ANALYSIS OF METHYL PARATHION IN AIR BY GC/MS

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1.0 SCOPE AND APPLICATION

The objective of this standard operating procedure is to provide guidance on the requirements for the analysis of methyl parathion in air samples using gas chromatography/mass spectrometry (GC/MS), selective ion monitoring mode. The method is based on NIOSH Method 5600 for the analysis of Organophosphorus Pesticides in air samples.

2.0 METHOD SUMMARY

The air samples are collected on two stage OVS-2 resin tubes, extracted with acetone:toluene 10:90 (v:v) and the extracts analyzed by GC/MS. Prior to GC/MS analysis, a 1-mL aliquot of the extract is spiked with the internal standard phenanthrene-d_{10}. The extract is then analyzed for methyl parathion. Identification and quantitation is made by comparing the retention times and mass spectral data of methyl parathion with that of methyl parathion from calibration standards as follows:

The GC oven is temperature programmed to separate the methyl parathion on a fused silica capillary column, which is then detected with the mass spectrometer (MS). The methyl parathion eluting from the GC column is identified by comparing its measured mass spectra and retention time to reference spectra and retention time in a user created database. Reference spectra and retention times for methyl parathion are obtained by the measurement of calibration standards under the same conditions used for sample extracts. The concentration of methyl parathion is calculated by relating the MS response of the quantitation ion produced by methyl parathion, to the MS response of the quantitation ion produced by the internal standard phenanthrene-d_{10}.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

The OVS-2 resin used for sampling is housed in a glass tube that has been flame sealed.

To preserve and store air samples collected on OVS-2 tubes:

1. Place plastic caps on the OVS-2 tube ends.
2. Place the sample in a whirl bag. If duplicate samples are collected, place both tubes in one whirl bag.
3. Protect the samples from light and refrigerate at 4°C (±2°C) from the time of receipt until extraction and analysis.
4. Recommended maximum holding time is two weeks.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

1. High humidity and temperature, and high sampling flow rates may decrease the absorption capacity of the resin. Contaminants may migrate from the front portion to the back portion of the tube.

2. Impurities in the purge gas and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by analyzing laboratory reagent blanks.

3. Samples can be contaminated by diffusion of methyl parathion through the septum seal into the sample during storage and handling. A holding blank or field blank carried through the holding period and the analysis protocol serves as a check on such contamination. One holding (field) blank per batch of

samples should be analyzed.

5.0 EQUIPMENT/APPARATUS

1. Micro syringes - Hamilton gas tight syringes: 10, 25, 50, 100, 500, and 1000 µL, 0.006 inch ID needle.

2. OVS-2 resin tubes - OVS-2 tubes (SKC, Inc. Catalog No. 226-30-16 or equivalent).

3. Balance - Analytical, capable of accurately weighing ±0.0001 g.

4. Water bath sonicator

5. Serum vial - 10 mL, crimp top with Teflon cap liner.

6. Volumetric flasks - class A with ground-glass stoppers: 5, 10, 25, and 50 mL volumes.

7. Vials - 2 mL for GC autosampler.

8. Desorption vials - Supelco 3.7 mL vials (Cat #2-7136 or equivalent), screw cap with Teflon cap liner.

9. Gas Chromatography/Mass Spectrometer (GC/MS)

   A GC/MS system which meets the following specifications will be used:

   Gas Chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases is required.

   The GC must be capable of temperature programming and should be equipped with variable-constant differential flow controllers so that the column flow rate will remain constant throughout the temperature program operation. In addition, a split/splitless injection port is required.

   Capillary Gas Chromatography Columns - Any gas chromatography column that meets the performance criteria of separating the calibration mixture of this method is acceptable. One useful column has been identified.

      Column -- 30 m x 0.25 mm ID, Restek XTI-5 (crossbonded SE-54), fused silica capillary with a 0.50 µm film thickness.

   Mass spectrometer - The mass spectrometer must be capable of electron ionization at a nominal electron energy of 70 eV, and must be capable of scanning in the selective ion monitoring (SIM) mode. The ions to be monitored are: 187, 188, and 189 for d_{10}-phenanthrene; and 109, 125, and 263 for methyl parathion. The mass spectrometer must produce a spectrum that meets all criteria in Appendix A when 50 ng of decafluorotriphenyl- phosphine (DFTPP) is introduced into the GC.

   GC/MS interface - Any gas chromatograph to mass spectrometer interface that allows 20 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria may be used. The capillary column is directly coupled with the analyzer, providing maximum sensitivity.

Data system - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

The computer software should be capable of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectra from the GC peak with spectral data in a user-created database. The software must allow integration of the ion abundance of any specific ion between specified times or scan number limits. The software should also allow the calculation of response factors (or construction of a second or third order regression calibration curve), response factor statistics (mean and standard deviation), and concentrations of analytes using either the calibration curve or the equation in Section 8.

6.0 REAGENTS

All standard solutions are prepared and documented in accordance with EPA/SERAS SOP #1012, "Preparation of Standard Solutions".

1. Toluene (glass distilled, suitable for GC)

2. Methylene chloride (glass distilled, suitable for GC)

3. Acetone (glass distilled, suitable for GC)

4. Decafluorotriphenylphosphine (DFTPP).

   Prepare a 50 µg/mL daily working standard solution of DFTPP by diluting 50 µL of a commercially available 25,000 µg/mL (Supelco catalog number 4-8724 or equivalent) in 25.0 mL of methylene chloride. Protect the DFTPP from light and refrigerate at 4°C (±2°C). This solution must be replaced every 12 months, or sooner if comparison with quality control check samples indicates a problem.

5. Internal standard

   Purchase a 2000 µg/mL solution (Supelco catalog number 4-8710 or equivalent) of Phenanthrene - d_{10}. Prepare serial dilutions in methylene chloride of the 2000 µg/mL to a working stock of 20 µg/mL.

   Protect the solution from light and refrigerate at 4 °C (±2 °C). This solution must be replaced every 12 months, or sooner if comparison with quality control check samples indicates a problem.

6. Matrix Spike/Matrix Spike Duplicate Solution:

   Prepare a 10,000 µg/mL stock solution of methyl parathion by the addition of .100 gm ± 5% of methyl parathion (Chem Service Catalog Number F996), to 10 mL of 10:90 acetone:toluene (V:V).

   Prepare the 100 µg/mL solution of matrix spike mix by diluting 100 µL of the 10,000 µg/mL stock solution to 10.0 mL in methylene chloride. Store the spiking solution at 4°C (±2°C in Teflon-sealed

containers, protected from light. The solution should be checked frequently for stability. These solutions must be replaced every 12 months, or sooner if comparison with quality control check samples indicates a problem.

7. Calibration Standards

Prepare calibration standards at six concentration levels (0.05, 0.1, 0.5, 1.0, 5, and 25 µg/mL). Prepare a working stock of all compounds at 10,000 µg/mL as in Step 5. Prepare serial dilutions in methylene chloride of the 10,000 µg/mL solution to obtain the 6 levels of calibration standards. These solutions must be replaced every 12 months, or sooner if comparison with quality control check samples indicates a problem.

7.0 PROCEDURES

7.1 Preparation and Extraction

7.1.1 Sampler

The sample consists of a glass tube, 11-mm ID x 13-mm OD x 50 mm long, with the outlet; and drawn to a 6-mm OD x 25 mm long tube. The enlarged part of the tube contains a 270 mg front section of 20/60 mesh OVS-2 sorbent or equivalent held in place by a 9 to 1-mm OD quartz fiber filter and polytetrafluoroethylene (PTFE) retaining ring. The front section is separated from the back section of 140 mg OVS-2 sorbent or equivalent with a short plug of polyurethane foam. The back section is held in place by a long plug of polyurethane foam. The tube is available commercially as the OVS-2 sampler.

- Remove cap from large end and remove PFTE retainer ring.
- Remove the filter and the front OVS-2 section and place it in a 4-mL screw top vial. Label the vial "front" along with the sample name.
- Remove the short polyurethane foam plug along with the backup OVS-2 and place in a second 4-mL screw top vial. Label this vial "back" along with the sample name.
- Pipette 2.0 mL of desorbing solvent consisting of 10:90 acetone:toluene (V:V) in each of the vials. Screw the tops on tightly.
- Immerse vials approximately 15 mm in an ultrasonic bath for 30 minutes. Let it stand for 30 minutes.
- Transfer 1.0 mL of the extract from each vial to a 1 mL auto sample vial.

7.1.2 Matrix Spike/Matrix Spike Duplicate Extraction

Break the glass in the front portion of the tube. Spike the OVS-2 resin with 20 µL of the matrix spiking solution; take care that the syringe is in the middle of the OVS-2 resin in the front portion of the tube. Place only the front portion of the resin into a vial and extract the spike as in

Section 7.1.1.

7.1.3 Lot Blank Preparation

An unopened blank of each lot of tubes used is included with each batch of the samples. The lot blank is broken up without disturbing the contents and then treated exactly as a sample as in Section 7.1.1.

7.2 GC/MS Operating Conditions

The following GC/MS operating conditions are recommended:

Column	Restek XTI-5 (crossbonded SE-54 or equivalent) 30 meter x 0.25 mm ID, 0.50 µm film thickness
Injection Temperature	260° C
Transfer Temperature	260 ° C
Source Temperature	260 ° C
Temperature Program	100 ° C 15 ° C/min to 290 ° C hold for 1 min
Splitless Injection	Split time = 0.75 min
Injection Volume	2 µL

7.3 Tune (DFTPP)

The instrument must be tuned to meet the ion abundance criteria listed in Appendix A for a 50-ng (1µL) injection of DFTPP. This criteria must be demonstrated every 24 hours during analysis.

7.4 Initial Calibration

1. Add 20 µL of the internal standard phenanthrene-d_{10} to each 1-mL aliquot of calibration standards.

2. Inject 2 µL each of the calibration standards after a successful DFTPP analysis.

3. Calculate and tabulate the relative response factor (RRF) against the concentration for each compound by using the equation listed below. The primary ion from the specific internal standard must be used for quantitation.

   The average RRF and percent relative standard deviation (%RSD) must also be calculated and tabulated.

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_X = Area of the characteristic ion for the compound to be measured

C_{IS} = Concentration of the internal standard (ng/µL)
A_{IS} = Area of the characteristic ion for the internal standard.
C_X = Concentration of the compound to be measured (ng/µL)

The % RSD of the RRF for each methyl parathion has been tentatively adopted to be less than or equal to 30%. The average RRF of methyl parathion should not be less than 0.05.

7.5 Continuing Calibration

A check of the initial calibration curve must be performed every 24 hours during analysis.

1. Inject 2 µL of a 1.0 µg/mL methyl parathion standard containing internal standard phenanthrene-d_{10}.

2. Calculate and tabulate the daily RRF for each compound. All daily RRF should be equal to or greater than 0.05.

3. Calculate the percent difference (% D) of each daily RRF compared to the average RRF from the initial calibration curve. The % D for all compounds can be calculated using the equation listed below and must be less than or equal to 25%.

$$\%D = \frac{RRF_{Daily} - RRF_{Average}}{RRF_{Average}} \times 100$$

4. All sample and standards are quantitated using the response factors from the daily calibration check.

7.6 Sample Analysis

Sample extracts may be analyzed only after the GC/MS system has met the DFTPP, initial calibration, and continuing calibration requirements mentioned above. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

1. Add 20 µL of the internal standard phenanthrene-d_{10} into the lot blank(s) the MS/MSD, and all the sample extracts.

2. Inject 2 µL each of the MS/MSD, lot blank(s), and all the sample extracts.

3. If the analyst has reason to believe that diluting the final extracts will be necessary, an undiluted run may not be required.

4. If methyl parathion is detected at a level greater than the highest calibration standard, sample extracts must be diluted so that the methyl parathion response is within the linear range established during calibration.

5. If dilutions of sample extracts are made, additional internal standards must be added to maintain the required concentration (400 ng/mL) of each internal standard in the extract.

7.7 Identification of Methyl Parathion

Methyl Parathion identification will be conducted by comparison of the sample mass spectrum to the mass spectrum of a standard of the methyl parathion. Two criteria must be satisfied to verify the identifications:

- Elution of the methyl parathion in the sample at the same GC relative retention time as the methyl parathion standard.
- Correspondence of the methyl parathion in the sample and the reference methyl parathion mass spectra.

1. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

2. For comparison of standard and sample component mass spectra, reference mass spectra must be obtained from the 1.0 µg/mL standard. These standard spectra may be obtained from the run used to obtain reference RRTs.

3. The requirements for qualitative verification by comparison of mass spectra are as follows:

   a. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

   b. The relative intensities of ions specified in (a) must agree within ±20% between the standard and sample spectra. (For example: for an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30-70%.)

   c. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the quantitation limit, report the actual value followed by "J", e.g., "3J".

4. If a compound cannot be verified by all of the criteria in step 3, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the analyst shall report that identification and proceed with the calculation in Section 8.0. The analyst should note in the case narrative that technical judgment was utilized.

7.8 Desorption Efficiencies

The desorption efficiencies (DE) were determined for methyl parathion at 0.1, 2.0, and 50 µg. Three replicate OVS-2 tubes at each level were spiked with methyl parathion, extracted with acetone:toluene 10:90 and analyzed by GC/MS (SIM). The desorption efficiencies are listed in Appendix B. The DE were calculated as follows:

$$DE = \frac{\text{Amount Spiked } \mu}{C_S \times V}$$

$$C_S\ \mu\ /mL\ = \frac{(A_s)(C_{is})}{(A_{is})\,(RRF)}$$

where:

C_S = concentration found (µg/mL)
V = Volume extracted
A_S = Area of the characteristic ion for the compound to be measured
C_{IS} = Amount of internal standard added (µg/mL)
A_{IS} = Area of the characteristic ion for the specific internal standard (IS)
RRF = Relative response factor from the analysis of calibration standard

The Relative Response Factor (RRF) is obtained from the standard. It is calculated as follows:

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

The desorption efficiencies will be determined for each lot of tubes, or once per year, whichever is more frequent. This SOP will be updated when new DE studies are conducted. Supporting documentation will be kept in a file in the laboratory.

7.9 Method Detection Limits

The Method Detection Limits (MDL) OVS-2 listed in Appendix C were determined by analyzing seven OVS-2 resin tubes spiked with 100 ng methyl parathion which is equivalent to 50 ng/mL in the extract. The 50 ng/mL solution represents the lowest concentration on the linear range of the five-point calibration curve. The spiked tubes were subsequently extracted with acetone: toluene 10:90 as in Section 7.1 and analyzed by GC/MS. Method detection limits are determined annually or for each lot of OVS-2, which ever is more frequent. This SOP will be updated when new MDL studies are conducted. Supporting documentation will be kept in a file in the laboratory.

$$MDL = (S)(t_{(n-1,1-alpha=0.99)})$$

where:

$t_{(n-1,1-alpha = 0.99)}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom
n = number of replicates
S = the standard deviation of the replicate analyses

$$S = \sqrt{\sum \frac{(x - x_{ave})^2}{n-}}$$

where:

X_j = each individual concentration
X_{ave} = mean concentration

injections $t_{(n-1,1-alpha\ =\ 0.99)}$ = 3.143. Therefore, substituting into equation above yields:

$$MDL = 3.143\ x\ S$$

The detection limits obtained here are to support the actual MDL of 0.2 µg/sample used in the results tables.

8.0 CALCULATIONS

8.1 Methyl Parathion

The methyl parathion identified by the GC/MS method shall be quantitated by the internal standard method. The internal standard (IS) used shall be phenanthrene-d_{10}. The EICP area of the characteristic ions of the methyl parathion and IS are used. Methyl parathion concentrations and concentration conversions are calculated as follows:

1. Amount of analyte in total sample (µg/sample):

$$\mu\ /\text{sample} = \frac{(A_s)(C_{is})}{(A_{is})\ (RRF)}\ x\ V \times\ \text{DE}$$

where:

A_S = Area of characteristic ion for the analyte to be measured
C_{IS} = Concentration of internal standard (µg/mL)
V = Extraction Volume (mL)
DE = Desorption efficiency
A_{IS} = Area of characteristic ion for the internal standard
RRF = Relative response factor of analyte

The Relative Response Factor (RRF) is calculated from the calibration standard solution mixture using:

$$RRF = \frac{(A_u)(C_{is})}{(A_{is})(C_u)}$$

where:

$\mathbf{A_U}$ = Area of characteristic ion of analyte in the standard mixture
$\mathbf{A_S}$ = Area of characteristic ion of internal standard in the standard mixture
$\mathbf{C_S}$ = Concentration of internal standard in the standard mixture (µg/mL)
$\mathbf{C_U}$ = Concentration of analyte in the standard mixture (µg/mL)

2. Concentration of analyte in $\mu g/m^3$:

$$\text{Concentration } (\mu g/m^3) = \frac{(\text{Total } \mu g \text{ front} + \text{Total } \mu g \text{ back})}{\text{Liters sampled}} \times \frac{1000L}{1m^3}$$

8.2 Matrix Spike/Matrix Spike Duplicate Recoveries

The MS/MSD will be determined by spiking the OVS-2 tubes with 220 µg of methyl parathion. No air will be drawn through the OVS tubes. The amount recovered (µg/sample) can be calculated using the following equation:

$$\mu g\ sample = \frac{(A_S)(C_{is})}{(A_{is})(RRF)} \times V \times DE$$

where:

A_S = Area of characteristic ion for the compound to be measured
C_{IS} = Concentration of the internal standard (µg/mL)
V = Extraction volume (mL)
DE = Desorption efficiency
A_{IS} = Area of characteristic ion for the specific internal standard
RRF = Relative response factor of analyte

The percent recovery for the MS/MSD and relative percent difference (RPD) can be calculated using the equations below:

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

and

$$RPD = 100 \times \frac{|D_1 - D_2|}{(D_1 + D_2)} \times 0.5$$

where:

SSR = Spike sample results
SR = Sample results
SA = Amount of spike added
D_1 = First sample value
D_2 = Second sample value (duplicate)

The vertical bars in the formula above indicate the absolute value of the difference; hence RPD is always expressed as a positive value.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Tune (DFTPP)

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument tune criteria specified in Appendix A. The

purpose of this instrument check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of DFTPP.

1. The analysis of DFTPP must be performed every 24 hours during the analysis.

2. The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Appendix A.

9.2 Initial Calibration for Methyl Parathion

Prior to the analysis of samples and required blanks, and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of six concentrations to determine the linearity of response utilizing methyl parathion standards.

1. The levels of the initial calibration standards for methyl parathion are 0.05, 0.1, 0.5, 1.0, 5, and 25 µg/mL.

2. The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of methyl parathion. Criteria have not been established for the minimum RRF and %RSD. However, tentative criteria have been adopted at this time. The minimum RRF of each compound at each concentration level in the initial calibration across all six points is tentatively adapted to be equal to or greater than 0.05; the %RSD is tentatively adopted to not exceed 30%.

9.3 Continuing Calibration for Methyl Parathion

Once the GC/MS system has been calibrated, the calibration must be verified each 24-hour time period for each GC/MS system during the analysis.

1. The level of the continuing calibration standard for methyl parathion is 1.0 µg/mL.

2. The standard is to be analyzed every 24 hours after an acceptable DFTPP analysis.

3. The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of methyl parathion from the initial calibration and the RRF of methyl parathion in the continuing calibration standard. Criteria have not been established for the minimum RRF and %D. However, tentative criteria have been adopted at this time. The minimum RRF of methyl parathion in the continuing calibration is tentatively adopted to be greater than or equal to 0.05. The %D is tentatively adopted to not exceed 25%.

4. If any of the requirements listed in Item 3 are not met, another initial calibration will be analyzed.

9.4 Lot Blank Analysis

A lot blank is an unopened OVS-2 resin tube from the same lot as the sample tubes. The purpose of the lot blank is to determine the levels of contamination associated with the manufacture, extraction, and

analysis of the samples

1. One lot blank must be extracted and analyzed for every lot represented in the sampling event for each project.

2. The lot blank must contain less than or equal to the MDL of methyl parathion.

3. If a lot blank exceeds the limits for contamination above, the analyst must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective action taken and documented before further sample analysis proceeds.

9.5 Dilution Analysis

If the concentration of any sample extract exceeds the initial calibration range, that sample extract must be diluted and reanalyzed as described in Section 7.6, steps 4 and 5.

1. Use the results of the original analysis to determine the approximate dilution factor required to get the methyl parathion within the initial calibration range.

2. The dilution factor chosen should keep the response of methyl parathion in the upper half of the initial calibration range of the instrument.

3. Do not submit data for more than two analyses, i.e., the original sample and one dilution, or, from the most concentrated dilution analyzed and one further dilution.

9.6 Matrix Spike/Matrix Spike Duplicate Recoveries

The purpose of spiking methyl parathion into two OVS-2 vials is to evaluate the effects of the resin matrix on the methods used in this SOP.

1. The MS/MSD must be prepared for every 20 samples for each project.

2. The recoveries of methyl parathion are calculated according to the procedures in Section 8.2. The relative percent difference between the results of the matrix spike and the matrix spike duplicate are calculated according to the procedures in Section 8.2.

3. No quality control limits for recovery and relative percent difference are available.

10.0 DATA VALIDATION

Data validation will be performed by the Data Validation and Report Writing Group and therefore it is not applicable to this SOP. However, data is considered satisfactory for submission purposes when the requirements mentioned below are met.

1. All samples must be analyzed, within the holding time, under an acceptable tune, initial calibration, and continuing calibration check at the required frequency.

2. An acceptable lot blank will be submitted with each batch of samples.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to U.S. EPA, OSHA and corporate health and safety practices. More specifically, refer to ERT/SERAS SOP #3013, SERAS Laboratory Safety Program.

12.0 REFERENCES

Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Third Edition, SW-846, September 1986.

U.S. EPA Contract Laboratory Program (CLP), Statement of Work for Organic Analysis, Revision 2/88.

U.S. EPA Contract Laboratory Program (CLP), Statement of Work for Organic Analysis, Document Number OLM01.0 (including revisions through OLM01.8).

NIOSH Manual of Analytical Methods, Fourth Edition, 8/15/94, Method 5600.

APPENDIX A
Ion Abundance Criteria for Tune (DFTPP)
SOP #1825
March, 1995

Ion Abundance Criteria for Tune (DFTPP)

Mass	Ion Abundance Criteria
51	30.0 - 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Present
70	Less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	Greater than 0.75 percent of mass 198
441	Present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

APPENDIX B
Desorption Efficiencies
SOP #1825
March, 1995

DESORPTION EFFICIENCIES
OVS-2 TUBE LOT # 702
ANALYZED 3/1/95

	0.1 µg	2.0 µg	50 µg
Methyl Parathion	1.61865	1.14549	0.79181

APPENDIX C
MDL Results for Methyl Parathion in OVS-2 Tubes
SOP #1825
March, 1995

MDL RESULTS FOR METHYL PARATHION IN OVS-2 TUBES
02/24/95

Results (µg)

Compound	Spike #1	Spike #2	Spike #3	Spike #4	Spike #5	Spike #6	Spike #7	S	MDL
Methyl Parathion	0.12726	0.13128	0.12586	0.12062	0.11082	0.1248	0.11658	.00696	.02188

Actual spike = 0.1 µg/tube