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. | |
| Injection volume: | 2 µL | |
| Temperatures: | Injector: | 250°C |
| | Detector: | 200°C |
| | Column: | Initial: 130°C |
| | | Rate : 20°C/min. |
| | | Final: 230°C |
| Helium carrier gas flow: | 10 mL/min. | |
| Helium make-up gas flow: | 80 mL/min. | |
| Retention time: | ~3.7 minutes | |

10.2 Sample Analysis

Prepare a five-point standard curve by injecting constant volumes of acephate standard solutions. Use constant volume injections for sample extracts as well. Sample responses not bracketed by the standard curve require dilution and reinjection. Inject a curve check standard every 3-4 sample injections.

## 11.0 CALCULATIONS

Calculations for instrumental analysis are conducted using a validated software application to create a standard curve based on linear regression. The regression functions are used to calculate a best fit line (from a set of standard concentrations in µg/mL versus peak response) and to determine concentrations of the analyte found during sample analysis from the calculated best fit line.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

| | | |
|---|---|---|
| y | = | peak response |
| x | = | µg/mL found for peak of interest |
| m | = | slope |
| b | = | y-intercept |

The calculations for µg/section found and percent recovery (for fortified samples) are:

1. The amount of acephate found (in µg/section) in the sample is calculated according to the following equation:

$$\mu g/section = \mu g/mL\ found \times \frac{final\ vol.(mL)}{one\ (1)\ section} \times \frac{mL\ solv.}{mL\ aliq.} \times GC\ dilution\ factor$$

where:

| | | |
|---|---|---|
| µg/section | = | total µg of analyte present per section of sample analyzed |
| µg/mL found | = | µg/mL of analyte found as determined by the analysis |
| one (1) section | = | amount of sample taken through the extraction process |
| mL solv. | = | extraction solvent volume (2000 mL) |
| mL aliq. | = | volume of sample extract taken from initial extraction (80 mL) |

| | | |
|---|---|---|
| mL final volume | = | volume of final extract submitted to instrumentation (2.0 mL) |
| GC dil. factor | = | dilution of sample extract required to produce an analyte response bracketed by standard |

2. The percent recovery for fortified control samples is calculated as follows:

$$\%\ Recovery = \frac{\mu g/section\ found\ in\ fortified\ control - \mu g/section\ found\ in\ control}{\mu g/section\ added} x\,100$$

## 12.0 REFERENCES

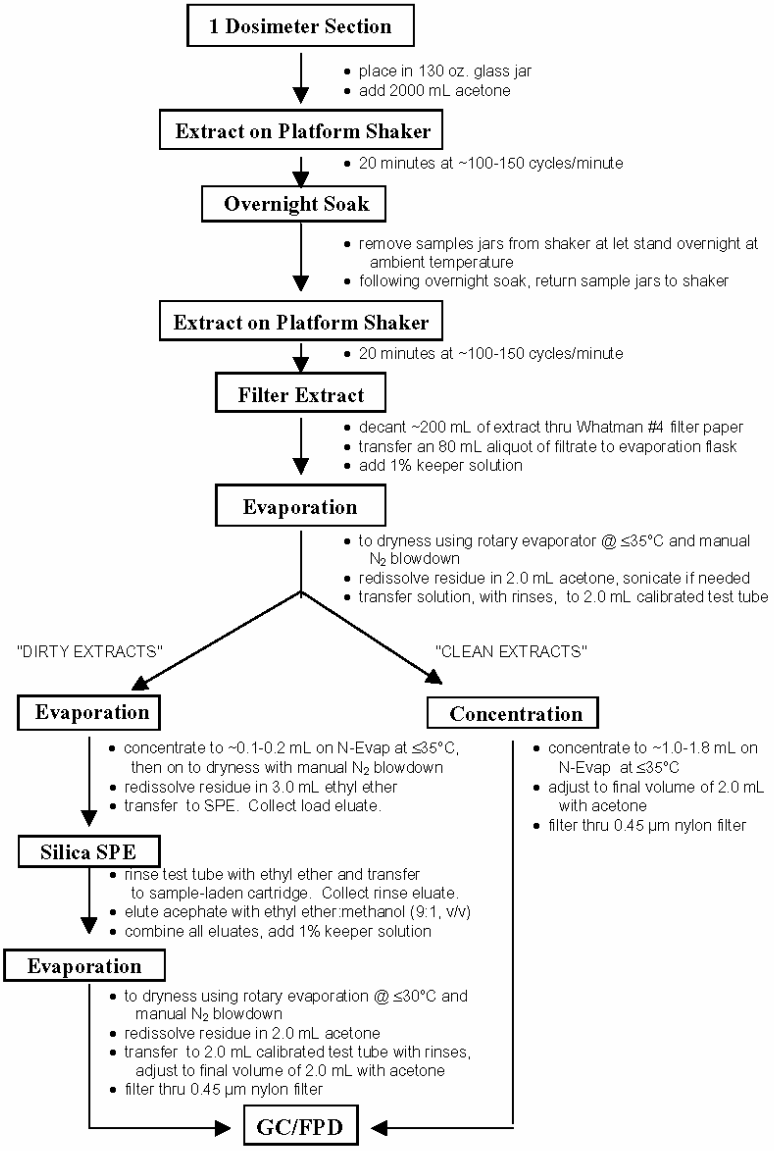
1. *Analytical Method for the Determination of Acephate in/on Worker Exposure Sampling Media Used in Worker Exposure Studies,* Method No. CCRL-MTH-030, dated May 23, 2001, CCRL (Central California Research Laboratories, Inc.), Fresno, California.

Method Author: Gary L. Westberg

**APPENDIX I**

Analysis Flowchart

**ANALYSIS FLOWCHART**



**APPENDIX II**

Quality Control for SPE Cartridges

Quality Control for SPE Cartridges

Silica SPE Cartridges

Add 50 µL of a standard solution @ 10 µg/mL (in acetone) to a 13 × 100 mm test tube. Add 0.2 mL of 1% keeper solution and evaporate to dryness using manual nitrogen blow down. Add 3.0 mL ethyl ether. Vortex mix. Follow Steps 9.0.1. through 9.0.7. of the procedure. Redissolve the residue in 5.0 mL of acetone and transfer directly, without rinses, to a 13 × 100 mm test tube. Submit to GC analysis. Final concentration is 0.1 µg acephate/mL.

## Determination of Acephate in Face/Neck Wipe Samples

# DETERMINATION OF

# ACEPHATE IN FACE/NECK WIPE SAMPLES

**Analytical Method No.: AHETF-AM-002**

Agricultural Handlers Exposure Task Force
(AHETF)

Morse Laboratories, Inc.
1525 Fulton Avenue
Sacramento, CA 95825
TE 916/481-3141

Method Prepared by:
Gary L. Westberg

Date Issued:
February 18, 2003

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**DETERMINATION OF ACEPHATE IN FACE/NECK WIPE SAMPLES**

## 1.0 PRINCIPLE

Acephate is extracted from cotton face/neck wipes with acetone. An aliquot of the extract is filtered and a measured volume of the filtrate is concentrated for GC analysis. Analysis is performed using gas chromatography employing flame photometric detection (FPD) in the phosphorous-specific mode. The method provides for a silica SPE cleanup if extract cleanup is deemed necessary (as determined by unacceptable chromatography resulting from co-elution of interfering compounds). The Limit of Quantification (LOQ) is 0.5 µg/sample.

## 2.0 EQUIVALENCE STATEMENT

During the conduct of this analysis, comparable apparatus, solvents, glassware, and techniques (such as sample extract evaporation) may be substituted for those described in this method, except where specifically stated. In the event a substituted piece of equipment or technique is used, its use will be documented in the study records.

## 3.0 APPARATUS AND EQUIPMENT

Assorted laboratory glassware:

| | |
|---|---|
| Balances: | Analytical balance capable of weighing to ±0.1 mg |
| | Top-loading balance capable of weighing to ±0.1 g |
| Evaporator: | Rotary evaporator equipped with a Dewar condenser (Buchler Instruments, Labconco, Lenexa, KS) |
| | N-Evap Laboratory Sample Evaporator, Model 115, attached to a nitrogen source (Organomation Associates, South Berlin, MA) |
| Evaporation flasks: | Glass, round or flat bottom, 125 mL |
| Extraction jars: | Wide-mouth glass jars with Teflon®-lined lids: 8 oz. (Qorpak, Pittsburgh, PA) |
| Funnels: | Glass, 100 mm diameter |
| Gas chromatograph: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |

| | |
|---|---|
| GC column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |
| GC guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica (Manufacturer: Restek) |
| GC inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. |
| Graduated cylinder: | Glass; 100 mL |
| Graduated mixing cylinder: | Glass; 500 mL |
| Microliter syringes: | Various sizes (Hamilton Co., Reno, NV) |
| Pasteur pipets: | Glass, 9 inch and 5¾ inch, disposable |
| Pipets: | Glass, graduated, serological; various sizes |
| Pipets: | Glass, volumetric, class A; various sizes |
| Platform shaker: | Eberbach Model 6000 (Eberbach Corp., Ann Arbor, MI) |
| Solid Phase Extraction Apparatus: | Vac Elut SPS 24 (Varian Analytical Instruments, Sunnyvale, CA) |
| Storage jars: | Wide-mouth glass jars with Teflon®-lined lids: 2 oz. (Qorpak, Pittsburgh, PA) |
| Test (culture) tubes: | Glass, 13×100 mm, 16 × 100 mm and 16×150 mm |
| Ultrasonic bath: | Branson Model 2210 ultrasonic bath (VWR Scientific, Bridgeport, NJ) |
| Volumetric flasks: | Glass; 250, 100, 50 and 25 mL |
| Vortex mixer: | Vortexer 2 (VWR Scientific, Bridgeport, NJ) |

## 4.0 REAGENTS AND MATERIALS

| | |
|---|---|
| Acetone: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Aerosol® OT Solution: | Aerosol® OT Solution, 10% w/w, aqueous, catalog #SA292-4 (Fisher Scientific, Fair Lawn, NJ) |
| 1-Decanol: | "Baker" (J.T. Baker Chemical Company, Phillipsburg, NJ) |
| Ethyl ether: | AR®, analytical reagent (Mallinckrodt Baker, Inc., Paris, Kentucky) |
| Filters: | Nylon, 25 mm, 0.45 µm (Alltech Assoc., Inc. Deerfield, IL) |
| Filter paper: | Whatman #4, 185 mm |
| Methanol: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Sodium Sulfate: | Analytical grade, anhydrous granular, #8024 (Mallinckrodt, St. Louis, MO) |
| Solid phase extraction tubes: | Silica Bond Elut LRC®, 10cc/500mg (Varian Sample Preparation Products, Harbor City, CA) |
| Water: | Deionized (DI) water (Polymetrics System, Morse Laboratories, Inc.) |

4.1 Reagents and Materials to be Prepared (including typical preparation instructions)

4.1.1 *1% (w/v) keeper solution:* Place 1.0 g 1-decanol in a 100 mL volumetric flask. Fill to mark with acetone. Mix well.

4.1.2 *Silica SPE elution mixture [ethyl ether:methanol (9:1, v/v)]:* To a 250 mL volumetric flask, add 25 mL of methanol. Fill to mark with ethyl ether. Mix well. Prepare daily. Sufficient for approximately 20 samples.

4.1.3 *Prewashed sodium sulfate:* Place approximately 60 g of sodium sulfate in a 100 mm glass funnel containing a Whatman #4 filter paper. Rinse with approximately 100 mL of acetone. Allow to dry. Sufficient for approximately 20 samples.

4.1.4 *0.01% Aerosol® OT Solution:* Pipet 0.5 mL of Aerosol® OT solution into a 500 mL graduated mixing cylinder. Fill to 500 mL mark with deionized water. Mix well.

## 5.0 STANDARD PREPARATION

### 5.1 Stock Standard Solution

Typically, 250 mg (corrected for purity) of acephate analytical standard is accurately weighed, quantitatively transferred to a 25 mL volumetric flask, and brought to volume with acetone to make a stock standard solution having a concentration of 10,000 µg/mL. This solution is to be stored at 1 to 8°C when not in use.

### 5.2 Fortification Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions are stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

1000 µg/mL: Transfer 2.5 mL of 10,000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

100 µg/mL: Transfer 2.5 mL of 1000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

10 µg/mL: Transfer 2.5 mL of 100 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

1.0 µg/mL: Transfer 2.5 mL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.1 µg/mL: Transfer 2.5 mL of 1.0 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

### 5.3 GC (Calibration) Standard Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions prepared in this section are to be stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

0.005 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 100 mL volumetric flask. Bring to volume in acetone. Mix well.

0.01 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.025 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 50 mL volumetric

flask. Bring to volume in acetone. Mix well.

0.05 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.10 µg/mL: Transfer 250 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

## 6.0 SAMPLE DESCRIPTION/PREPARATION

Each face/neck wipe sample consists of two 4 inch × 4 inch 100% cotton gauze sponges each wetted with 4 mL of 0.01% Aerosol® OT solution. Both gauze sponges are combined into one analytical sample. To simplify nomenclature, each gauze sponge is referred to as a "wipe" in this procedure.

## 7.0 SAMPLE FORTIFICATION

Fortify the sample by applying the appropriate amount of compound to both wipes using a graduated pipet or syringe. While applying the solution, make sure to distribute it over as much of the exposed sample as possible. Allow solvents to evaporate for approximately 10 minutes prior to extraction.

## 8.0 SAMPLE EXTRACTION

1. Place the sample (consisting of two wipes wetted with a total of 8 mL of 0.01% Aerosol OT solution) into an 8 oz. straight-sided round glass jar. Fortify, as applicable, at this point. (Example: Prepare a 1.0 µg/sample fortification by adding 1.0 mL of 1.0 µg/mL fortification solution to the sample). Add 100 mL acetone.

2. Cap the jar tightly and shake for 30 minutes on a platform shaker (placing the jar on its side). Set shaker speed so that the extraction solvent is reaching all areas of both wipes.

3. Decant approximately 70 mL through Whatman #4 filter paper. Discard the first ~40 mL through the paper and collect the next ~30 mL into a 2 oz. wide-mouth glass jar.

4. Remove a 5.0 mL aliquot (0.05 sample) and transfer to a 16 × 100 mm screw-cap culture tube, containing 3 g of sodium sulfate.

   Store the remaining extract at <10 °C (refrigerator or freezer).

5. Cap the tube and invert approximately 10 times to mix. Follow this with an approximate ~30 second hand shake of the contents.

6. Remove a 2.0 mL aliquot (0.02 sample) and transfer to another 13 × 100 mm screw-cap culture tube. Add 0.2 mL of 1% keeper solution and mix.

7. Concentrate to ~0.1-0.2 mL using a N-Evap set at ≤35 °C. Evaporate to dryness, with care, using manual nitrogen blowdown.

8. Redissolve residue in 1.0 mL of acetone. Sonicate, if necessary to dissolve/dislodge all residue. Mark the meniscus. Submit to GC analysis without any further cleanup. Resulting concentration is 1 mL = 0.02 sample. The concentration provides for a LOQ of 0.5 µg/sample.

   If SPE cleanup is required, proceed to Step 9.1.

9 If the extract appears to be too "dirty" or the resulting chromatogram indicates the need for extract cleanup, proceed to the silica SPE cartridge cleanup which follows. If required, aliquots of the retain extract (Step 4) are used to process through SPE cleanup.

9.1 Remove a 5.0 mL aliquot (0.05 sample) of the retain extract (Step 4) and transfer to a 16 × 100 mm screw-cap culture tube, containing 3 g of sodium sulfate.

9.2 Conduct Steps 5, 6 and 7 as written.

9.3 Redissolve the residue in 3.0 mL ethyl ether. Sonicate to dissolve/dislodge all residue.

9.4 Proceed to silica SPE cartridge cleanup (Section 9.0).

## 9.0 SILICA SPE CARTRIDGE CLEANUP

**Note:** Check or calibrate the SPE cartridges prior to use in order to ensure optimum method performance. In general, check one cartridge per lot number per box. This assessment should be conducted well in advance of needing the cartridges for sample analysis. Recovery of >90% is desired to ensure that a box of cartridges is suitable for use. The analyses are conducted on a reagent spike basis.

See Appendix II for detailed instructions on assessment of the SPE cartridges.

**Procedure:**

1. Set up Vac Elut system and support apparatus and proceed with the silica SPE cleanup. In general, set the vacuum to produce a flow rate of approximately 2-3 distinct drops/second (not continuous flow) for all elutions.

2. Condition a 500 mg silica SPE cartridge by passing 5 mL of ethyl ether through the cartridge. Do not let the cartridge go to dryness after conditioning. (Stop elution when conditioning solvent reaches top of frit.) Discard eluate.

3. Place a 16 × 150 mm test tube under the SPE cartridge. Collect all eluates from the following load and elution steps.

4. Pass the ethyl ether extract (3 mL) from Step 8.0.9.3 through the SPE cartridge. Stop elution when the solvent reaches the top of the frit. Collect eluate.

5. Wash the test tube from Step 4 one time with 3 mL of ethyl ether. Swirl to rinse the walls of the tube and pass the rinse through the sample-laden cartridge. Stop elution when solvent reaches top of frit. Collect eluate.

6. Elute the acephate with 3 × 5 mL portions of ethyl ether:methanol (9:1, v/v) solution. Each 5 mL portion is used to rinse the test tube from Step 4 (using sonication, if necessary) prior to passing through the cartridge. Collect all eluates. Quantitatively transfer the combined eluates to a 125 mL evaporation flask, using 2 mL of ethyl ether:methanol (9:1, v/v) solution to aid in the transfer.

7. Add 0.2 mL of 1% keeper solution to the eluate and concentrate to ~0.1-0.2 mL using a rotary evaporator set at ≤30°C, then on to dryness, with care, using manual nitrogen blowdown.

8. Redissolve the residue in 1.0 mL of acetone. Quantitatively transfer extract, using small amounts of acetone to aid in the transfer, to a 13 × 100 mm screw-cap culture tube calibrated at 1.0 mL.

9. Concentrate to ~0.6-0.8 mL using an N-Evap evaporator set at ≤35°C. Bring to a final volume of 1.0 mL with acetone. Sonicate to dissolve/dislodge all residue. Submit to GC analysis. Resulting concentration is 1 mL = 0.02 sample. This concentration provides for an LOQ of 0.5 µg/sample.

## 10.0 GAS CHROMATOGRAPHIC ANALYSIS

**Note:** The column and conditions stated in the method have proved to have been satisfactory for the matrix being analyzed. The specific column packing/coating, carrier gas, column temperature and flow rate listed are typical conditions for this analysis. Specific conditions used will be noted on each chromatographic run and will not otherwise be documented.

10.1 Operating Conditions

| | |
|---|---|
| Instrument: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |
| Inlet design: | "Purged/packed" recommended. "Capillary split-splitless" **is not** recommended. When set up to simulate a purged packed inlet, a capillary split-splitless inlet is often operated with the purge valve off over the course of the injection which, with megabore columns, tends to cause non-reproducible injections as well as non-linearity. |
| Column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |
| Guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica |
| Inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. |
| Injection volume: | 2 µL |
| Temperatures: | Injector: 250°C<br>Detector: 200°C<br>Column: Initial: 130°C<br>Rate : 20°C/min.<br>Final: 230°C |
| Helium carrier gas flow: | 10 mL/min. |
| Helium make-up gas flow: | 80 mL/min. |
| Retention time: | ~3.7 minutes |

10.2 Sample Analysis

Prepare a five-point standard curve by injecting constant volumes of acephate standard solutions. Use constant volume injections for sample extracts as well. Sample responses not bracketed by the standard curve require dilution and reinjection. Inject a curve check standard every 3-4 sample injections.

## 11.0 CALCULATIONS

Calculations for instrumental analysis are conducted using a validated software application to create a standard curve based on linear regression. The regression functions are used to calculate a best fit line (from a set of standard concentrations in µg/mL versus peak response)
and to determine concentrations of the analyte found during sample analysis from the calculated best fit line.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

| | | |
|---|---|---|
| y | = | peak response |
| x | = | µg/mL found for peak of interest |
| m | = | slope |
| b | = | y-intercept |

The calculations for µg/sample found and percent recovery (for fortified samples) are:

1. The amount of acephate found (in µg/sample) in the sample is calculated according

$$\mu g/sample = \mu g/mL\ found \times \frac{final\ vol.(mL)}{one\ (1)\ sample} \times \frac{mL\ solv.}{mL\ aliq.} \times GC\ dilution\ \ factor$$

to the following equation:
where:

| | | |
|---|---|---|
| µg/sample | = | total µg of analyte present per sample (2 wipes) analyzed |
| µg/mL found | = | µg/mL of analyte found as determined by the analysis |
| one (1) sample | = | amount of sample taken through the extraction process (equivalent to 2 wipes combined) |
| mL solv. | = | extraction solvent volume (100 mL) |
| mL aliq. | = | volume of sample extract taken from initial extraction (2.0 mL) |

| | | |
|---|---|---|
| mL final volume | = | volume of final extract submitted to instrumentation (1.0 mL) |
| GC dil. factor | = | dilution of sample extract required to produce an analyte response bracketed by standard |

2. The percent recovery for fortified control samples is calculated as follows:

$$\%\ Recovery = \frac{\mu g/sample\ found\ in\ fortified\ control - \mu g/sample\ found\ in\ control}{\mu g/sample\ added} \times 100$$

## 12.0 REFERENCES

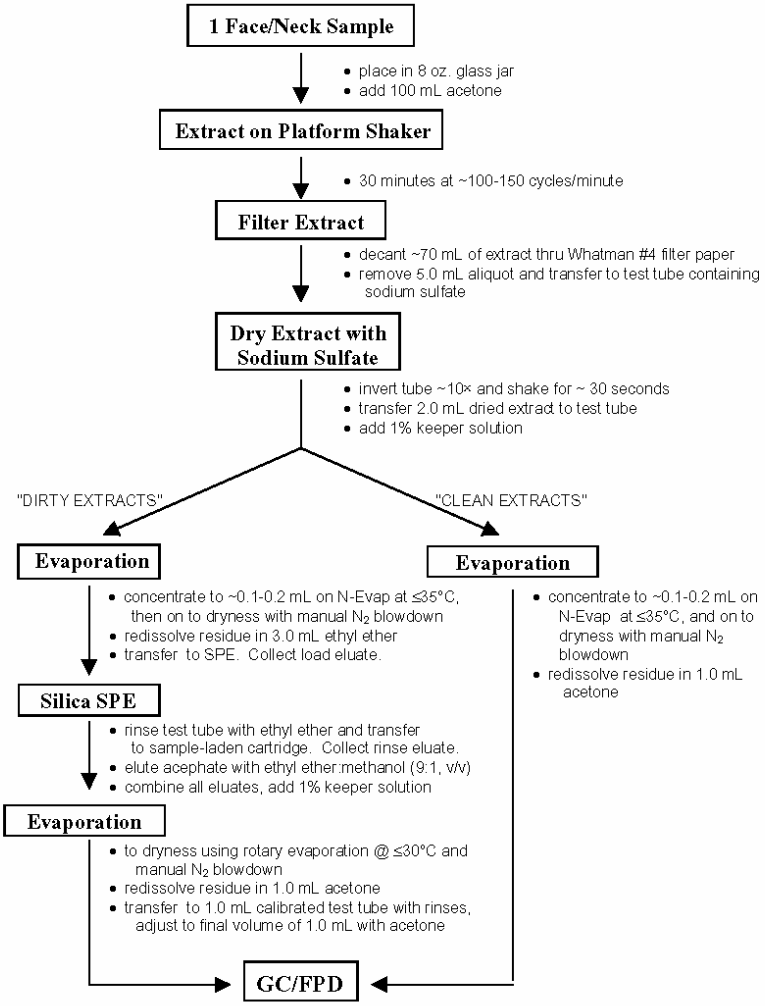
1. *Analytical Method for the Determination of Acephate in/on Worker Exposure Sampling Media Used in Worker Exposure Studies,* Method No. CCRL-MTH-030, dated May 23, 2001, CCRL (Central California Research Laboratories, Inc.), Fresno, California.

Method Author: Gary L. Westberg

**APPENDIX I**

Analysis Flowchart

**ANALYSIS FLOWCHART**



**APPENDIX II**

Quality Control for SPE Cartridges

Quality Control for SPE Cartridges

Silica SPE Cartridges

Add 50 µL of a standard solution @ 10 µg/mL (in acetone) to a 13 × 100 mm test tube. Add 0.2 mL of 1% keeper solution and evaporate to dryness using manual nitrogen blow down. Add 3.0 mL ethyl ether. Vortex mix. Follow Steps 9.0.1. through 9.0.7. of the procedure. Redissolve the residue in 5.0 mL of acetone and transfer directly, without rinses, to a 13 × 100 mm test tube. Submit to GC analysis. Final concentration is 0.1 µg acephate/mL.

## Determination of Acephate in Handwash Exposure Samples

# DETERMINATION OF

# ACEPHATE IN HANDWASH EXPOSURE SAMPLES

**Analytical Method No.: AHETF-AM-003**

Agricultural Handlers Exposure Task Force
(AHETF)

Morse Laboratories, Inc.
1525 Fulton Avenue
Sacramento, CA 95825
TE 916/481-3141

Method Prepared by:
Gary L. Westberg

Date Issued:
February 19, 2003

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**DETERMINATION OF ACEPHATE IN HANDWASH SOLUTION**

## 1.0 PRINCIPLE

Acephate is extracted from handwash exposure samples using solid phase extraction (SPE). Acephate is retained on an activated charcoal SPE cartridge (AC-2 Sep-Pak® Plus) when passed through the cartridge in aqueous solution. It is eluted from the SPE cartridge with acetone. The acetone eluate is evaporated to dryness, then reconstituted in a volume of acetone suitable to attain the desired LOQ. The concentrate is submitted to gas chromatographic analysis employing flame photometric detection (FPD) in the phosphorous-specific mode. The Limit of Quantification (LOQ) is 0.2 µg/sample.

## 2.0 EQUIVALENCE STATEMENT

During the conduct of this analysis, comparable apparatus, solvents, glassware, and techniques (such as sample extract evaporation) may be substituted for those described in this method, except where specifically stated. In the event a substituted piece of equipment or technique is used, its use will be documented in the study records.

## 3.0 APPARATUS AND EQUIPMENT

Assorted laboratory glassware:

| | |
|---|---|
| Balances: | Analytical balance capable of weighing to ±0.1 mg |
| | Top-loading balance capable of weighing to ±0.1 g |
| Evaporator: | Rotary evaporator equipped with a Dewar condenser (Buchler Instruments, Labconco, Lenexa, KS) |
| | N-Evap Laboratory Sample Evaporator, Model 115, attached to a nitrogen source (Organomation Associates, South Berlin, MA) |
| Evaporation flasks: | Glass, round or flat bottom, 125 mL |
| Gas chromatograph: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |
| GC column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |

| | |
|---|---|
| GC guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica (Manufacturer: Restek) |
| GC inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. |
| Graduated cylinders: | Glass; 100 and 50 mL |
| Graduated mixing cylinder: | Glass; 500, 250, 100 and 50 mL |
| Microliter syringes: | Various sizes (Hamilton Co., Reno, NV) |
| Pasteur pipets: | Glass, 9 inch and 5¾ inch, disposable |
| Pipets: | Glass, graduated, serological; various sizes |
| Pipets: | Glass, volumetric, class A; various sizes |
| Reservoir (for SPE cartridges): | Polyethylene, 75 mL |
| Solid Phase Extraction Apparatus: | Vac Elut SPS 24 (Varian Analytical Instruments, Sunnyvale, CA) |
| Test (culture) tubes: | Glass, 13×100 mm and 16×150 mm |
| Ultrasonic bath: | Branson Model 2210 ultrasonic bath (VWR Scientific, Bridgeport, NJ) |
| Volumetric flasks: | Glass; 250, 100, 50 and 25 mL |
| Vortex mixer: | Vortexer 2 (VWR Scientific, Bridgeport, NJ) |

## 4.0 REAGENTS AND MATERIALS

| | |
|---|---|
| Acetone: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Aerosol® OT solution: | Aerosol® OT Solution, 10% w/w, aqueous, catalog #SA292-4 (Fisher Scientific, Fair Lawn, NJ) |
| 1-Decanol: | "Baker" (J.T. Baker Chemical Company, Phillipsburg, NJ) |

| | |
|---|---|
| Ethyl acetate: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Ethyl ether: | AR®, analytical reagent (Mallinckrodt Baker, Inc., Paris, Kentucky) |
| Hydrochloric Acid: | Trace metal grade (Fisher Scientific, Pittsburgh, PA) |
| Methanol: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Solid phase extraction tubes: | AC-2 Sep-Pak® Plus Cartridge, 450 mg activated charcoal, Part JJAN20229 (Waters Corporation, Milford, MA) |
| | Silica Bond Elut LRC®, 10cc/500mg (Varian Sample Preparation Products, Harbor City, CA) |
| Water: | Deionized (DI) water (Polymetrics System, Morse Laboratories, Inc.) |

4.1 Reagents and Materials to be Prepared (including typical preparation instructions)

4.1.1 *0.01% Aerosol® OT solution*: Pipet 0.5 mL of 10% Aerosol® OT solution into a 500-mL graduated mixing cylinder. Fill to 500 mL mark with deionized water. Mix well.

4.1.2 *1% (w/v) keeper solution:* Place 1.0 g 1-decanol in a 100 mL volumetric flask. Fill to mark with acetone. Mix well.

4.1.3 *Silica SPE elution mixture [ethyl ether:methanol (9:1, v/v)]:* To a 250 mL volumetric flask, add 25 mL of methanol. Fill to mark with ethyl ether. Mix well. Prepare daily. Sufficient for approximately 20 samples.

4.1.4 *1N HCl:* Place 220 mL of DI water in a 250-mL graduated mixing cylinder. Carefully add 20 mL of concentrated HCl. Mix well. Sufficient for ~24 samples.

**5.0 STANDARD PREPARATION**

5.1 Stock Standard Solution

Typically, 250 mg (corrected for purity) of acephate analytical standard is accurately weighed, quantitatively transferred to a 25 mL volumetric flask, and brought to volume with acetone to make a stock standard solution having a concentration of 10,000 µg/mL. This solution is to be stored at 1 to 8°C when not in use.

5.2 Fortification Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions are stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

1000 µg/mL: Transfer 2.5 mL of 10,000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

100 µg/mL: Transfer 2.5 mL of 1000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

10 µg/mL: Transfer 2.5 mL of 100 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

1.0 µg/mL: Transfer 2.5 mL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.1 µg/mL: Transfer 2.5 mL of 1.0 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

5.3 GC (Calibration) Standard Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions prepared in this section are to be stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

0.005 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 100 mL volumetric flask. Bring to volume in acetone. Mix well.

0.01 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.025 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.05 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.10 µg/mL: Transfer 250 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

## 6.0 SAMPLE DESCRIPTION/PREPARATION

Each handwash sample consists of 500 mL of 0.01% Aerosol® OT solution used to wash both the left and right hands of one person. A 50 mL aliquot, representing 0.1 sample, is taken for analysis.

1. All samples are kept in the frozen state until thawed just prior to analysis. (Completion of thawing should coincide with the start of analysis—optimally within 1-2 hours.)

2. Thawing of samples may be conducted in a refrigerator overnight up to a maximum of 3 days. If necessary, the thawing process may be completed in a 45°C water bath (applies especially to samples thawed overnight.)

3. Place sample bottles in a clean beaker when thawing in a water bath to prevent sample loss.

4. Thawed samples must be well mixed prior to handling.

## 7.0 SAMPLE FORTIFICATION

Concurrent quality control samples (control and procedural recovery samples) are prepared from either field-generated or laboratory-generated control handwash solution. In either case, the procedural recovery sample is prepared by placing 50 mL of "control" 0.01% Aerosol® OT solution directly into a graduated mixing cylinder (50 or 100 mL) where it is fortified with an appropriate amount of acephate standard solution. If possible, the fortification solution volume used is kept ≤1.0 mL. Following fortification, the sample is mixed well to thoroughly distribute the analyte.

## 8.0 SAMPLE EXTRACTION

**Note:** Check or calibrate the SPE cartridges prior to use in order to ensure optimum method performance. In general, check one cartridge per lot number per box. This assessment should be conducted well in advance of needing the cartridges for sample analysis. Recovery of >90% is desired to ensure that a box of cartridges is suitable for use. The analyses are conducted on a reagent spike basis.

See Appendix II for detailed instructions on assessment of the SPE cartridges.

1. Set up Vac Elut system and support apparatus and proceed with AC-2 (activated charcoal) solid phase extraction. In general, set vacuum to produce a flow rate of approximately 2-3 mL/min for all elutions.

2. Attach a 75-mL SPE polyethylene reservoir to an AC-2 Sep-Pak® Plus cartridge and condition by passing 5 mL of acetone followed by 10 mL of deionized water, 10 mL of 1N HCl and 10 mL of deionized water through the cartridge. Do not let the cartridge to go to dryness after conditioning. (Stop elution when conditioning solvent reaches top of frit.) Discard eluate.

3. Using a graduated cylinder (50 or 100 mL), load 50 mL of handwash sample (0.10 sample) onto the cartridge and allow the sample to pass through, discarding the eluate. Do not allow the sample cartridge to go dry.

4. Rinse the SPE reservoir with 10 mL of deionized water and pass through the AC-2 cartridge, discarding the eluate.

5. Allow the cartridge to dry while pulling a vacuum for 30 to 60 seconds.

6. Add 5 mL of acetone to the cartridge and allow approximately 2 to 3 mL to pass through into a 16 × 150 mm test tube. (This tube is used to collect, and thus combine, all remaining eluates.) Stop the elution to permit the activated charcoal to soak with the acetone for approximately 15 minutes. Following the soak, add an additional 5 mL acetone to the cartridge.

7. Restart the elution process. Continue eluting acephate with the remaining volume of acetone. Before the cartridge goes dry, add a second 10-mL volume of acetone eluant to complete the elution (~20 mL total elution volume). A 15-minute soak period is not required with the second 10-mL volume of eluant.

8. Quantitatively transfer the combined eluates to a 125-mL evaporation flask. Add 5 mL of ethyl acetate and 0.2 mL of 1% keeper solution.

9. Evaporate to dryness using rotary evaporation equipped with a water bath set at ≤35°C. If residual water is present, add an additional 5 mL of ethyl acetate and 10 mL of acetone and re-evaporate to ~0.2 mL. Evaporate to dryness using manual nitrogen blowdown.

10. Redissolve residue in 2.0 mL of acetone. Sonicate, if necessary to dissolve/dislodge all residue. Quantitatively transfer extract, using small amounts of acetone to aid in the transfer, to a 13 × 100 mm screw-cap culture tube calibrated at 2.0 mL.

    If no SPE cleanup is required, proceed to Step 11. If SPE cleanup is required, proceed to Step 13.1.

11. Concentrate to ~1.0-1.8 mL using an N-Evap evaporator set at ≤35°C. Bring to a final volume of 2.0 mL with acetone.

12. Submit to GC analysis without any further cleanup. Resulting concentration is 1 mL = 0.05 sample. This concentration provides for an LOQ of 0.2 µg/sample.

13. If the extract appears to be too "dirty" or the resulting chromatogram indicates the need for extract cleanup, proceed to the silica SPE cartridge cleanup which follows. If it has been determined by analysis that cleanup is required, a fresh 50-mL aliquot of the handwash sample must be processed through the entire procedure through and including Step 10.

13.1 Concentrate the acetone extract from Step 10 to ~0.1-0.2 mL using an N-Evap evaporator set at ≤35°C, then evaporate to dryness with manual nitrogen blowdown.

13.2 Redissolve the residue in 3.0 mL ethyl ether. Sonicate to dissolve/dislodge all residue.

13.3 Proceed to silica SPE cartridge cleanup (Section 9.0).

## 9.0 SILICA SPE CARTRIDGE CLEANUP

**Note:** Check or calibrate the SPE cartridges prior to use in order to ensure optimum method performance. In general, check one cartridge per lot number per box. This assessment should be conducted well in advance of needing the cartridges for sample analysis. Recovery of >90% is desired to ensure that a box of cartridges is suitable for use. The analyses are conducted on a reagent spike basis.

See Appendix II for detailed instructions on assessment of the SPE cartridges.

**Procedure:**

1. Set up Vac Elut system and support apparatus and proceed with the silica SPE cleanup. In general, set the vacuum to produce a flow rate of approximately 2-3 distinct drops/second (not continuous flow) for all elutions.

2. Condition a 500 mg silica SPE cartridge by passing 5 mL of ethyl ether through the cartridge. Do not let the cartridge go to dryness after conditioning. (Stop elution when conditioning solvent reaches top of frit.) Discard eluate.

3. Place a 16 × 150 mm test tube under the SPE cartridge. Collect all eluates from the following load and elution steps.

4. Pass the ethyl ether extract (3 mL) from Step 8.0.13.2 through the SPE cartridge. Stop elution when the solvent reaches the top of the frit. Collect eluate.

5. Wash the test tube from Step 4 one time with 3 mL of ethyl ether. Swirl to rinse the walls of the tube and pass the rinse through the sample-laden cartridge. Stop elution when solvent reaches top of frit. Collect eluate.

6. Elute the acephate with 3 × 5 mL portions of ethyl ether:methanol (9:1, v/v) solution. Each 5 mL portion is used to rinse the test tube from Step 4 (using sonication, if necessary) prior to passing through the cartridge. Collect all eluates. Quantitatively transfer the combined eluates to a 125 mL evaporation flask, using 2 mL of ethyl ether:methanol (9:1, v/v) solution to aid in the transfer.

7. Add 0.2 mL of 1% keeper solution to the eluate and concentrate to ~0.1-0.2 mL using a rotary evaporator set at ≤30°C, then on to dryness, with care, using manual nitrogen blow down.

8. Redissolve the residue in 2.0 mL of acetone. Quantitatively transfer extract, using small amounts of acetone to aid in the transfer, to a 13 × 100 mm screw-cap culture tube calibrated at 2.0 mL.

9. Concentrate to ~1.0-1.8 mL using an N-Evap evaporator set at ≤35°C. Bring to a final volume of 2.0 mL with acetone. Sonicate to dissolve/dislodge all residue.

10. Submit to GC analysis. Resulting concentration is 1 mL = 0.05 sample. This concentration provides for an LOQ of 0.2 µg/sample.

## 10.0 GAS CHROMATOGRAPHIC ANALYSIS

**Note:** The column and conditions stated in the method have proved to have been satisfactory for the matrix being analyzed. The specific column packing/coating, carrier gas, column temperature and flow rate listed are typical conditions for this analysis. Specific conditions used will be noted on each chromatographic run and will not otherwise be documented.

### 10.1 Operating Conditions

Instrument: Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation.

Inlet design: "Purged/packed" recommended. "Capillary split-splitless" **is not** recommended. When set up to simulate a purged packed inlet, a capillary split-splitless inlet is often operated with the purge valve off over the course of the injection which, with megabore columns, tends to cause non-reproducible injections as well as non-linearity.

| | | |
|---|---|---|
| Column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 | |
| Guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica | |
| Inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™ | |
| Injection volume: | 2 µL | |
| Temperatures: | Injector: | 250°C |
| | Detector: | 200°C |
| | Column: | Initial: 130°C |
| | | Rate : 20°C/min. |
| | | Final: 230°C |
| Helium carrier gas flow: | 10 mL/min. | |
| Helium make-up gas flow: | 80 mL/min. | |
| Retention time: | ~3.7 minutes | |

10.2 Sample Analysis

Prepare a five-point standard curve by injecting constant volumes of acephate standard solutions. Use constant volume injections for sample extracts as well. Sample responses not bracketed by the standard curve require dilution and reinjection. Inject a curve check standard every 3-4 sample injections.

## 11.0 CALCULATIONS

Calculations for instrumental analysis are conducted using a validated software application to create a standard curve based on linear regression. The regression functions are used to calculate a best fit line (from a set of standard concentrations in µg/mL versus peak response) and to determine concentrations of the analyte found during sample analysis from the calculated best fit line.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

| | | |
|---|---|---|
| y | = | peak response |
| x | = | µg/mL found for peak of interest |
| m | = | slope |
| b | = | y-intercept |

The calculations for µg/sample found and percent recovery (for fortified samples) are:

1. The amount of acephate found (in µg/sample) in the sample is calculated according to the following equation:

$$\mu g/sample = \mu g/mL\ found \times \frac{final\ vol.(mL)}{0.1\ sample} \times GC\ dilution\ factor$$

where:

| | | |
|---|---|---|
| µg/sample | = | total µg of analyte present per handwash sample |
| µg/mL found | = | µg/mL of analyte found as determined by the analysis |
| 0.1 sample | = | amount of sample taken through the extraction process (equivalent to 50 mL of the 500 total mL of wash solution generated) |
| mL final volume | = | volume of final extract submitted to instrumentation (2.0 mL) |
| GC dil. factor | = | dilution of sample extract required to produce an analyte response bracketed by standard |

2. The percent recovery for fortified control samples is calculated as follows:

$$\%\ Recovery = \frac{\mu g/sample\ found\ in\ fortified\ control - \mu g/sample\ found\ in\ control}{\mu g/sample\ added} \times 100$$

## 12.0 REFERENCE

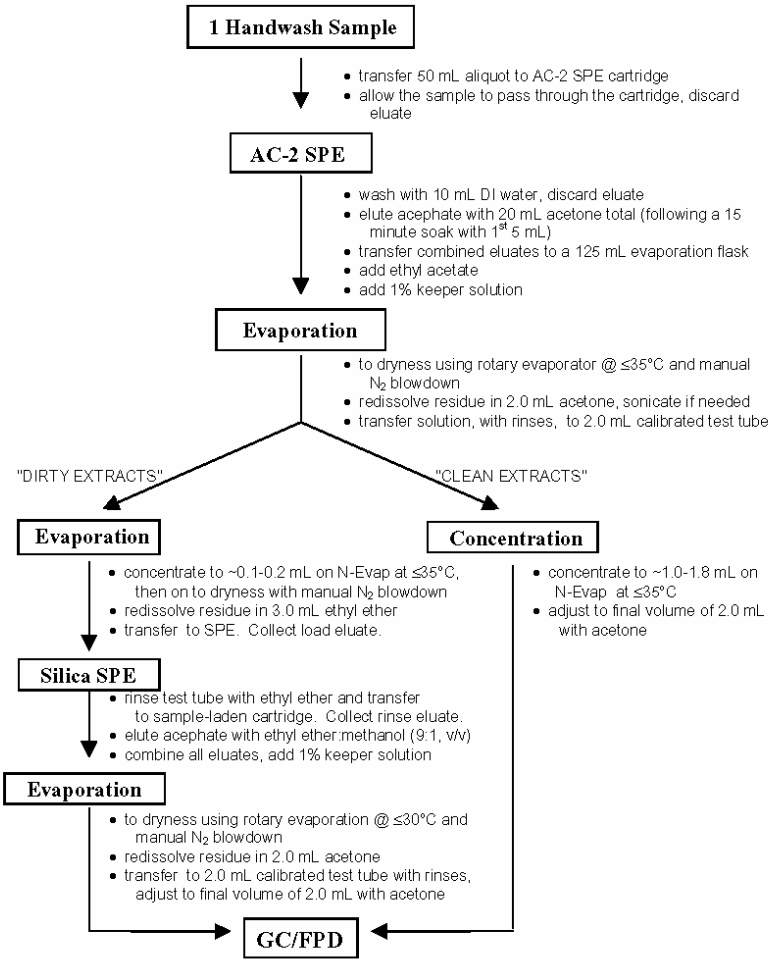
1. *Analytical Method for the Determination of Acephate in/on Worker Exposure Sampling Media Used in Worker Exposure Studies,* Method No. CCRL-MTH-030, dated May 23, 2001, CCRL (Central California Research Laboratories, Inc.), Fresno, California.

Method Author: Gary L. Westberg

**APPENDIX I**

Analysis Flowchart

**ANALYSIS FLOWCHART**



**APPENDIX II**

Quality Control for SPE Cartridges

Quality Control for SPE Cartridges

AC-2 SPE Cartridges

Add 50 µL of a standard solution @ 10 µg/mL (in acetone) to a 125 mL evaporation flask. Add 0.2 mL of 1% keeper solution and evaporate to dryness using manual nitrogen blow down. Add 50 mL DI water. Follow Steps 8.0.1. through 8.0.9. of the procedure. Redissolve the residue in 5.0 mL of acetone and transfer directly, without rinses, to a 13 × 100 mm test tube. Submit to GC analysis. Final concentration is 0.1 µg acephate/mL.

Silica SPE Cartridges

Add 50 µL of a standard solution @ 10 µg/mL (in acetone) to a 13 × 100 mm test tube. Add 0.2 mL of 1% keeper solution and evaporate to dryness using manual nitrogen blow down. Add 3.0 mL ethyl ether. Vortex mix. Follow Steps 9.0.1. through 9.0.7. of the procedure. Redissolve the residue in 5.0 mL of acetone and transfer directly, without rinses, to a 13 × 100 mm test tube. Submit to GC analysis. Final concentration is 0.1 µg acephate/mL.

**Determination of Acephate in OVS Air Sampling Tubes**

# DETERMINATION OF

# ACEPHATE IN OVS AIR SAMPLING TUBES

**Analytical Method No.: AHETF-AM-004**

Agricultural Handlers Exposure Task Force
(AHETF)

Morse Laboratories, Inc.
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TE 916/481-3141

Method Prepared by:
Gary L. Westberg

Date Issued:
February 26, 2003

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**DETERMINATION OF ACEPHATE IN OVS AIR SAMPLING TUBES**

## 1.0 PRINCIPLE

The contents of each OVS air sampling tube (referred to as "sorbent tube" throughout the rest of this procedure) are divided into two separate samples, each representing a specific section of the tube (front or rear). A provision is made, whereby both sections may be analyzed together as one sample. See Section 6.0 for further discussion on sample description.

Acephate is extracted from each sorbent tube section with acetone using sonication. An aliquot of the extract is evaporated to dryness, reconstituted in acetone, then submitted to gas chromatographic analysis employing flame photometric detection (FPD). The method does provide for an optional Silica SPE purification step which can be used on samples requiring additional cleanup. The limit of quantitation (LOQ), using the approach which combines both sections of the sorbent tube into a single analysis, is 0.01 µg/tube.

## 2.0 EQUIVALENCE STATEMENT

During the conduct of this analysis, comparable apparatus, solvents, glassware, and techniques (such as sample extract evaporation) may be substituted for those described in this method, except where specifically stated. In the event a substituted piece of equipment or technique is used, its use will be documented in the study records.

## 3.0 APPARATUS AND EQUIPMENT

Assorted laboratory glassware:

| | |
|---|---|
| Balances: | Analytical balance capable of weighing to ±0.1 mg |
| | Top-loading balance capable of weighing to ±0.1 g |
| Evaporator: | Rotary evaporator equipped with a Dewar condenser (Buchler Instruments, Labconco, Lenexa, KS) |
| | N-Evap Laboratory Sample Evaporator, Model 115, attached to a nitrogen source (Organomation Associates, South Berlin, MA) |
| Evaporation flasks: | Glass, round or flat bottom, 125 mL |
| Forceps: | Dissecting, curved, 117 mm |

| | |
|---|---|
| Gas chromatograph: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |
| GC column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |
| GC guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica (Manufacturer: Restek) |
| GC inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™. |
| Microliter syringes: | Various sizes (Hamilton Co., Reno, NV) |
| Pasteur pipets: | Glass, 9 inch and 5¾ inch, disposable |
| Pipets: | Glass, graduated, serological; various sizes |
| Pipets: | Glass, volumetric, class A; various sizes |
| Q-tips®: | 100% cotton swabs with wood sticks (Chesebrough-Pond's USA CO., Greenwich, CT) |
| Solid Phase Extraction Apparatus: | Vac Elut SPS 24 (Varian Analytical Instruments, Sunnyvale, CA) |
| Test (culture) tubes: | Glass, 13×100 mm and 16×150 mm |
| Ultrasonic bath: | Branson Model 2210 ultrasonic bath (VWR Scientific, Bridgeport, NJ) |
| Vials: | Glass, 15 mL with Teflon® lined cap (Supelco, Supelco Park Bellefonte, PA) |
| Volumetric flasks: | Glass; 250, 100, 50 and 25 mL |
| Vortex mixer: | Vortexer 2 (VWR Scientific, Bridgeport, NJ) |

## 4.0 REAGENTS AND MATERIALS

| | |
|---|---|
| Acetone: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |

| | |
|---|---|
| 1-Decanol: | "Baker" (J.T. Baker Chemical Company, Phillipsburg, NJ) |
| Ethyl ether: | AR®, analytical reagent (Mallinckrodt Baker, Inc., Paris, Kentucky) |
| Methanol: | OmniSolv®, high purity (EM Science, Gibbstown, NJ) |
| Solid phase extraction tubes: | Silica Bond Elut LRC®, 10cc/500mg (Varian Sample Preparation Products, Harbor City, CA) |
| Sorbent tubes: | Tube, OVS, XAD-2; catalog number 226-30-16A (SKC, Inc., Eighty Four, PA) |

4.1 Reagents and Materials to be Prepared (including typical preparation instructions)

4.1.1 *1% (w/v) keeper solution:* Place 1.0 g 1-decanol in a 100 mL volumetric flask. Fill to mark with acetone. Mix well.

4.1.2 *Silica SPE elution mixture [ethyl ether:methanol (9:1, v/v)]:* To a 250 mL volumetric flask, add 25 mL of methanol. Fill to mark with ethyl ether. Mix well. Prepare daily. Sufficient for approximately 20 samples.

**5.0 STANDARD PREPARATION**

5.1 Stock Standard Solution

Typically, 250 mg (corrected for purity) of acephate analytical standard is accurately weighed, quantitatively transferred to a 25 mL volumetric flask, and brought to volume with acetone to make a stock standard solution having a concentration of 10,000 µg/mL. This solution is to be stored at 1 to 8°C when not in use.

5.2 Fortification Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions are stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

1000 µg/mL: Transfer 2.5 mL of 10,000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

100 µg/mL: Transfer 2.5 mL of 1000 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

10 µg/mL: Transfer 2.5 mL of 100 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

1.0 µg/mL: Transfer 2.5 mL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.1 µg/mL: Transfer 2.5 mL of 1.0 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

5.3 GC (Calibration) Standard Solutions

Typically the following concentrations of acephate standard solution are prepared. All solutions prepared in this section are to be stored at 1 to 8°C when not in use. Use a microliter syringe to dispense volumes ≤500 µL.

0.004 µg/mL: Transfer 40 µL of 10 µg/mL standard solution to a 100 mL volumetric flask. Bring to volume in acetone. Mix well.

0.01 µg/mL: Transfer 50 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

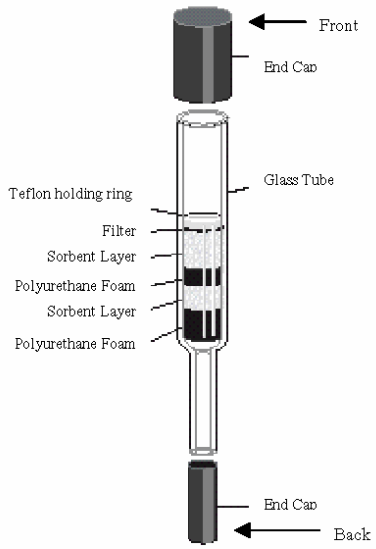
0.025 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 50 mL volumetric flask. Bring to volume in acetone. Mix well.

0.05 µg/mL: Transfer 125 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

0.10 µg/mL: Transfer 250 µL of 10 µg/mL standard solution to a 25 mL volumetric flask. Bring to volume in acetone. Mix well.

## 6.0 SAMPLE DESCRIPTION

The contents of each OVS sampling tube are divided into two separate samples. The front section, which consists of a Teflon® retainer ring, a glass fiber filter, 270 mg of XAD-2 sorbent and a foam separator plug represents one sample while the rear or back-up section, consisting of 140 mg XAD-2 sorbent and a foam retention plug, represents the second sample. A provision is made in the method to analyze both sorbent sections together as one sample for worker sorbent tubes. See diagram below:




**7.0 SAMPLE FORTIFICATION**

The "front section" (consisting of 270 mg XAD-2 sorbent) of the control sorbent tubes will be used to prepare fortified control samples. The sample is fortified by applying the appropriate amount of compound using a syringe. Fortify with a volume of fortification solution no greater than 50 µL. Allow solvent to evaporate for approximately 10 minutes prior to extraction.

**8.0 SAMPLE EXTRACTION**

8.1 Sample Preparation

8.1.1 Sections analyzed separately

1. If applicable, fortify samples at this time. Fortify the sorbent tube by passing the needle of a syringe, containing the appropriate amount of fortification solution (e.g., a 0.01 µg fortification is obtained by adding 10 µL of a 1.0 µg/mL solution), through the glass fiber filter and injecting the contents into the sorbent contained in the front section of the tube.

2. Using forceps, transfer the Teflon retainer ring, the glass fiber filter, and the XAD-2 sorbent from the front section of the tube to a glass vial (approximately 15 mL) labeled "front".

3. Wet a cotton swab (e.g. Q-Tips) in acetone, shake out the excess solvent and, ensuring that the solvent does not wet the foam separator plug, wash the inside of the glass sorbent tube to remove any acephate residue and assist in removal of residual resin deposited on the glass. Repeat this step with a second cotton swab, then allow the tube to dry. Transfer any remaining XAD-2 resin beads into the "front" sample.

4. Using a pair of scissors, cut wooden sticks just below cotton swab. Discard the sticks and transfer the swabs to the "front" sample.

5. Using the same forceps, transfer the foam separator plug from the front section of the tube to the "front" sample.

6. In a similar fashion, transfer the XAD-2 sorbent and foam plug from the rear section of the tube to a glass vial (approximately 15 mL) labeled "back".

7. Place the empty sorbent tube on top of the "front" vial and pass 2 × 5.0 mL acetone through the tube, rinsing the walls of the tube while doing so. The total volume of the extraction solvent is 10.0 mL. Tightly cap the vial.

8. Add 10.0 mL of acetone to the "back" sample. Tightly cap the vial. Proceed to Section 8.2.

8.1.2 Sections combined for analysis

Use the following procedure if both sections of the sorbent tube are to be combined and analyzed as a single sample.

1. If applicable, fortify samples at this time. Fortify the sorbent tube by passing the needle of a syringe, containing the appropriate amount of fortification solution (e.g., a 0.01 µg fortification is obtained by adding 10 µL of a 1.0 µg/mL solution), through the glass fiber filter and injecting the contents into the sorbent contained in the front section of the tube.

2. Using forceps, transfer the entire contents of the sorbent tube into a glass vial (approximately 15 mL). The vial thus contains the Teflon retainer ring, glass fiber filter, both XAD-2 sorbent packings and both foam plugs.

3. Place the empty sorbent tube on top of the vial and pass 2 × 5.0 mL acetone through the tube, rinsing the walls of the tube while doing so. The total volume of the extraction solvent is 10.0 mL. Tightly cap the vial. Proceed to Section 8.2.

8.2 Sample Extraction

**From this point on, individual sections (front or back) and combined sections are processed identically.**

1. Cap the vial tightly and sonicate for 2 hours.

2. Following extraction, allow the solids to settle.

3. Remove a 4.0 mL aliquot and transfer to a 13 × 100 mL screw-cap culture tube.

   **Note:** A retain aliquot of 4.0 mL is transferred to a 13 × 100 mL screw-cap culture tube and stored at <10°C (refrigerator or freezer) for future use, especially if SPE cleanup is required.

4. Add 0.2 mL keeper solution (See Section 4.0), vortex mix and evaporate to ~0.1-0.2 mL using an N-EVAP at ≤35°C. Manually evaporate to dryness with nitrogen, add 0.50 mL of acetone and sonicate for ~30 seconds. Transfer to a 2 mL autosampler vial. Submit for GC analysis. Resulting concentration is 1 mL = 0.8 sample.

   **Note:** A "sample" represents either a whole sorbent tube (both sections combined) or an individual section of the sorbent tube, depending on which extraction approach was taken.

5. If the extract appears to be too "dirty" or the resulting chromatogram indicates the need for extract cleanup, proceed to the silica SPE cartridge cleanup which follows. If it has been determined by analysis that cleanup is required, the retain aliquot from Step 3 must be processed through the SPE cartridge cleanup.

5.1 Add 0.2 mL keeper solution (See Section 4.0) to the retain aliquot, vortex mix and evaporate to ~0.1-0.2 mL using an N-EVAP at ≤35°C. Manually evaporate to dryness with nitrogen.

5.2 Redissolve the residue in 3.0 mL ethyl ether. Sonicate to dissolve/dislodge all residue.

5.3 Proceed to silica SPE cartridge cleanup (Section 9.0).

## 9.0 SILICA SPE CARTRIDGE CLEANUP

**Note:** Check or calibrate the SPE cartridges prior to use in order to ensure optimum method performance. In general, check one cartridge per lot number per box. This assessment should be conducted well in advance of needing the cartridges for sample analysis. Recovery of >90% is desired to ensure that a box of cartridges is suitable for use. The analyses are conducted on a reagent spike basis.

See Appendix II for detailed instructions on assessment of the SPE cartridges.

**Procedure:**

1. Set up Vac Elut system and support apparatus and proceed with the silica SPE cleanup. In general, set the vacuum to produce a flow rate of approximately 2-3 distinct drops/second (not continuous flow) for all elutions.

2. Condition a 500 mg silica SPE cartridge by passing 5 mL of ethyl ether through the cartridge. Do not let the cartridge go to dryness after conditioning. (Stop elution when conditioning solvent reaches top of frit.) Discard eluate.

3. Place a 16 × 150 mm test tube under the SPE cartridge. Collect all eluates from the following load and elution steps.

4. Pass the ethyl ether extract (3 mL) from Step 8.2.5.2 through the SPE cartridge. Stop elution when the solvent reaches the top of the frit. Collect eluate.

5. Wash the test tube from Step 4 one time with 3 mL of ethyl ether. Swirl to rinse the walls of the tube and pass the rinse through the sample-laden cartridge. Stop elution when solvent reaches top of frit. Collect eluate.

6. Elute the acephate with 3 × 5 mL portions of ethyl ether:methanol (9:1, v/v) solution. Each 5 mL portion is used to rinse the test tube from Step 4 (using sonication, if necessary) prior to passing through the cartridge. Collect all eluates. Quantitatively transfer the combined eluates to a 125 mL evaporation flask, using 2 mL of ethyl ether:methanol (9:1, v/v) solution to aid in the transfer.

7. Add 0.2 mL of 1% keeper solution to the eluate and concentrate to ~0.1-0.2 mL using a rotary evaporator set at ≤30°C, then on to dryness, with care, using manual nitrogen blowdown.

8. Redissolve the residue in 2.0 mL of acetone. Quantitatively transfer extract, to a 13×100 mm screw-cap culture tube.

9. Concentrate to 0.1-0.2 mL using an N-Evap evaporator set at ≤35°C, then on to dryness, with care, using manual nitrogen blowdown. Redissolve residue in 0.50 mL of acetone. Sonicate to dissolve/dislodge all residue. Transfer to a 2 mL autosampler vial.

10. Submit to GC analysis. Resulting concentration is 1 mL = 0.8 sample.

## 10.0 GAS CHROMATOGRAPHIC ANALYSIS

**Note:** The column and conditions stated in the method have proved to have been satisfactory for the matrix being analyzed. The specific column packing/coating, carrier gas, column temperature and flow rate listed are typical conditions for this analysis. Specific conditions used will be noted on each chromatographic run and will not otherwise be documented.

### 10.1 Operating Conditions

| | |
|---|---|
| Instrument: | Hewlett-Packard (HP) 6890 gas chromatograph equipped with a flame photometric detector in the phosphorous mode, an HP6890 Autosampler and an HPG2070AA ChemStation. |
| Inlet design: | "Purged/packed" recommended. "Capillary split-splitless" **is not** recommended. When set up to simulate a purged packed inlet, a capillary split-splitless inlet is often operated with the purge valve off over the course of the injection which, with megabore columns, tends to cause non-reproducible injections as well as non-linearity. |
| Column: | 30 M × 0.53 mm i.d. fused silica column crossbonded with 1.0 µm film thickness Rtx-200 |
| Guard column: | 3.0 M × 0.53 mm i.d. non-polar dimethyl deactivated fused silica |
| Inlet liner: | Open-top Uniliner® sleeve packed with Carbo Frit™ |
| Injection volume: | 2 µL |
| Temperatures: | Injector: 250°C<br>Detector: 200°C<br>Column: Initial: 130°C<br>Rate : 20°C/min.<br>Final: 230°C |

| | |
|---|---|
| Helium carrier gas flow: | 10 mL/min. |
| Helium make-up gas flow: | 80 mL/min. |
| Retention time: | ~3.7 minutes |

10.2 Sample Analysis

Prepare a five-point standard curve by injecting constant volumes of acephate standard solutions. Use constant volume injections for sample extracts as well. Sample responses not bracketed by the standard curve require dilution and reinjection. Inject a curve check standard every 3-4 sample injections.

## 11.0 CALCULATIONS

Calculations for instrumental analysis are conducted using a validated software application to create a standard curve based on linear regression. The regression functions are used to calculate a best fit line (from a set of standard concentrations in µg/mL versus peak response)
and to determine concentrations of the analyte found during sample analysis from the calculated best fit line.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

| | | |
|---|---|---|
| y | = | peak response |
| x | = | µg/mL found for peak of interest |
| m | = | slope |
| b | = | y-intercept |

Depending on the extraction approach taken, the results are reported accordingly. Thus if the contents of only one section of the sorbent tube are extracted, the results are expressed in **µg/section.** If the contents of the entire sorbent tube are extracted, the results are expressed in **µg/tube**. In either case, the calculations are identical except for the value entered for the amount of sample taken through the extraction process.

If the sections of the sorbent tube are analyzed separately, a calculation is provided which combines the two results into a single µg/tube value.

The calculations for µg/section and/or µg/tube found and percent recovery (for fortified samples) are:

1. The amount of acephate found (in µg/section or µg/tube) in the sample is calculated according to one of the following equations, as applicable:

$$\mu g/section = \mu g/mL\ found \times \frac{final\ vol.(mL)}{one(1)\ section} \times \frac{mL\ solv.}{mL\ aliq.} \times GC\ dilution\ \ factor$$

$$\mu g/tube = \mu g/mL\ found \times \frac{final\ vol.(mL)}{one(1)\ sorbent\ tube} \times \frac{mL\ solv.}{mL\ aliq.} \times GC\ dilution\ \ factor$$

where:

| | | |
|---|---|---|
| µg/section | = | total µg of analyte present in the section of sorbent tube analyzed |
| µg/tube | = | total µg of analyte present in the whole sorbent tube |
| µg/mL found | = | µg/mL of analyte found as determined by the analysis |
| 1 (one) section | = | amount of sample taken through the extraction process in the case of individual section analysis |
| 1 (one) sorbent tube | = | amount of sample taken through the extraction process in the case of combined section analysis |
| mL solv. | = | extraction solvent volume (10 mL) |
| mL aliq. | = | volume of sample extract taken from initial extraction (4 mL) |
| mL final volume | = | volume of final extract submitted to instrumentation (0.5 mL) |
| GC dil. factor | = | dilution of sample extract required to produce an analyte response bracketed by standard |

2. If the sections of the sorbent tube are analyzed separately, the µg/tube value is computed using the following equation:

$$\mu g/tube = \mu g/front\ section + \mu g/back\ section$$

3. The percent recovery for fortified control samples is calculated as follows:

$$\%\ Recovery = \frac{\mu g/sample\ found\ in\ fortified\ control - \mu g/sample\ found\ in\ control}{\mu g/sample\ added} \times 100$$

where:

sample = section or tube, as appropriate

## 12.0 REFERENCE

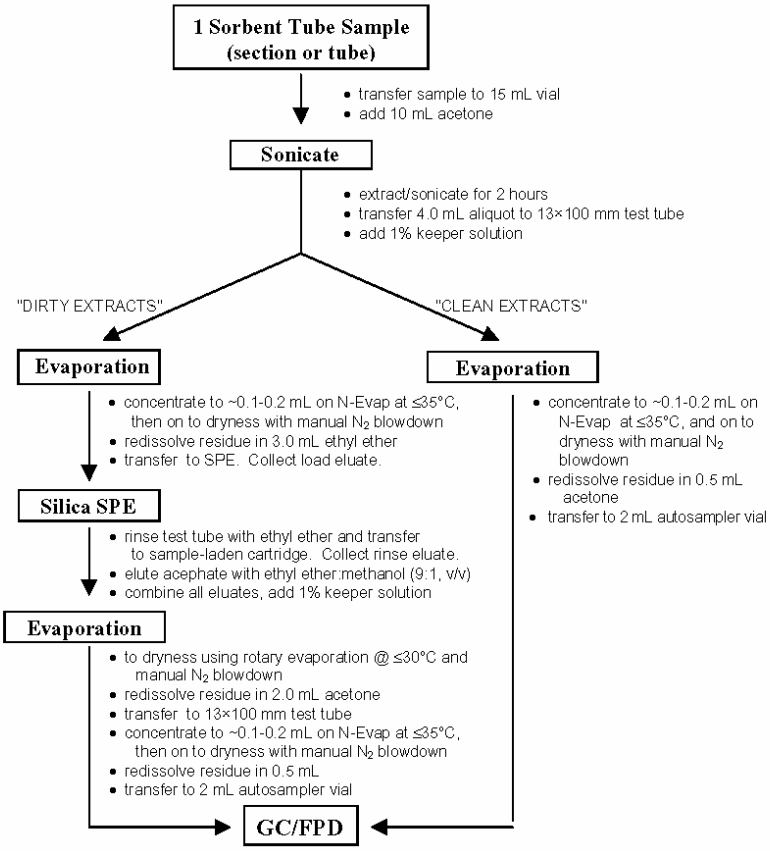
1. *Analytical Method for the Determination of Acephate in/on Worker Exposure Sampling Media Used in Worker Exposure Studies,* Method No. CCRL-MTH-030, dated May 23, 2001, CCRL (Central California Research Laboratories, Inc.), Fresno, California.

Method Author: Gary L. Westberg

**APPENDIX I**

Analysis Flowchart

**ANALYSIS FLOWCHART**



**APPENDIX II**

Quality Control for SPE Cartridges

Quality Control for SPE Cartridges

Silica SPE Cartridges

Add 50 µL of a standard solution @ 10 µg/mL (in acetone) to a 13 × 100 mm test tube. Add 0.2 mL of 1% keeper solution and evaporate to dryness using manual nitrogen blow down. Add 3.0 mL ethyl ether. Vortex mix. Follow Steps 9.0.1. through 9.0.7. of the procedure. Redissolve the residue in 5.0 mL of acetone and transfer directly, without rinses, to a 13 × 100 mm test tube. Submit to GC analysis. Final concentration is 0.1 µg acephate/mL.

## Appendix C. Sample Extraction Spreadsheet

Residue Results
Analyte: Acephate
Matrix: Inner dosimeters

| Sample Identification | Set # | Amt Samp sect | Fort Level ug/sect | Extraction Date | Analysis Date | mL solv | mL aliq | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/sect | reported ug/sect | ug/sect corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1055G cont.1 | 1a | 1 | | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.5 | | |
| 1055G fort.cont.1 | 1a | 1 | 0.5 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 87.4 | | 0.0080 | 0.4 | 0.4 | 0.4 | 80.0 |
| 1055G fort.cont.2 | 1a | 1 | 0.5 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 83.5 | | 0.0076 | 0.38 | 0.4 | 0.4 | 76.0 |
| 1055G fort.cont.3 | 1a | 1 | 0.5 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 89.6 | | 0.0083 | 0.415 | 0.4 | 0.4 | 83.0 |
| 1055G fort.cont.4 | 1a | 1 | 1.0 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 221.9 | | 0.0239 | 1.195 | 1.2 | 1.2 | 120 |
| 1055G fort.cont.5 | 1a | 1 | 1.0 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 212.3 | | 0.0227 | 1.135 | 1.1 | 1.1 | 114 |
| 1055G fort.cont.6 | 1a | 1 | 1.0 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 1 | 215.6 | | 0.0231 | 1.155 | 1.2 | 1.2 | 116 |
| 1055G fort.cont.7 | 1a | 1 | 10000 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 4000 | 438.5 | | 0.0494 | 9880 | 9880 | 9880 | 98.8 |
| 1055G fort.cont.8 | 1a | 1 | 10000 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 4000 | 445.5 | | 0.0502 | 10040 | 10000 | 10000 | 100 |
| 1055G fort.cont.9 | 1a | 1 | 10000 | 05/07/03 | 05/21/03 | 2000 | 80 | 2.0 | 2 | 4000 | 443.3 | | 0.0499 | 9980 | 9980 | 9980 | 99.8 |
| std.curve 0.025 ug/mL | 1a | | | | 05/21/03 | | | | | | 233.3 | 0.77 | | | | | |
| std.curve 0.10 ug/mL | 1a | | | | 05/21/03 | | | | | | 890.6 | 2.57 | | | | | |
| std.curve 0.05 ug/mL | 1a | | | | 05/21/03 | | | | | | 457.4 | 3.14 | | | | | |
| std.curve 0.025 ug/mL | 1a | | | | 05/21/03 | | | | | | 228.5 | -1.49 | | | | | |
| std.curve 0.01 ug/mL | 1a | | | | 05/21/03 | | | | | | 102.6 | -1.94 | | | | | |
| std.curve 0.005 ug/mL | 1a | | | | 05/21/03 | | | | | | 53.3 | -19.95 | | | | | |
| std.curve 0.05 ug/mL | 1a | | | | 05/21/03 | | | | | | 451.9 | 1.85 | | | | | |
| std.curve 0.10 ug/mL | 1a | | | | 05/21/03 | | | | | | 852.4 | -1.93 | | | | | |
| std.curve 0.10 ug/mL | 1a | | | | 05/21/03 | | | | | | 853.9 | -1.76 | | | | | |

Lower case letter designation with set number indicates retain extract analysis.

Residue Results
Analyte: Acephate
Matrix: Inner dosimeters

| Sample Identification | Set # | Amt Samp sect | Fort Level ug/sect | Extraction Date | Analysis Date | mL solv | mL aliq | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/sect | reported ug/sect | ug/sect corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1055G cont.3 | 3 | 1 | | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.5 | | |
| 1055G fort.cont.19 | 3 | 1 | 0.5 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 92.5 | | 0.0081 | 0.405 | 0.4 | 0.4 | 81.0 |
| 1055G fort.cont.20 | 3 | 1 | 0.5 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 86.8 | | 0.0074 | 0.37 | 0.4 | 0.4 | 74.0 |
| 1055G fort.cont.21 | 3 | 1 | 0.5 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 89.0 | | 0.0077 | 0.385 | 0.4 | 0.4 | 77.0 |
| 1055G fort.cont.22 | 3 | 1 | 1.0 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 178.8 | | 0.0177 | 0.885 | 0.9 | 0.9 | 88.5 |
| 1055G fort.cont.23 | 3 | 1 | 1.0 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 189.6 | | 0.0189 | 0.945 | 0.9 | 0.9 | 94.5 |
| 1055G fort.cont.24 | 3 | 1 | 1.0 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 1 | 181.5 | | 0.0180 | 0.9 | 0.9 | 0.9 | 90.0 |
| 1055G fort.cont.25 | 3 | 1 | 10000 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 4000 | 444.8 | | 0.0473 | 9460 | 9460 | 9460 | 94.6 |
| 1055G fort.cont.26 | 3 | 1 | 10000 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 4000 | 442.6 | | 0.0471 | 9420 | 9420 | 9420 | 94.2 |
| 1055G fort.cont.27 | 3 | 1 | 10000 | 05/08/03 | 05/09/03 | 2000 | 80 | 2.0 | 2 | 4000 | 451.6 | | 0.0481 | 9620 | 9620 | 9620 | 96.2 |
| std.curve 0.025 ug/mL | 3 | | | | 05/09/03 | | | | | | 240.9 | -1.57 | | | | | |
| std.curve 0.10 ug/mL | 3 | | | | 05/09/03 | | | | | | 935.0 | 1.89 | | | | | |
| std.curve 0.05 ug/mL | 3 | | | | 05/09/03 | | | | | | 484.5 | 3.46 | | | | | |
| std.curve 0.01 ug/mL | 3 | | | | 05/09/03 | | | | | | 112.4 | 3.01 | | | | | |
| std.curve 0.005 ug/mL | 3 | | | | 05/09/03 | | | | | | 58.0 | -15.11 | | | | | |
| std.curve 0.025 ug/mL | 3 | | | | 05/09/03 | | | | | | 239.0 | -2.41 | | | | | |
| std.curve 0.05 ug/mL | 3 | | | | 05/09/03 | | | | | | 474.8 | 1.30 | | | | | |
| std.curve 0.10 ug/mL | 3 | | | | 05/09/03 | | | | | | 892.7 | -2.82 | | | | | |

Lower case letter designation with set number indicates retain extract analysis.

Residue Results
Analyte: Acephate
Matrix: Face/Neck Wipes

| Sample Identification | Set # | Amt Samp | Fort Level ug/samp | Extraction Date | Analysis Date | mL solv | mL aliq | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/samp | reported ug/samp | ug/samp corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1055C cont.2 | 2 | 1 | | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.5 | | |
| 1055C fort.cont.10 | 2 | 1 | 0.5 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 91.0 | | 0.0106 | 0.53 | 0.5 | 0.5 | 106 |
| 1055C fort.cont.11 | 2 | 1 | 0.5 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 91.6 | | 0.0106 | 0.53 | 0.5 | 0.5 | 106 |
| 1055C fort.cont.12 | 2 | 1 | 0.5 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 90.6 | | 0.0105 | 0.525 | 0.5 | 0.5 | 105 |
| 1055C fort.cont.13 | 2 | 1 | 1.0 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 134.2 | | 0.0166 | 0.83 | 0.8 | 0.8 | 83.0 |
| 1055C fort.cont.14 | 2 | 1 | 1.0 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 140.5 | | 0.0174 | 0.87 | 0.9 | 0.9 | 87.0 |
| 1055C fort.cont.15 | 2 | 1 | 1.0 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 1 | 134.2 | | 0.0166 | 0.83 | 0.8 | 0.8 | 83.0 |
| 1055C fort.cont.16 | 2 | 1 | 100 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 40 | 313.8 | | 0.0415 | 83 | 83.0 | 83.0 | 83.0 |
| 1055C fort.cont.17 | 2 | 1 | 100 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 40 | 301.2 | | 0.0397 | 79.4 | 79.4 | 79.4 | 79.4 |
| 1055C fort.cont.18 | 2 | 1 | 100 | 05/07/03 | 05/22/03 | 100 | 2.0 | 1.0 | 2 | 40 | 302.7 | | 0.0399 | 79.8 | 79.8 | 79.8 | 79.8 |
| std.curve 0.025 ug/mL | 2 | | | | 05/22/03 | | | | | | 219.4 | 13.51 | | | | | |
| std.curve 0.10 ug/mL | 2 | | | | 05/22/03 | | | | | | 780.2 | 6.19 | | | | | |
| std.curve 0.05 ug/mL | 2 | | | | 05/22/03 | | | | | | 383.6 | 2.32 | | | | | |
| std.curve 0.01 ug/mL | 2 | | | | 05/22/03 | | | | | | 89.0 | 2.83 | | | | | |
| std.curve 0.005 ug/mL | 2 | | | | 05/22/03 | | | | | | 47.7 | -8.95 | | | | | |
| std.curve 0.025 ug/mL | 2 | | | | 05/22/03 | | | | | | 201.7 | 3.68 | | | | | |
| std.curve 0.05 ug/mL | 2 | | | | 05/22/03 | | | | | | 366.6 | -2.40 | | | | | |
| std.curve 0.05 ug/mL | 2 | | | | 05/22/03 | | | | | | 331.4 | -12.16 | | | | | |
| std.curve 0.10 ug/mL | 2 | | | | 05/22/03 | | | | | | 705.2 | -4.22 | | | | | |

Residue Results
Analyte: Acephate
Matrix: Face/Neck Wipes

| Sample Identification | Set # | Amt Samp | Fort Level ug/samp | Extraction Date | Analysis Date | mL solv | mL aliq | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/samp | reported ug/samp | ug/samp corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1055C cont.4 | 4 | 1 | | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.5 | | |
| 1055C fort.cont.28 | 4 | 1 | 0.5 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 86.7 | | 0.0080 | 0.4 | 0.4 | 0.4 | 80.0 |
| 1055C fort.cont.29 | 4 | 1 | 0.5 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 80.6 | | 0.0073 | 0.365 | 0.4 | 0.4 | 73.0 |
| 1055C fort.cont.30 | 4 | 1 | 0.5 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 96.8 | | 0.0091 | 0.455 | 0.5 | 0.5 | 91.0 |
| 1055C fort.cont.31 | 4 | 1 | 1.0 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 160.6 | | 0.0163 | 0.815 | 0.8 | 0.8 | 81.5 |
| 1055C fort.cont.32 | 4 | 1 | 1.0 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 180.0 | | 0.0185 | 0.925 | 0.9 | 0.9 | 92.5 |
| 1055C fort.cont.33 | 4 | 1 | 1.0 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 1 | 171.2 | | 0.0175 | 0.875 | 0.9 | 0.9 | 87.5 |
| 1055C fort.cont.34 | 4 | 1 | 100 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 40 | 404.2 | | 0.0439 | 87.8 | 87.8 | 87.8 | 87.8 |
| 1055C fort.cont.35 | 4 | 1 | 100 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 40 | 407.2 | | 0.0442 | 88.4 | 88.4 | 88.4 | 88.4 |
| 1055C fort.cont.36 | 4 | 1 | 100 | 05/09/03 | 05/13/03 | 100 | 2.0 | 1.0 | 2 | 40 | 430.0 | | 0.0468 | 93.6 | 93.6 | 93.6 | 93.6 |
| std.curve 0.10 ug/mL | 4 | | | | 05/13/03 | | | | | | 874.8 | -2.87 | | | | | |
| std.curve 0.10 ug/mL | 4 | | | | 05/13/03 | | | | | | 896.6 | -0.41 | | | | | |
| std.curve 0.10 ug/mL | 4 | | | | 05/13/03 | | | | | | 914.9 | 1.66 | | | | | |
| std.curve 0.05 ug/mL | 4 | | | | 05/13/03 | | | | | | 475.6 | 3.92 | | | | | |
| std.curve 0.01 ug/mL | 4 | | | | 05/13/03 | | | | | | 106.1 | 1.55 | | | | | |
| std.curve 0.005 ug/mL | 4 | | | | 05/13/03 | | | | | | 51.4 | -20.70 | | | | | |
| std.curve 0.025 ug/mL | 4 | | | | 05/13/03 | | | | | | 237.1 | -0.09 | | | | | |
| std.curve 0.025 ug/mL | 4 | | | | 05/13/03 | | | | | | 240.4 | 1.40 | | | | | |
| std.curve 0.01 ug/mL | 4 | | | | 05/13/03 | | | | | | 104.0 | -0.82 | | | | | |
| std.curve 0.005 ug/mL | 4 | | | | 05/13/03 | | | | | | 51.7 | -20.00 | | | | | |
| std.curve 0.05 ug/mL | 4 | | | | 05/13/03 | | | | | | 469.7 | 2.59 | | | | | |

Residue Results
Analyte: Acephate
Matrix: Handwashes

| Sample Identification | Set # | Samp (HW) | Fort Level ug/samp | Extraction Date | Analysis Date | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/samp | reported ug/samp | ug/samp corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1058A cont.5 | 5 | 0.1 | | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.2 | | |
| 1058A fort.cont.37 | 5 | 0.1 | 0.2 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 85.3 | | 0.0081 | 0.162 | 0.2 | 0.2 | 81.0 |
| 1058A fort.cont.38 | 5 | 0.1 | 0.2 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 79.1 | | 0.0074 | 0.148 | 0.1 | 0.1 | 74.0 |
| 1058A fort.cont.39 | 5 | 0.1 | 0.2 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 82.8 | | 0.0078 | 0.156 | 0.2 | 0.2 | 78.0 |
| 1058A fort.cont.40 | 5 | 0.1 | 1.0 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 426.9 | | 0.0459 | 0.918 | 0.9 | 0.9 | 91.8 |
| 1058A fort.cont.41 | 5 | 0.1 | 1.0 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 411.3 | | 0.0442 | 0.884 | 0.9 | 0.9 | 88.4 |
| 1058A fort.cont.42 | 5 | 0.1 | 1.0 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1 | 396.3 | | 0.0425 | 0.85 | 0.8 | 0.9 | 85.0 |
| 1058A fort.cont.43 | 5 | 0.1 | 1000 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1000 | 417.6 | | 0.0449 | 898 | 898 | 898 | 89.8 |
| 1058A fort.cont.44 | 5 | 0.1 | 1000 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1000 | 453.4 | | 0.0489 | 978 | 978 | 978 | 97.8 |
| 1058A fort.cont.45 | 5 | 0.1 | 1000 | 05/12/03 | 05/15/03 | 2.0 | 2 | 1000 | 513.8 | | 0.0556 | 1112 | 1110 | 1110 | 111 |
| std.curve 0.025 ug/mL | 5 | | | | 05/15/03 | | | | 234.1 | -1.72 | | | | | |
| std.curve 0.025 ug/mL | 5 | | | | 05/15/03 | | | | 230.4 | -3.36 | | | | | |
| std.curve 0.10 ug/mL | 5 | | | | 05/15/03 | | | | 908.2 | -0.74 | | | | | |
| std.curve 0.05 ug/mL | 5 | | | | 05/15/03 | | | | 477.0 | 2.97 | | | | | |
| std.curve 0.025 ug/mL | 5 | | | | 05/15/03 | | | | 242.0 | 1.78 | | | | | |
| std.curve 0.01 ug/mL | 5 | | | | 05/15/03 | | | | 104.2 | 1.79 | | | | | |
| std.curve 0.005 ug/mL | 5 | | | | 05/15/03 | | | | 51.3 | -13.64 | | | | | |
| std.curve 0.025 ug/mL | 5 | | | | 05/15/03 | | | | 240.7 | 1.21 | | | | | |
| std.curve 0.01 ug/mL | 5 | | | | 05/15/03 | | | | 104.6 | 2.23 | | | | | |
| std.curve 0.10 ug/mL | 5 | | | | 05/15/03 | | | | 916.1 | 0.13 | | | | | |
| std.curve 0.005 ug/mL | 5 | | | | 05/15/03 | | | | 57.0 | -1.01 | | | | | |

Residue Results
Analyte: Acephate
Matrix: Handwashes

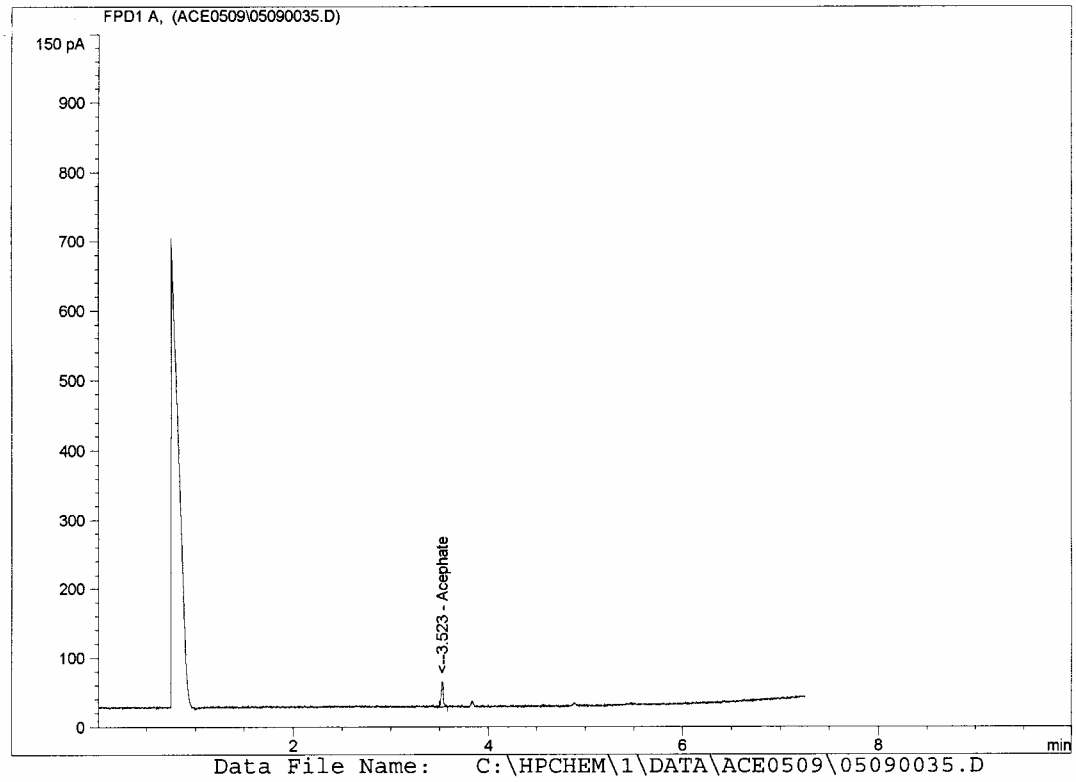
| Sample Identification | Set # | Samp (HW) | Fort Level ug/samp | Extraction Date | Analysis Date | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/samp | reported ug/samp | ug/samp corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1058A cont.9 | 9 | 0.1 | | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 0 | | 0.0000 | 0 | <0.2 | | |
| 1058A fort.cont.67 | 9 | 0.1 | 0.2 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 13125 | | 0.0089 | 0.178 | 0.2 | 0.2 | 89.0 |
| 1058A fort.cont.68 | 9 | 0.1 | 0.2 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 12257 | | 0.0084 | 0.168 | 0.2 | 0.2 | 84.0 |
| 1058A fort.cont.69 | 9 | 0.1 | 0.2 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 12512 | | 0.0086 | 0.172 | 0.2 | 0.2 | 86.0 |
| 1058A fort.cont.70 | 9 | 0.1 | 1.0 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 95581 | | 0.0522 | 1.044 | 1.0 | 1.0 | 104 |
| 1058A fort.cont.71 | 9 | 0.1 | 1.0 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 87248 | | 0.0479 | 0.958 | 1.0 | 1.0 | 95.8 |
| 1058A fort.cont.72 | 9 | 0.1 | 1.0 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1 | 87754 | | 0.0481 | 0.962 | 1.0 | 1.0 | 96.2 |
| 1058A fort.cont.73 | 9 | 0.1 | 1000 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1000 | 90303 | | 0.0495 | 990 | 990 | 990 | 99.0 |
| 1058A fort.cont.74 | 9 | 0.1 | 1000 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1000 | 85551 | | 0.0470 | 940 | 940 | 940 | 94.0 |
| 1058A fort.cont.75 | 9 | 0.1 | 1000 | 05/19/03 | 05/20/03 | 2.0 | 2 | 1000 | 86060 | | 0.0472 | 944 | 944 | 944 | 94.4 |
| std.curve 0.025 ug/mL | 9 | | | | 05/20/03 | | | | 44586 | 1.67 | | | | | |
| std.curve 0.10 ug/mL | 9 | | | | 05/20/03 | | | | 189983 | 1.87 | | | | | |
| std.curve 0.05 ug/mL | 9 | | | | 05/20/03 | | | | 91888 | 0.58 | | | | | |
| std.curve 0.025 ug/mL | 9 | | | | 05/20/03 | | | | 41622 | -4.56 | | | | | |
| std.curve 0.01 ug/mL | 9 | | | | 05/20/03 | | | | 17218 | 10.28 | | | | | |
| std.curve 0.005 ug/mL | 9 | | | | 05/20/03 | | | | 7281 | 16.10 | | | | | |
| std.curve 0.05 ug/mL | 9 | | | | 05/20/03 | | | | 89986 | -1.42 | | | | | |
| std.curve 0.025 ug/mL | 9 | | | | 05/20/03 | | | | 41115 | -5.63 | | | | | |
| std.curve 0.10 ug/mL | 9 | | | | 05/20/03 | | | | 183789 | -1.39 | | | | | |
| std.curve 0.05 ug/mL | 9 | | | | 05/20/03 | | | | 91784 | 0.47 | | | | | |

Residue Results
Analyte: Acephate
Matrix: OVS Tubes

| Sample Identification | Set # | Samp tube | Fort Level ug/tube | Extraction Date | Analysis Date | mL solv | mL aliq | Final Vol mL | Inj Vol uL | GC Dil Fact | Peak Resp | Back Calc of Std Pk Resp % Diff | ug/mL found | ug/tube | reported ug/tube | ug/tube corr. for cont. | % Rec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1058B cont.7 | 7 | 1 | | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 0.0 | | 0.0000 | 0 | <0.01 | | |
| 1058B fort.cont.55 | 7 | 1 | 0.01 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 62.0 | | 0.0059 | 0.007375 | 0.007 | 0.007 | 73.8 |
| 1058B fort.cont.56 | 7 | 1 | 0.01 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 72.9 | | 0.0070 | 0.00875 | 0.009 | 0.009 | 87.5 |
| 1058B fort.cont.57 | 7 | 1 | 0.01 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 78.9 | | 0.0076 | 0.0095 | 0.010 | 0.010 | 95.0 |
| 1058B fort.cont.58 | 7 | 1 | 50 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 452.4 | | 0.0456 | 45.6 | 45.6 | 45.6 | 91.2 |
| 1058B fort.cont.59 | 7 | 1 | 50 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 362.6 | | 0.0365 | 36.5 | 36.5 | 36.5 | 73.0 |
| 1058B fort.cont.60 | 7 | 1 | 50 | 05/13/03 | 05/14/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 444.0 | | 0.0448 | 44.8 | 44.8 | 44.8 | 89.6 |
| std.curve 0.025 ug/mL | 7 | | | | 05/14/03 | | | | | | 236.1 | -5.52 | | | | | |
| std.curve 0.025 ug/mL | 7 | | | | 05/14/03 | | | | | | 248.5 | -0.47 | | | | | |
| std.curve 0.01 ug/mL | 7 | | | | 05/14/03 | | | | | | 109.1 | 6.89 | | | | | |
| std.curve 0.10 ug/mL | 7 | | | | 05/14/03 | | | | | | 977.1 | -0.93 | | | | | |
| std.curve 0.05 ug/mL | 7 | | | | 05/14/03 | | | | | | 509.7 | 2.95 | | | | | |
| std.curve 0.025 ug/mL | 7 | | | | 05/14/03 | | | | | | 245.9 | -1.53 | | | | | |
| std.curve 0.01 ug/mL | 7 | | | | 05/14/03 | | | | | | 111.7 | 9.53 | | | | | |
| std.curve 0.004 ug/mL | 7 | | | | 05/14/03 | | | | | | 38.1 | -13.51 | | | | | |
| std.curve 0.10 ug/mL | 7 | | | | 05/14/03 | | | | | | 991.5 | 0.53 | | | | | |
| std.curve 0.004 ug/mL | 7 | | | | 05/14/03 | | | | | | 40.5 | -7.40 | | | | | |
| 1058B cont.8 | 8a | 1 | | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 1732 | | 0.0017 | 0.002125 | <0.01 | | |
| 1058B fort.cont.61 | 8a | 1 | 0.01 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 13438 | | 0.0078 | 0.00975 | 0.010 | 0.008 | 78.8 |
| 1058B fort.cont.62 | 8a | 1 | 0.01 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 14497 | | 0.0083 | 0.010375 | 0.010 | 0.008 | 78.8 |
| 1058B fort.cont.63 | 8a | 1 | 0.01 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 1 | 13447 | | 0.0078 | 0.00975 | 0.010 | 0.008 | 78.8 |
| 1058B fort.cont.64 | 8a | 1 | 50 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 81019 | | 0.0429 | 42.9 | 42.9 | 42.9 | 85.8 |
| 1058B fort.cont.65 | 8a | 1 | 50 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 79125 | | 0.0419 | 41.9 | 41.9 | 41.9 | 83.8 |
| 1058B fort.cont.66 | 8a | 1 | 50 | 05/14/03 | 05/19/03 | 10.0 | 4.0 | 0.50 | 2 | 800 | 80723 | | 0.0428 | 42.8 | 42.8 | 42.8 | 85.6 |
| std.curve 0.01 ug/mL | 8a | | | | 05/19/03 | | | | | | 19365 | 8.37 | | | | | |
| std.curve 0.025 ug/mL | 8a | | | | 05/19/03 | | | | | | 46735 | 0.31 | | | | | |
| std.curve 0.10 ug/mL | 8a | | | | 05/19/03 | | | | | | 192883 | 1.11 | | | | | |
| std.curve 0.05 ug/mL | 8a | | | | 05/19/03 | | | | | | 96219 | 1.64 | | | | | |
| std.curve 0.025 ug/mL | 8a | | | | 05/19/03 | | | | | | 44857 | -3.60 | | | | | |
| std.curve 0.01 ug/mL | 8a | | | | 05/19/03 | | | | | | 19132 | 7.16 | | | | | |
| std.curve 0.004 ug/mL | 8a | | | | 05/19/03 | | | | | | 6810 | 7.63 | | | | | |
| std.curve 0.05 ug/mL | 8a | | | | 05/19/03 | | | | | | 88935 | -5.94 | | | | | |

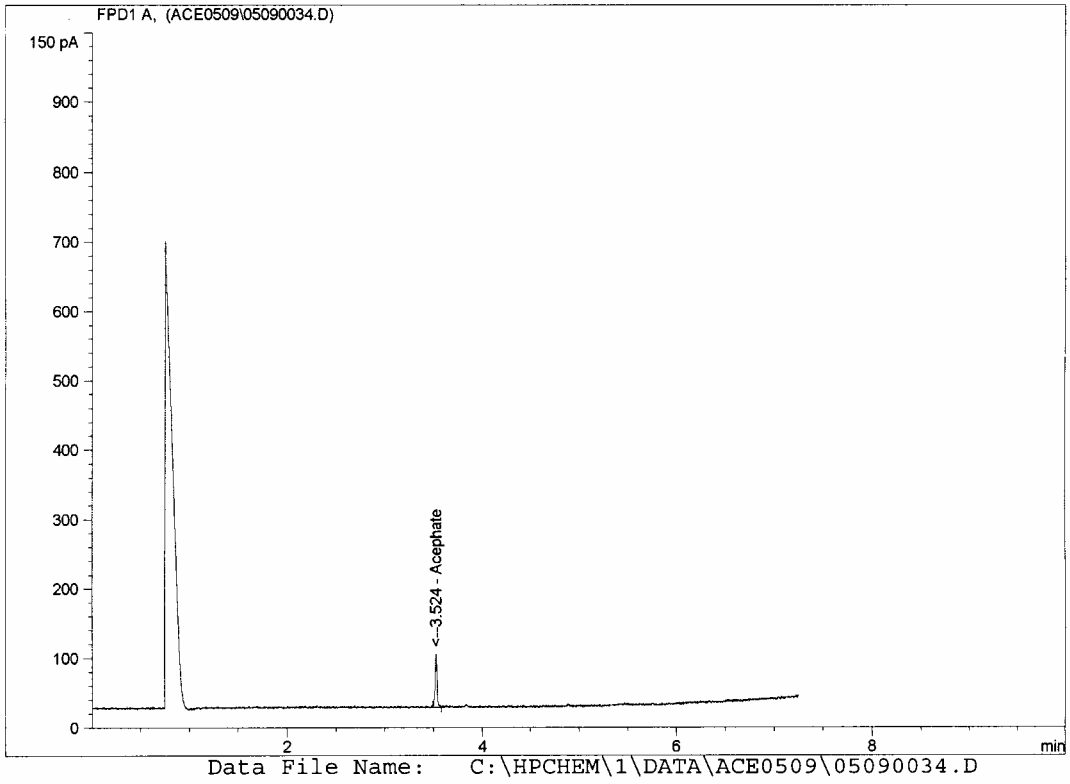
## Appendix D. Example Chromatograms and Standard Curve

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



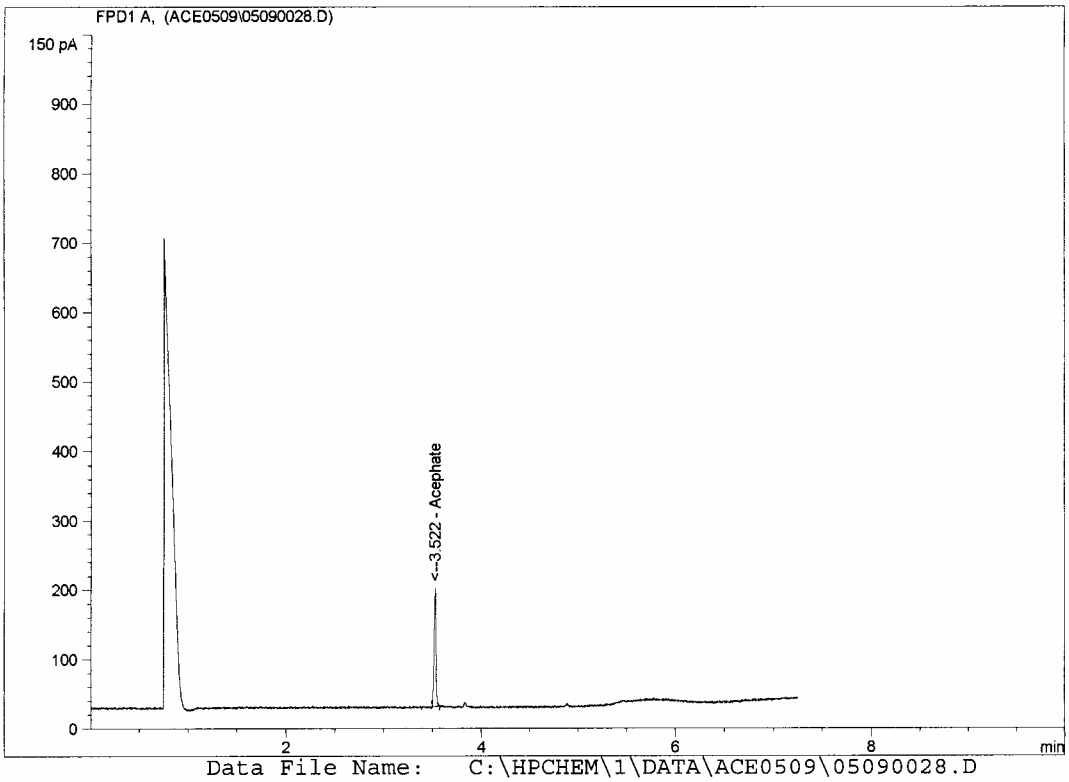

Acephate 0.005 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



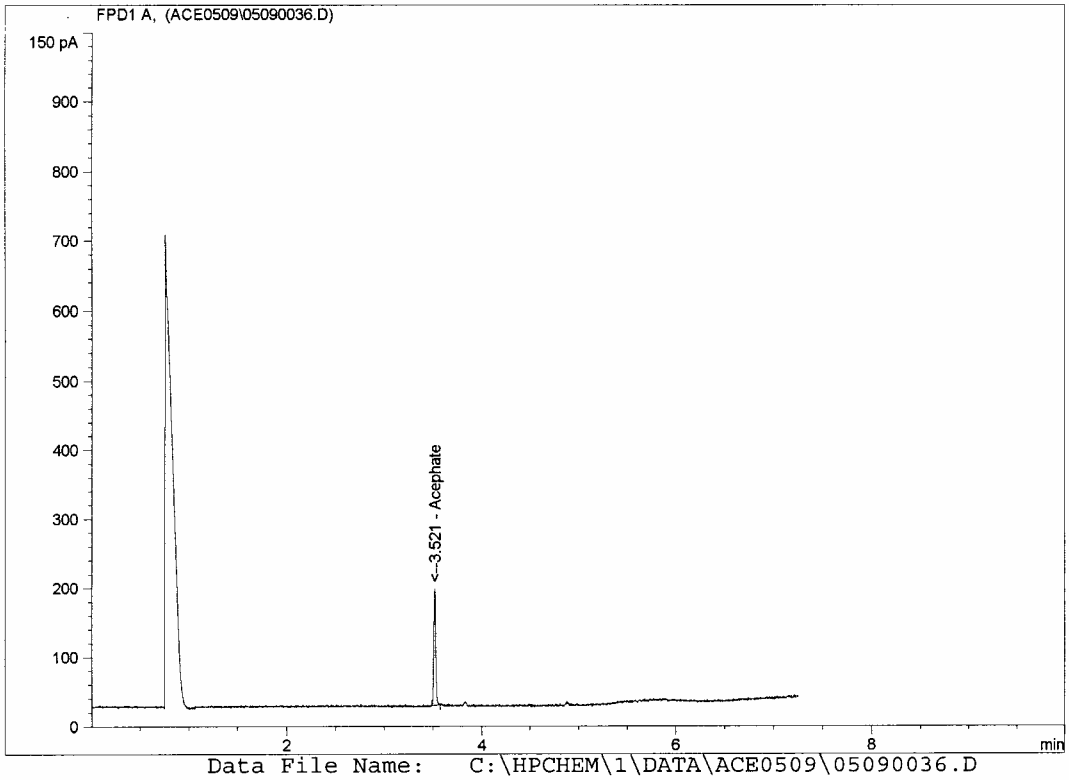

Acephate 0.01 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



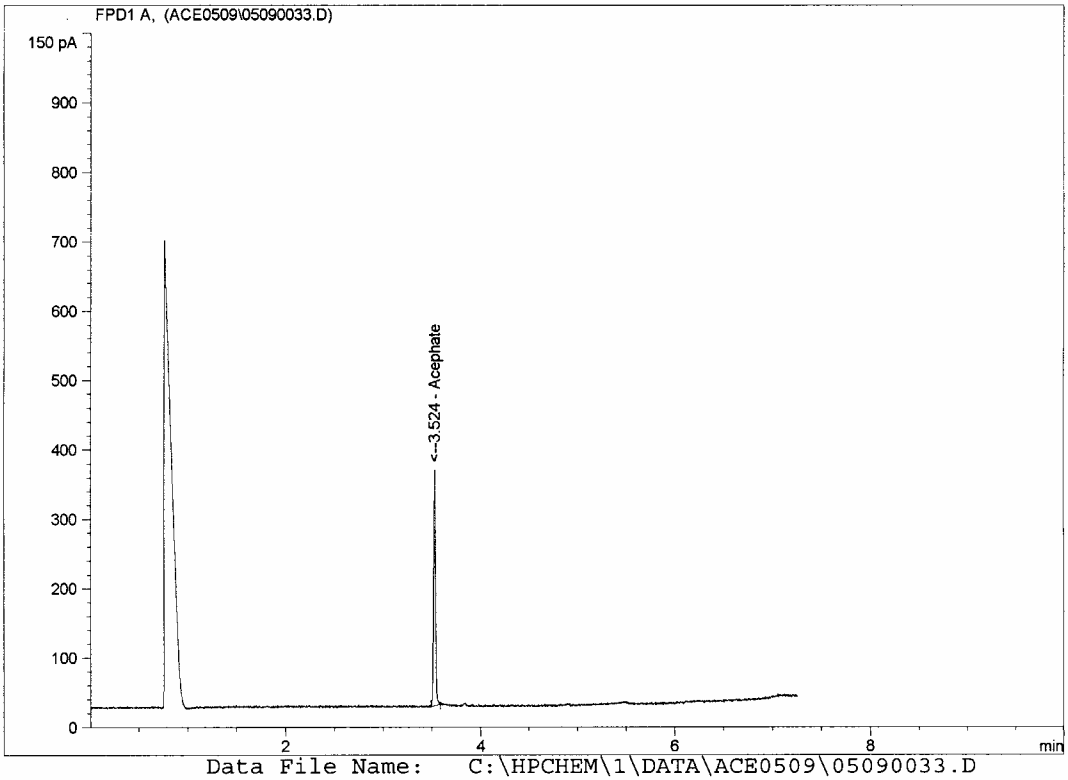

Acephate 0.025 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



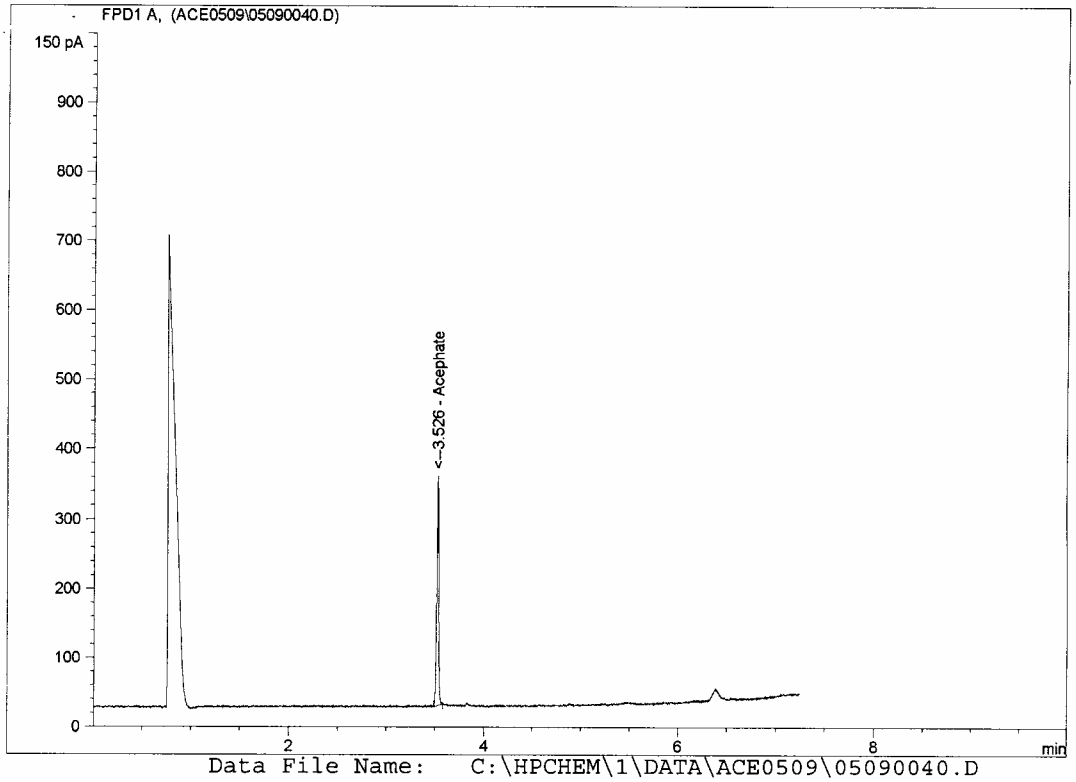

Acephate 0.025 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



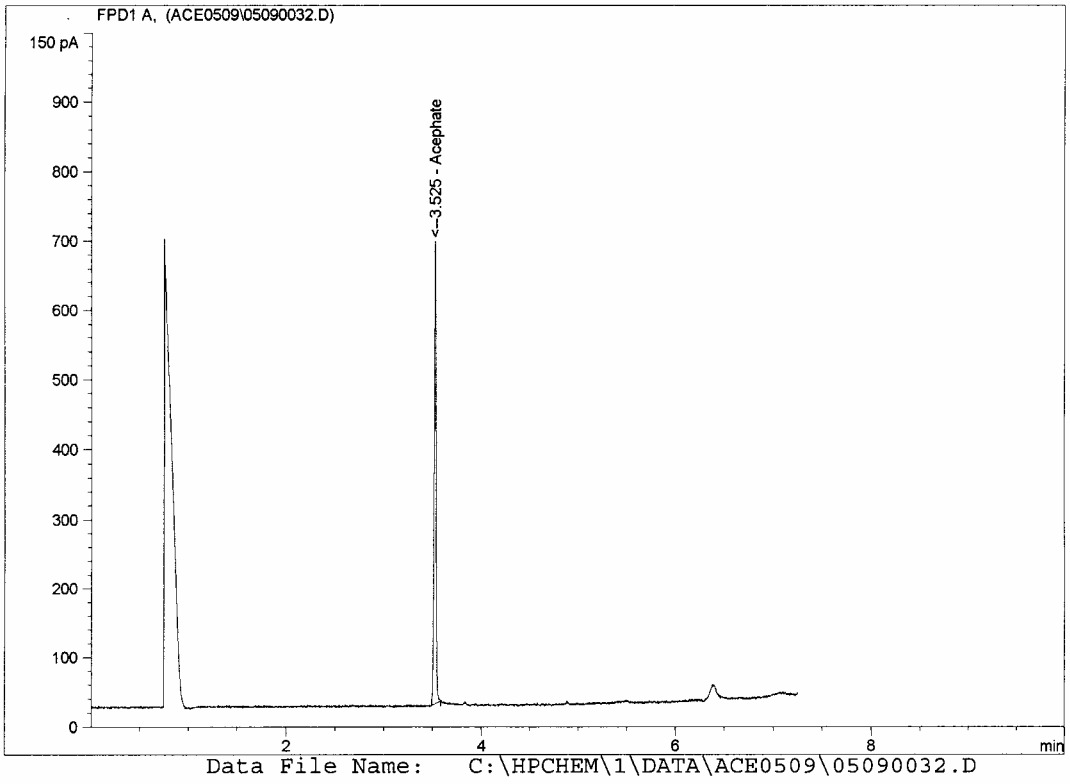

Acephate 0.05 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD,
SET #3



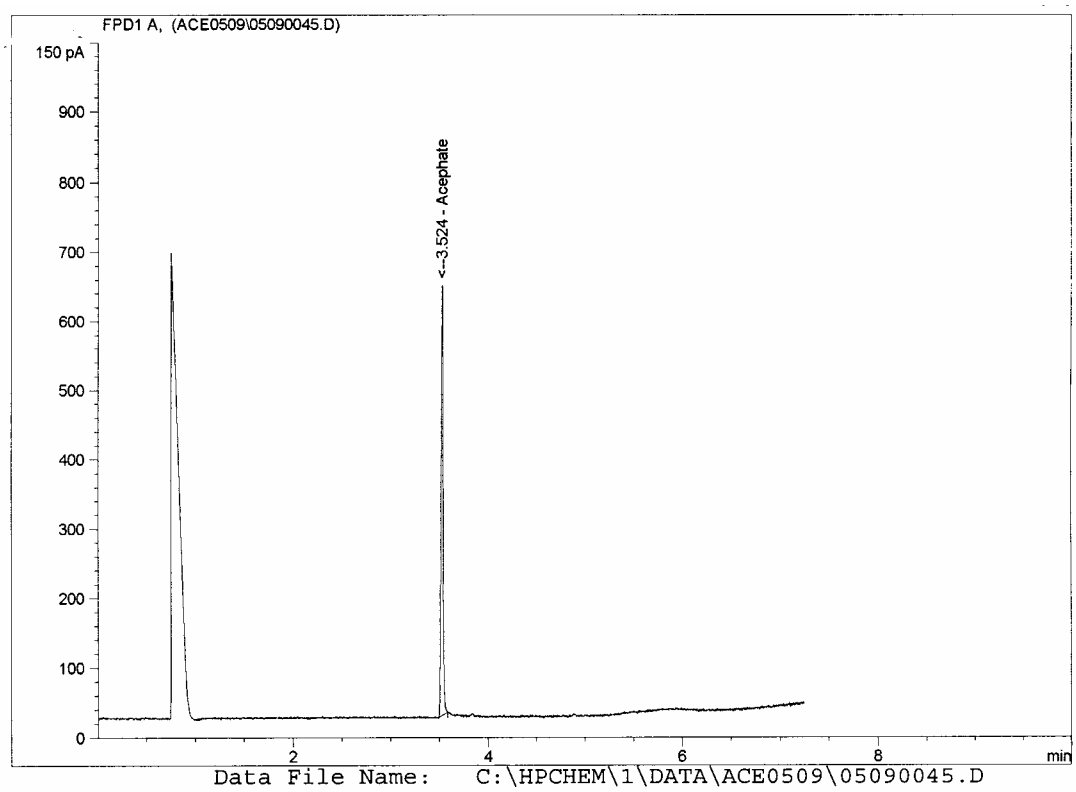

Acephate 0.05 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3



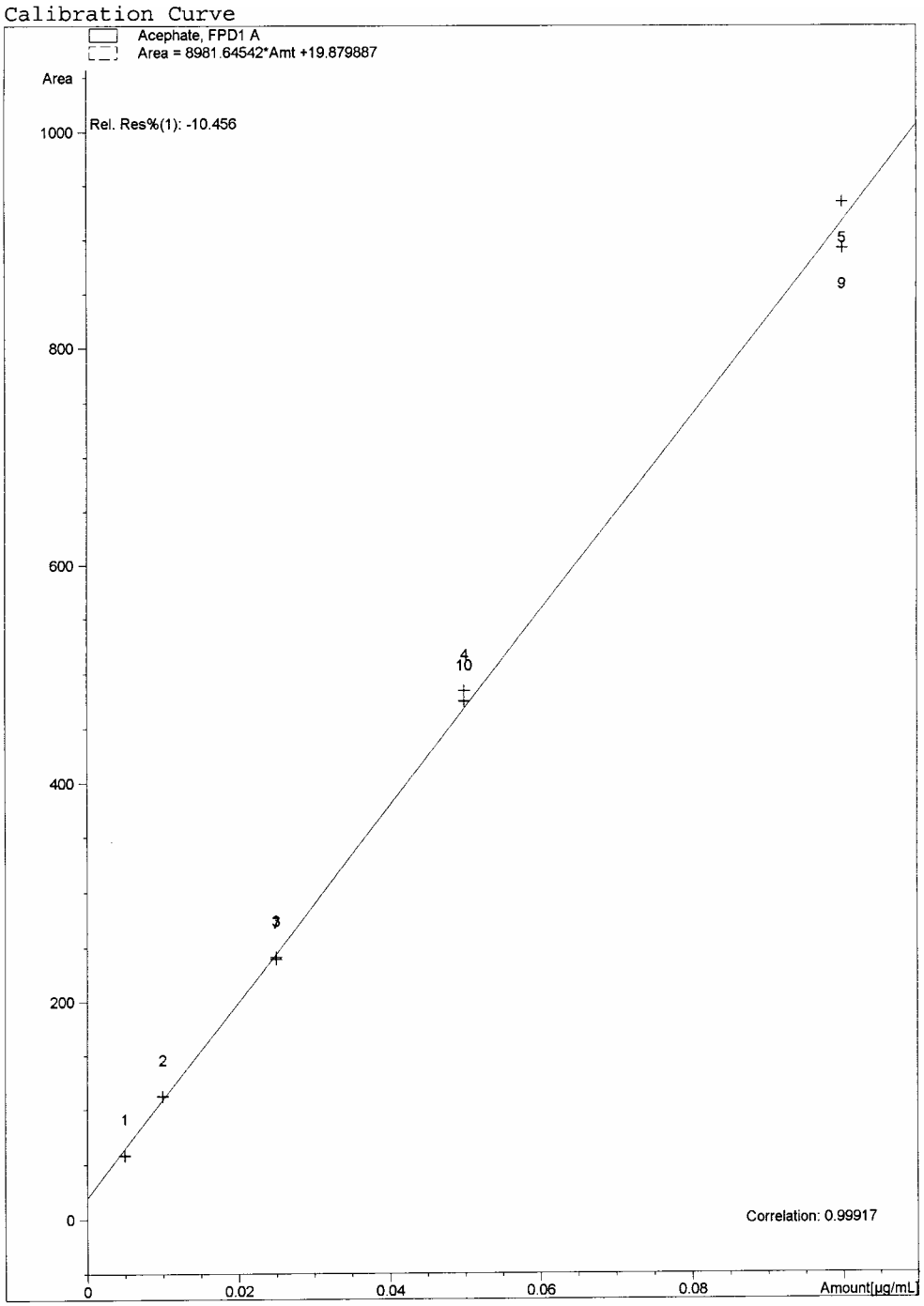

Acephate 0.10 µg/mL Standard, 2 µL

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER STANDARD, SET #3




Acephate 0.10 µg/mL Standard, 2 µL

EXAMPLE GC CALIBRATION CURVE
SET #3



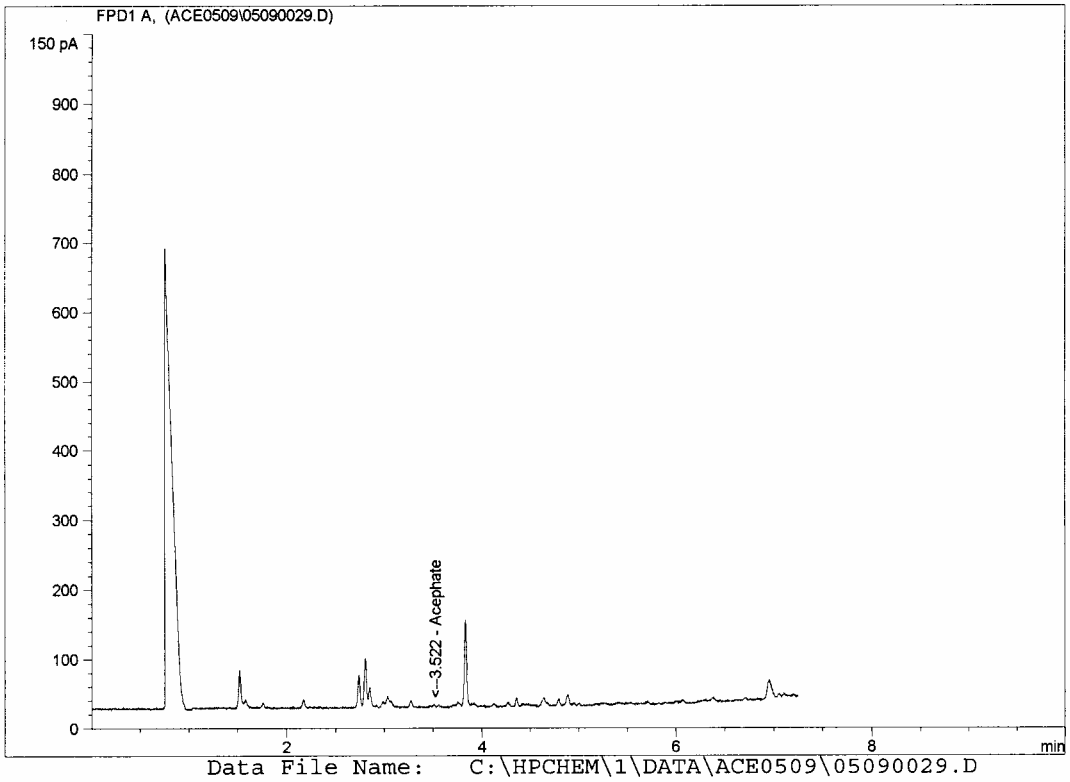

+ Peak response

r = 0.99917
m = 8981.64542
b = +19.879887

Equation y = mx+b

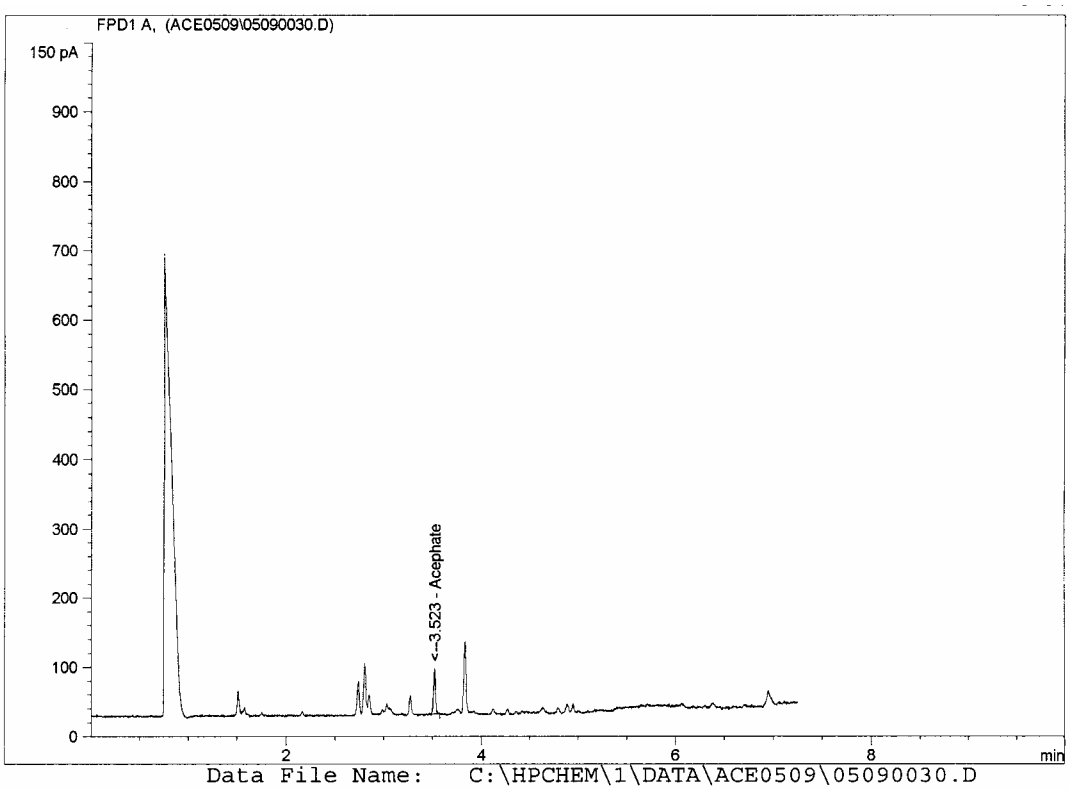
Acephate Calibration Curve

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER,
CONTROL SAMPLE, SET #3



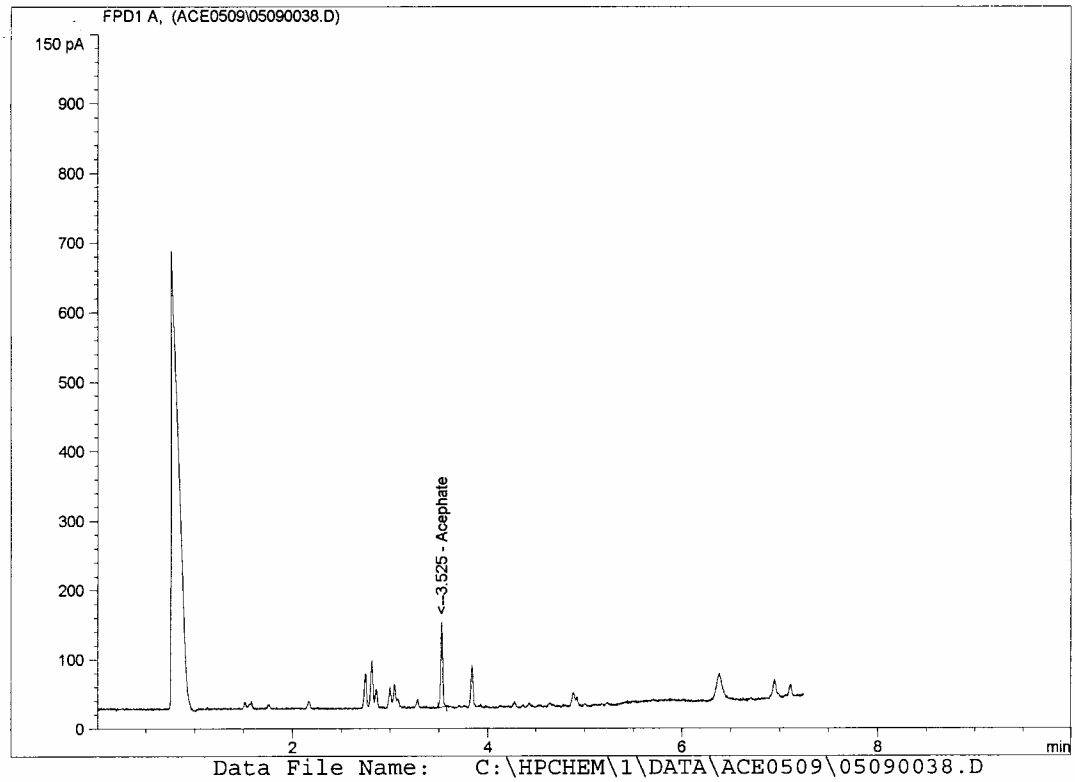

Acephate
1055G Control 3
2 µL of 0.02 section/mL injected

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER,
FORTIFIED CONTROL, SET #3



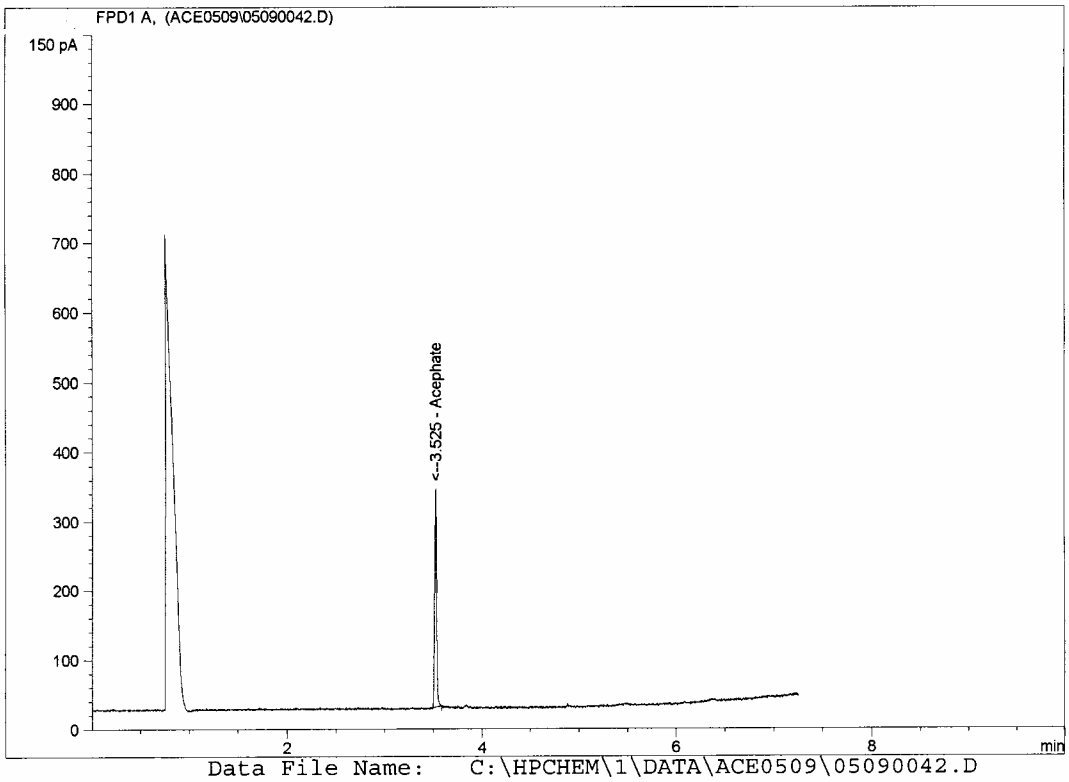

Acephate
1055G Fortified Control 19 @ 0.5 µg/section
2 µL of 0.02 section/mL injected

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER,
FORTIFIED CONTROL, SET #3



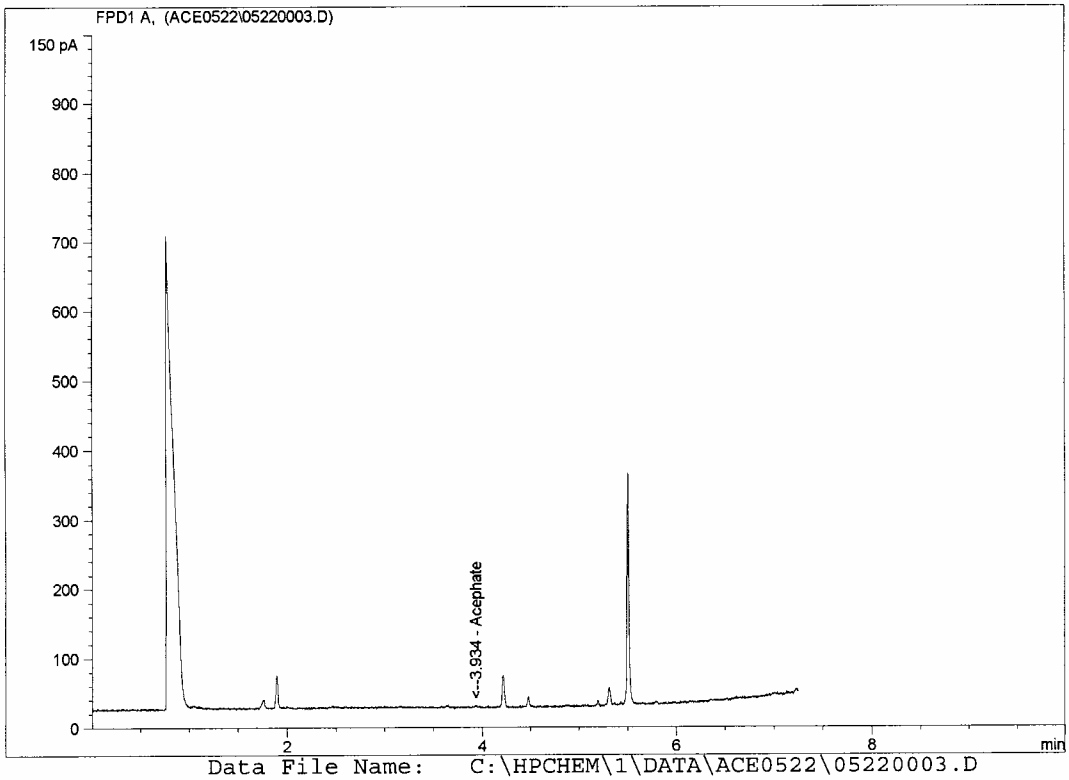

Acephate
1055G Fortified Control 22 @ 1.0 µg/section
2 µL of 0.02 section/mL injected

EXAMPLE GC CHROMATOGRAM OF INNER DOSIMETER,
FORTIFIED CONTROL, SET #3



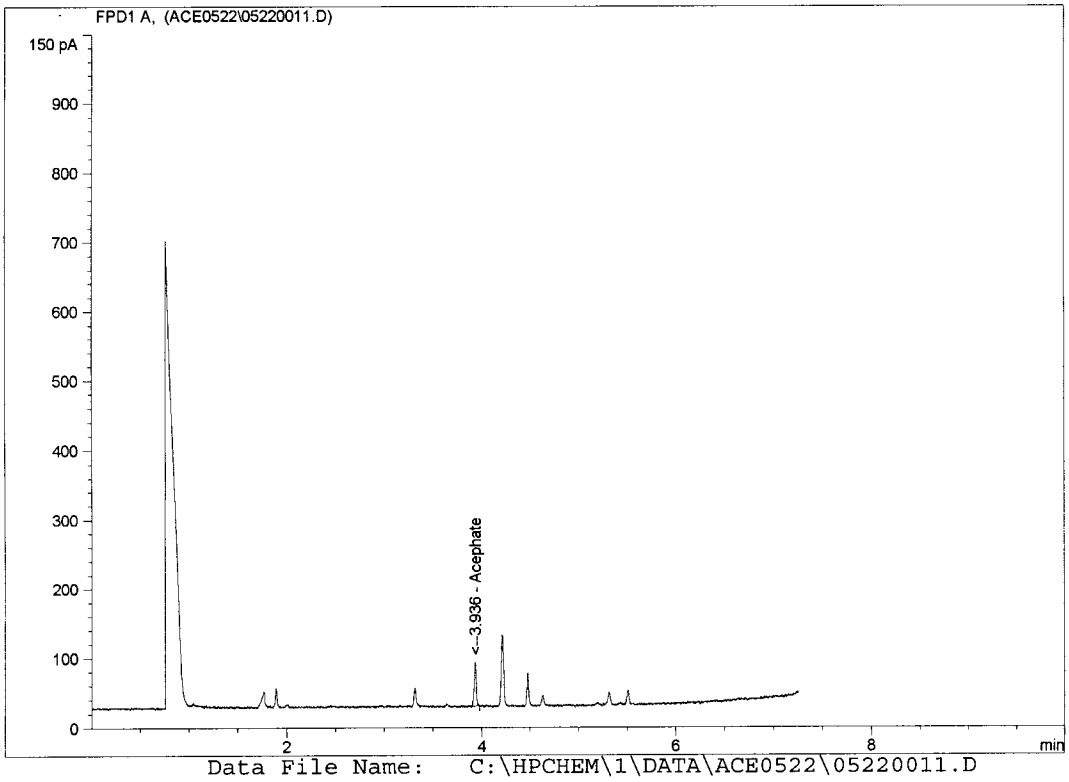

Acephate
1055G Fortified Control 25 @ 10,000 µg/section
2 µL of 0.02 section/mL injection (1-4000 dilution)

EXAMPLE GC CHROMATOGRAM OF FACE/NECK WIPES,
CONTROL SAMPLE, SET #2



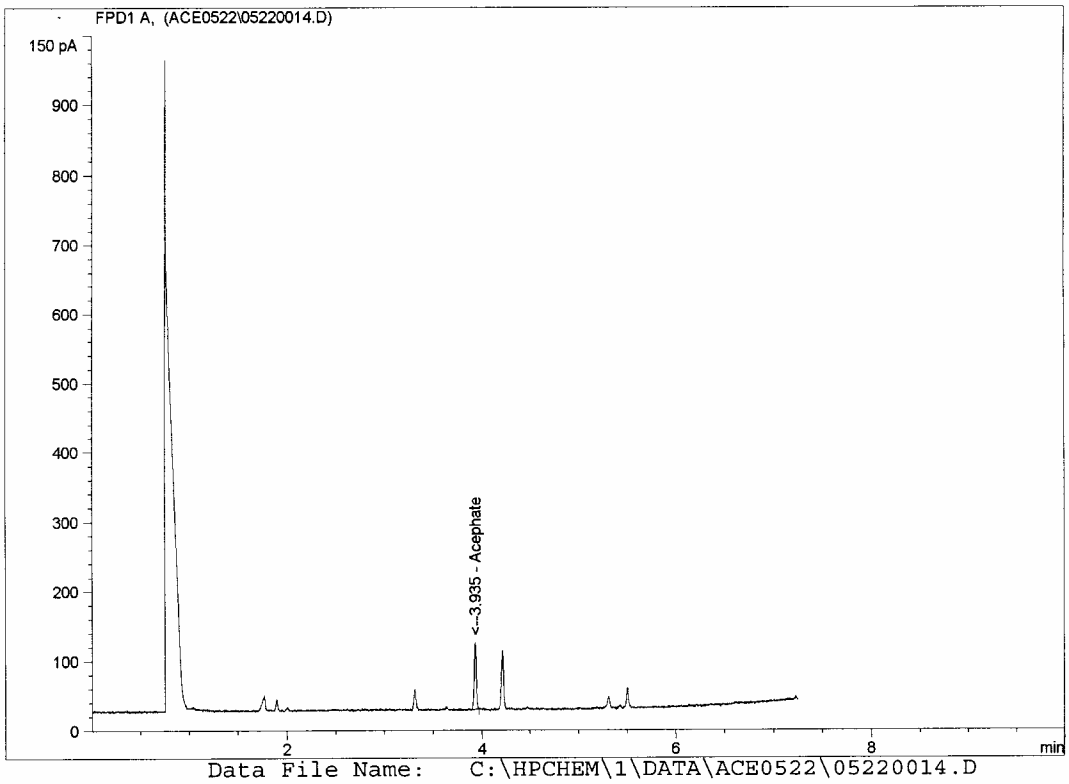

Acephate
1055C Control 2
2 µL of 0.02 wipe/mL

EXAMPLE GC CHROMATOGRAM OF FACE/NECK WIPES,
FORTIFIED CONTROL SAMPLE, SET #2



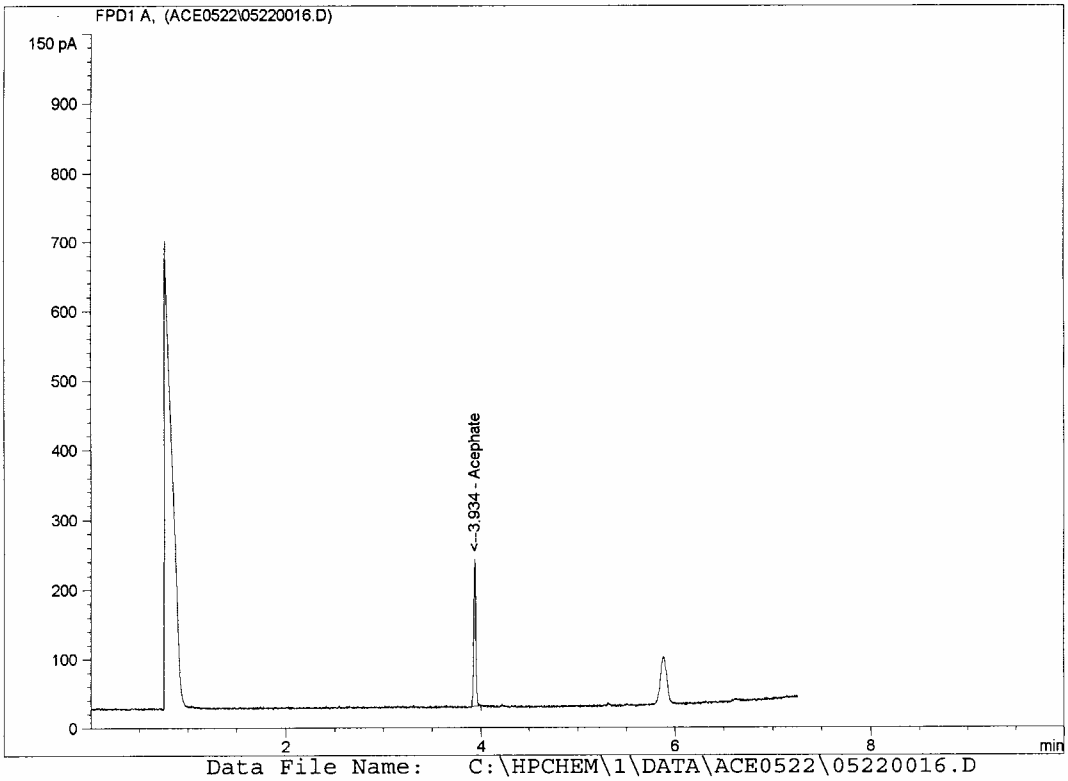

Acephate
1055C Fortified Control 12 @ 0.5 µg/wipe
2 µL of 0.02 wipe/mL

EXAMPLE GC CHROMATOGRAM OF FACE/NECK WIPES,
FORTIFIED CONTROL SAMPLE, SET #2



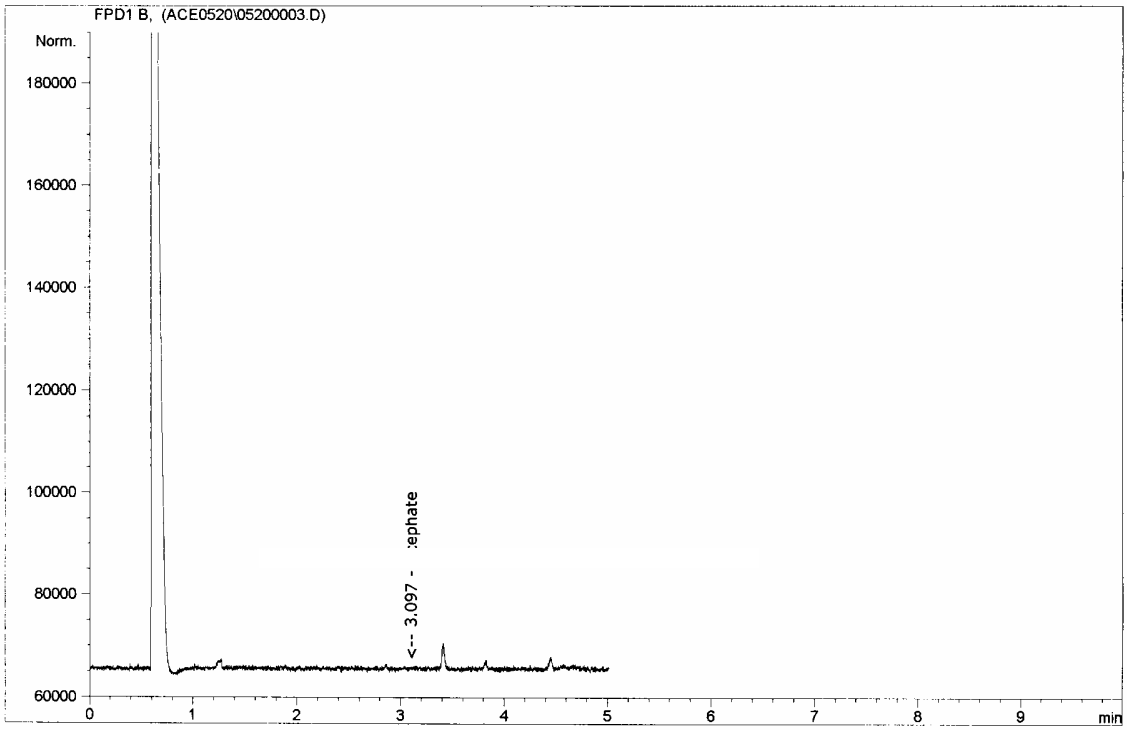

Acephate
1055C Fortified Control 14 @ 1.0 µg/wipe
2 µL of 0.02 wipe/mL injected

EXAMPLE GC CHROMATOGRAM OF FACE/NECK WIPES,
FORTIFIED CONTROL SAMPLE, SET #2



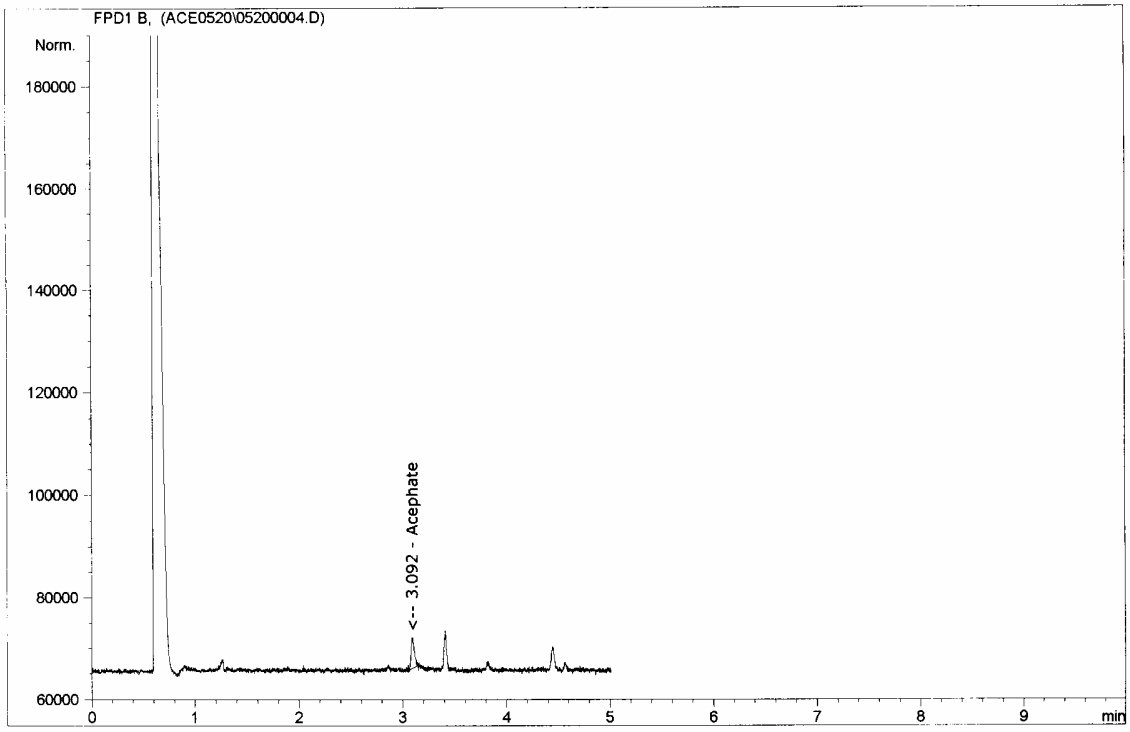

Acephate
1055C Fortified Control 16 @ 100 µg/wipe
2 µL of 0.02 wipe/mL injected (1-40 dilution)

EXAMPLE GC CHROMATOGRAM OF HANDWASH,
CONTROL SAMPLE, SET #9



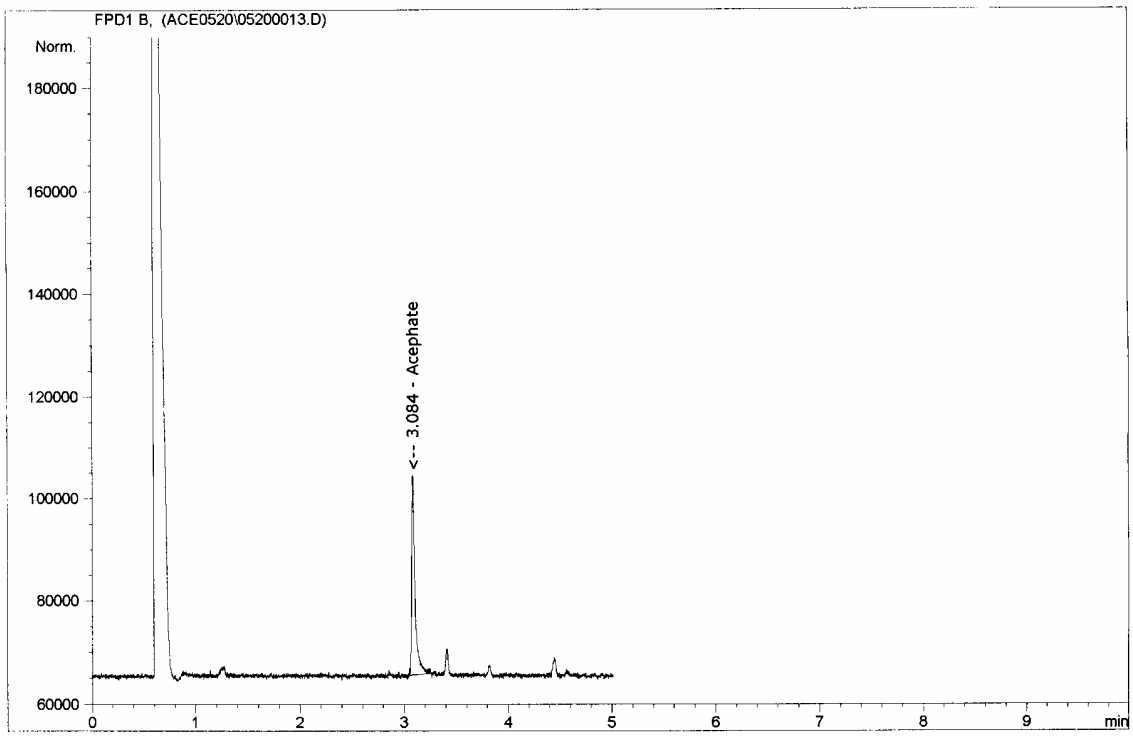

Acephate
1058A Control 9
2 µL of 0.05 sample/mL injected

EXAMPLE GC CHROMATOGRAM OF HANDWASH,
FORTIFIED CONTROL SAMPLE, SET #9



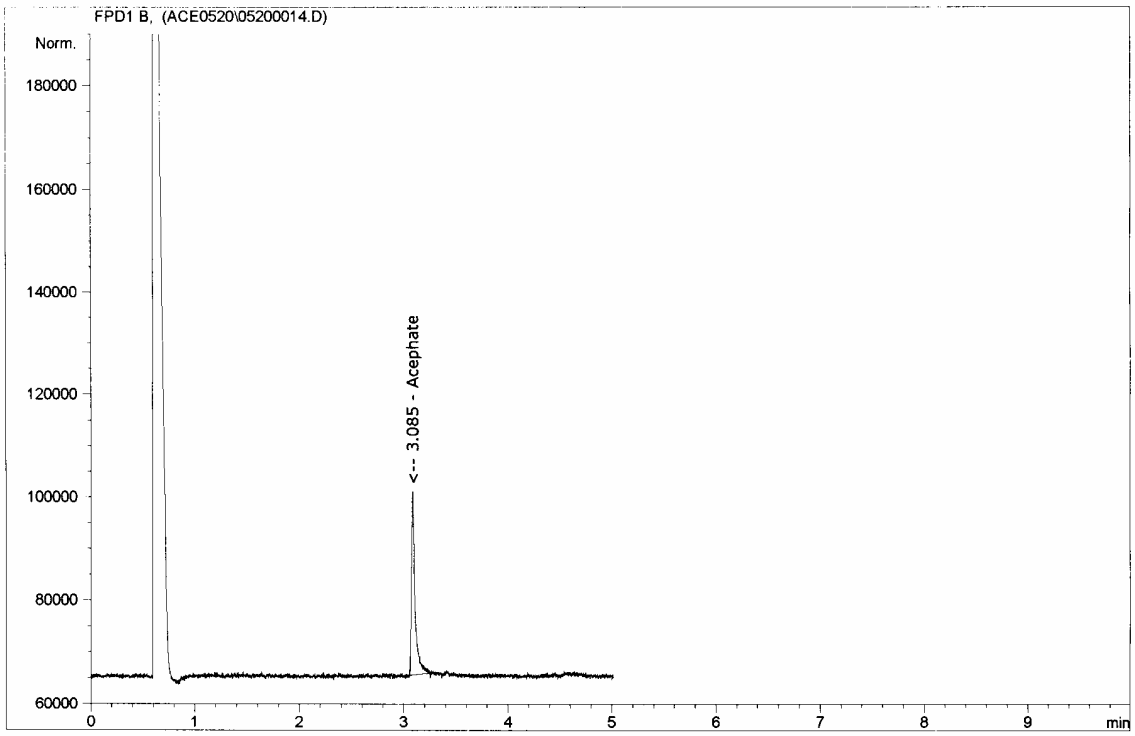

Acephate
1058A Fortified Control 67 @ 0.2 µg/sample
2 µL of 0.05 sample/mL injected

EXAMPLE GC CHROMATOGRAM OF HANDWASH,
FORTIFIED CONTROL SAMPLE, SET #9



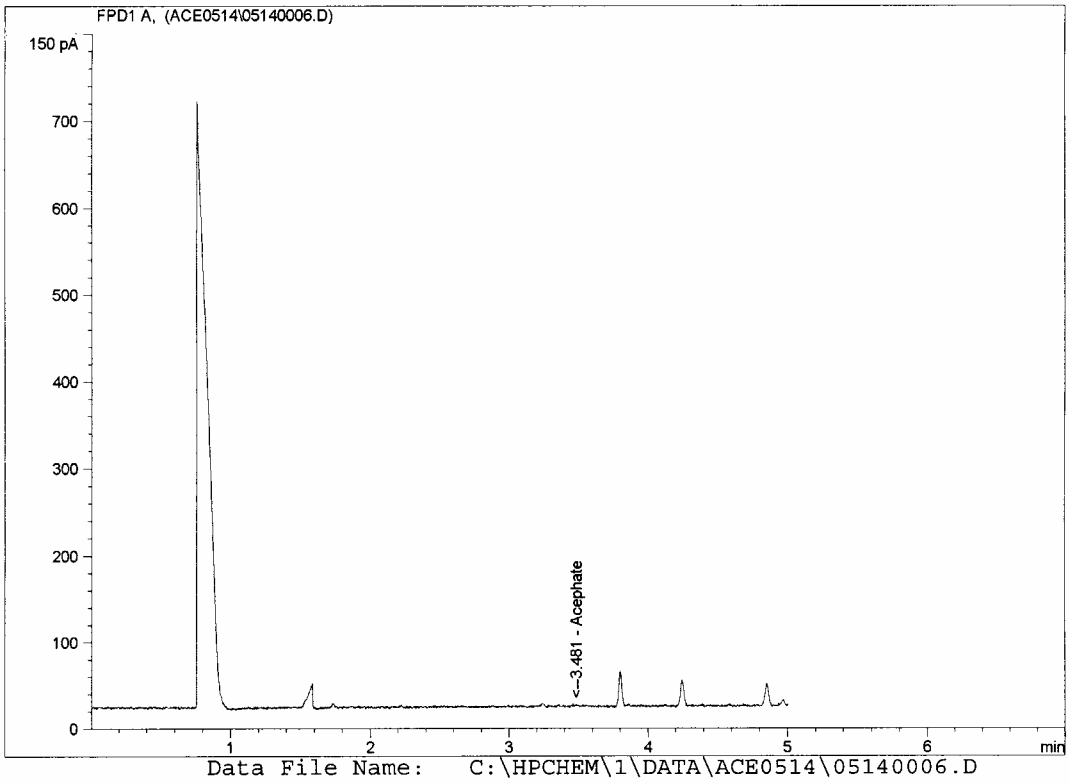

Acephate
1058A Fortified Control 71 @ 1.0 µg/sample
2 µL of 0.05 sample/mL injected

EXAMPLE GC CHROMATOGRAM OF HANDWASH,
FORTIFIED CONTROL SAMPLE, SET #9



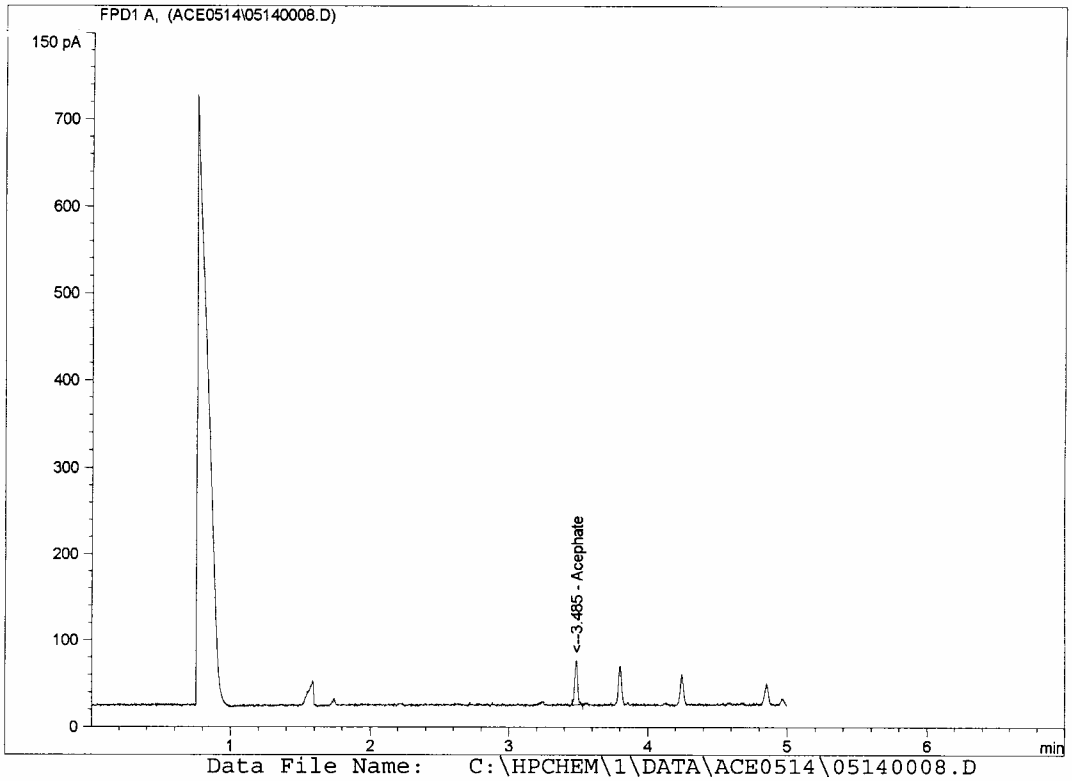

Acephate
1058A Fortified Control 74 @ 1000 µg/sample
2 µL of 0.05 sample/mL injected (1-1000 dilution)

EXAMPLE GC CHROMATOGRAM OF OVS TUBE,
FORTIFIED CONTROL SAMPLE, SET #7



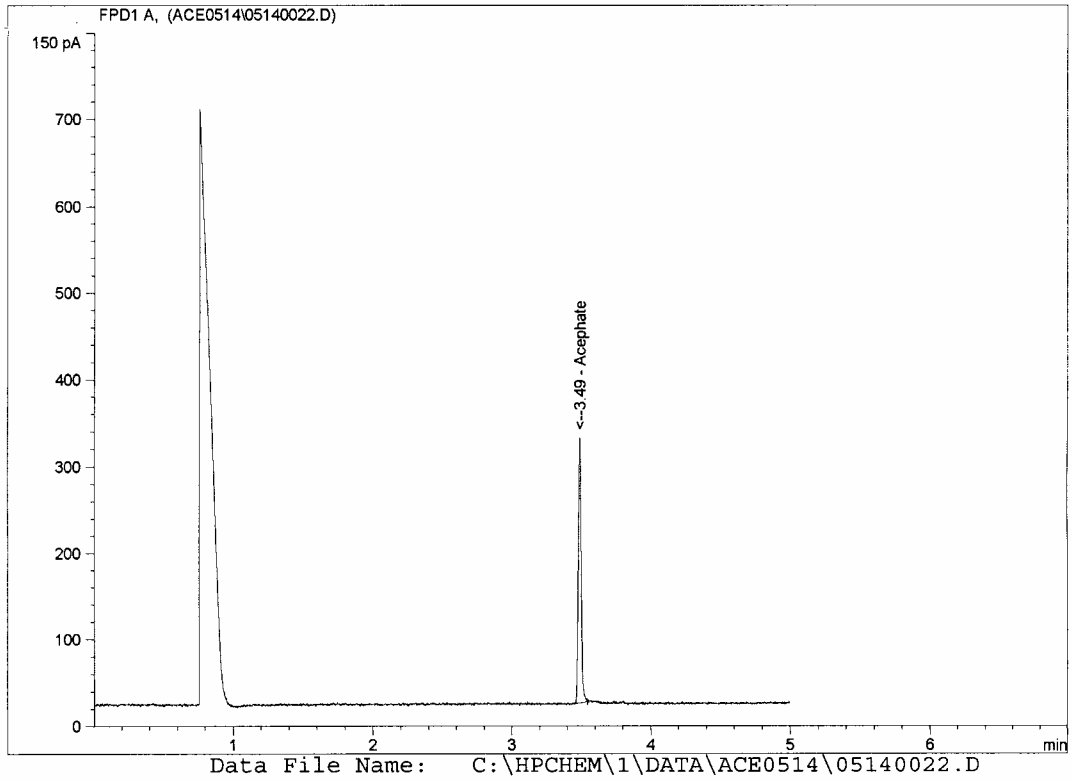

Acephate
1058B Control 7
2 µL of 0.8 tube/mL injected

EXAMPLE GC CHROMATOGRAM OF OVS TUBE,
FORTIFIED CONTROL SAMPLE, SET #7




Acephate
1058B Fortified Control 56 @ 0.01 µg/tube
2 µL of 0.8 tube/mL injected

EXAMPLE GC CHROMATOGRAM OF OVS TUBE,
FORTIFIED CONTROL SAMPLE, SET #7




Acephate
1058B Fortified Control 60 @ 50 µg/tube
2 µL of 0.8 tube/mL injected (1-800 dilution)