Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Molinate (S-methyl)

MRID #: 414218-03

Matrix: Soil

Analysis: GC/NPD

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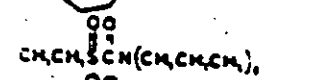
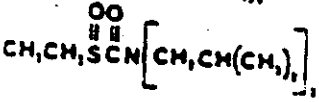
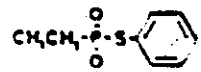
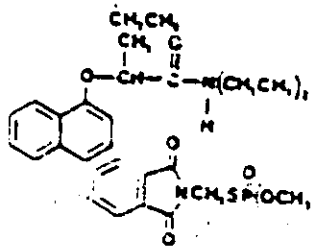
GAS CHROMATOGRAPHIC DETERMINATION OF EPTC SULFOXIDE, BUTYLATE SULFOXIDE, S-METHYL MOLINATE, FONOFOS OXON, DESETHYL NAPROPAMIDE, AND PHOSMET OXON RESIDUES IN SOIL

I. SUMMARY/INTRODUCTION

This method is intended for determining S-methyl molinate, EPTC sulfoxide, butylate sulfoxide, fonofos oxon, desethyl napropamide, and phosmet oxon residues in soils at levels of 0.01 ppm to 0.50 ppm. All the analytes are metabolites of active ingredients of registered compounds. The table below gives the analyte, the active ingredient of which it is a metabolite, and the chemical name and structure of the analyte.

Analyte	Ingredient	Chemical Name	Structure
S-methyl Molinate	Molinate	S-methyl hexahydro-1H-azepine-1-carbothioate	
EPTC Sulfoxide	EPTC	S-ethyl dipropylthiocarbamate sulfoxide	
Butylate Sulfoxide	Butylate	S-ethyl diisobutylthiocarbamate sulfoxide	
Fonofos Oxon	Fonofos	O-ethyl S-phenyl ethylphosphonothioate	
Desethyl Napropamide	Napropamide	N-ethyl-2-(1-naphthalenyloxy)propionamide	
Phosmet Oxon	Phosmet	N-(mercaptomethyl) phthalimide S-(O,O-dimethylphosphorothioate)	

S-Methyl molinate, EPTC sulfoxide, butylate sulfoxide, fonofos oxon, desethyl napropamide, and phosmet oxon are extracted directly from soil with water and toluene. The toluene extract is analyzed for S-methyl molinate, EPTC sulfoxide, butylate sulfoxide, fonofos oxon, desethyl napropamide, and phosmet oxon by capillary gas chromatography with nitrogen-specific detection.

II. MATERIALS/METHODS

The equipment and reagents described below were used to generate the data and chromatograms presented in this report. Equipment with equivalent performance specifications and reagents of comparable purity can be used.

A. Apparatus

1. Gas Chromatograph. Hewlett-Packard Model 5880A, equipped with on-column injector inlet, Hewlett-Packard Model 7673A automatic

sampler, nitrogen-phosphorus detector, and electronic integrator or data acquisition system. Any chromatographic system giving equivalent performance can be used.

2. Chromatographic Column. J & W DB-1 (crosslinked methyl silicone), 15 m x 0.53 mm x 1.5 µm thickness, or equivalent.

3. Glass Bottles. Four-ounce, wide mouth bottles with aluminum foil lined caps.

4. Syringe. 10, 100, and 500 microliter capacities, Hamilton 701N, 710N, 750N or equivalent.

5. Reciprocating Shaker. Eberbach Corporation, model 6010 or equivalent.

6. Centrifuge. IEC International, model C1582 or equivalent.

B. Reagents

1. Solvents. Toluene, Acetone, Nanograde® or equivalent.

2. NaCl, Anhydrous Na_2SO_4. Reagent grade.

3. S-Methyl Molinate, EPTC Sulfoxide, Butylate Sulfoxide, Fonofos Oxon, Desethyl Napropamide, and Phosmet Oxon. Analytical reference-standards S-methyl molinate, EPTC sulfoxide, butylate sulfoxide, fonofos oxon, desethyl napropamide, and phosmet oxon. Available from ICI Americas Inc., 1200 So. 47th Street, Box 4023, Richmond, CA 94804-0023, Attention: Environmental Sciences Department Manager.

4. Calibration and Fortification Solution.

To prepare a stock solution weigh to the 4th decimal place a convenient quantity, e.g. 50 mg, of analytical reference standard of known purity into a suitably sized bottle. Calculate the weight of solvent to add, based on the weight of reference standard taken, the purity of the reference standard, the density of the solvent, and the desired solution concentration, typically 1000 µg/mL, as follows:

$$S = \frac{W \times P \times D}{A}$$

where S = the weight of solvent to add (g),

W = the weight of primary standard taken (mg std),

P = the purity of the primary standard (mg a.i./mg std),

D = the density of the solvent (g/mL),

and A = the desired solution concentration (mg a.i./mL solvent)

Add the calculated weight of the appropriate solvent to the bottle, close the bottle with a polyseal cap, and mix thoroughly to dissolve the primary standard. Use toluene (D = 0.867 g/mL) for calibration solutions, and acetone (D = 0.792 g/mL) for fortification solutions.

To prepare working calibration solutions, dilute the stock calibration solution by weight with toluene to give solutions that contain 1.0, 0.1, and 0.01 µg/mL of each analyte to be determined or other concentrations as required.

Dilute the stock fortification solution by weight with acetone to give solutions that contain 10 µg/mL of each analyte to be determined, or other concentrations as required.

As discussed in Section III.A below, an analyte may exhibit an enhanced response in sample matrix, as demonstrated by high recoveries from fortified control samples. In such cases, the calibration solutions may be prepared in sample extract solution to compensate for the response enhancement. Prepare calibration solutions in the sample matrix by either of two methods: 1) evaporate the toluene from a known volume of working calibration solution and take the residue up to the original volume with extract from an untreated control sample, or 2) add, via a syringe, the required amount of stock calibration solution to a known volume of extract from an untreated control sample. The amount added must be small enough relative to the extract volume that dilution is insignificant. The later method is preferred if the analyte is volatile.

C. Analytical Procedure

1. Extraction

Weigh 40.0 g of thoroughly-mixed soil sample into a 4-oz wide mouth bottle. Add 40 mL of distilled water, 10 g of NaCl, and 40 mL of toluene. Cap the bottle with an aluminum foil-lined lid and shake it on the reciprocating shaker for 2 hours. Centrifuge for 10-20 minutes at 2000 rpm to aid the separation of the phases. Alternatively, use any convenient weight of soil, 20 g or more, and extract with water and toluene in a soil:water:toluene w:v:v ratio of 1:½-1:1; confirm the validity of the extraction method by analysis of fortified control samples. Remove the top (toluene) phase for analysis. Dry stored extracts with anhydrous Na_2SO_4.

The validity of the method must be confirmed by analysis of appropriate control and fortified samples with each set of samples analyzed. If method validation recoveries are adequate without added NaCl, the use of NaCl is not required. Similarly, if chromatographic sensitivity and reproduceability are adequate with split or splitless injection modes, on-column injection is not required.

2. Fortification

Analyze unfortified and fortified control samples with each set of treated samples to demonstrate method recovery according to the Quality Assurance SOP. For example, for 40-g samples, weigh 40 g of untreated control soil into a 4-oz wide-mouth bottle. Add 0.040 mL of the 10 µg/mL acetone fortification solution to produce a fortification level of 0.01 ppm, or add 20 µL of the 1000 µg/mL acetone fortification solution to produce a fortification level of 0.5 ppm. Add water, NaCl, and toluene and extract as above. If a different weight of soil is analyzed, use that weight and adjust the volume or concentration of fortification solution to give the desired analyte concentration. Extract using the same amounts of water, salt (if required), and toluene as for the treated samples.

D. Instrumentation

1. Operating Conditions

Follow the manufacturer's instructions for operation of the gas chromatograph and nitrogen-selective detector. Use these parameters for the analyses or other operating conditions that achieve equivalent sensitivity, reproducibility, and resolution.

Inlet	On-column injection
Oven initial temp.	100°C
Initial time	0.05 min
Temp. programming rate	25°C/min
Oven final time	9 min
Oven final temperature	260°C
Injector temperature	OFF
Detector temperature	300°C
Carrier gas	Helium
Carrier gas pressure	3 psi
Carrier gas flow	12 mL/min
Injection size	3 µL
Quantitation	Peak height (external standard)
Makeup-gas (helium)	22.5 mL/min
Air	140 mL/min
Hydrogen	4 mL/min

Under the above conditions the elution times of the analytes range from 2.5 to 5.7 minutes. See Figure 1 for typical chromatograms.

2. Calibration

The gas chromatograph is calibrated using the analyte calibration solutions specified in section II.B.3. Chromatographic sensitivity is established by analysis of the 0.01 µg/mL calibration solution. Quantitation of residues at levels above the detection limit is done by an external standard procedure in which peak heights or areas of analyte peaks in sample extracts are compared to corresponding peak heights or areas of analyte peaks in calibration solutions. See Section G, below, for details of calculational methods.

3. Analysis of Extracts

Inject the sample extracts using the same conditions used for calibration. The identity of the analyte peak in the sample chromatogram is assigned based upon the coincidence of retention times (within 0.03 minutes) with those of the calibration chromatograms. If the response of a peak identified as an analyte exceeds that of the highest calibration solution, dilute the sample extract until its response is within the calibrated range, or extend the calibration range by injection of calibration solutions at higher concentration. Reinject the calibration solution after every two to four sample injections and recalibrate as needed. Reinject the calibration solution at completion of the sample analysis.

E. Interferences

No clean-up is required when this procedure is utilized as described. However, extractives from soil occasionally contribute peaks with retention times near those of an analyte. Satisfactory resolution can usually be achieved with appropriate oven temperature manipulations or column choice. Appendix A shows typical chromatograms. Analyze extracts of samples from untreated plots to demonstrate the absence of interferences from sample matrices, solvents, or labware. Typically, the active ingredient or parent compound may be present in any sample analyzed for a metabolite. Always confirm that the active ingredient and the metabolite do not co-elute under the conditions of analysis.

F. Confirmatory Techniques

Unexpected positive results, as in untreated control or pre-application samples, should be confirmed by other means, preferably by GC/MS, mass selective detection, or use of a second capillary column of different polarity.

G. Calculations

Calculations are done in one of two ways. If the response is linear, a factor can be calculated as described in 1 below. If the response is non-linear, or if the analyst prefers, the analyte responses over a range of calibration solution concentrations can be fit to a linear or an exponential curve, and a factor can then be calculated as in 2 below for each point on the curve that corresponds to an analyte response in an injection of sample extract.

1. Linear Response, Direct Calculation of Factor

a. Calibration Factors for Linear Response

F = the response factor for the analyte (ppm per electronic unit), calculated as follows:

$$F = \frac{C}{P \times S}$$

where C = the concentration of analyte in the calibration solution (µg/mL)

S = the amount of initial sample represented by each milliliter of final extract solution injected (g/mL)

P = the peak area or height (electronic units) of the analyte peak in the chromatogram of the calibration solution

Averaged response factors for multiple injections of calibration solutions and for more than one concentration of calibration solution can be used as appropriate in the calculation of the concentration of the analyte in the sample, as described below.

b. Analyte in Sample

The concentration of the analyte in the original sample is calculated using an external standard method as follows:

$$ppm = F \times R$$

where ppm = the amount of analyte in the soil in parts per million

R = the peak area or height (electronic units) of the analyte peak in the chromatogram of the sample extract

and F = the response factor for the analyte (ppm per electronic unit), calculated as described above

Note for the above external standard calculations, equal volumes of both the extract and the calibration solutions are injected.

2. Curve Fit for Linear or Non-Linear Response

If the instrumental response to injections of calibration solutions is reproducible and either linear or exponentially non-linear, a concentration-response curve can be used for sample quantitation. Any valid curve-fitting program can be used. Input the concentration-response for each injection of calibration

solution. The program will generate the formula for the corresponding linear or exponential curve. From the formula, determine the calculated concentration for each injection of calibration solution as described below. The calculated and actual concentrations should agree within 10 % relative; that is, the ratio of the actual to the calculated concentration should be between .9 and 1.1. If the agreement is adequate, calculate the concentration of analyte in the sample, and corresponding response factor as follows:

a. Linear Response:

The formula will be of form $Y = mX + b$, where

Y = the concentration of the analyte, ppm,

X = the analyte response, peak height or area units,

and

m and b = constants calculated by the curve-fit program.

Since the analyte concentration should be zero if the response is zero, the constant b should be zero if there are no systematic errors in the analysis. However, it is not necessary for b to be zero for the calculational method to be valid, as long as calibration solution responses are reproducible and the calculated concentrations of the calibration solutions are within 10 % of the actual concentrations.

For each sample injection, determine Y by using the response, X, in the formula.

Calculate the response factor, F, from the formula:

$$F = Y/X$$

Note that this factor should be the same for any point on a linear curve which passes through the intercept; $b = 0$.

b. Exponential non-linear response:

The curve will be of form $Y = aX^b$, where

Y = the concentration of the analyte, ppm,

X = the analyte response, peak height or area units,

and

a and b = constants calculated by the curve-fit program.

For each sample injection, determine Y by using the response, X, in the formula.

Calculate the response factor, F, from the formula:

$$F = Y/X$$

The response factor will be different for each point on the curve.

III. DISCUSSION

A. Accuracy

Fortified soil samples were prepared as described in Section II.C.2 and analyzed to establish the accuracy of the method. Six replicate 40-g soil samples were fortified with 20 micrograms of each analyte and eight replicate 40-g samples were fortified with 0.4 µg of each analyte to give 0.5 and 0.01 ppm, respectively. The results of the analyses are given in Tables 1 and 2 and summarized below.

Summary of Method Validation Results

Analyte	Fortification Amount (µg)	Mean Amount Found (µg)	Mean Percent Recovery
S-methyl Molinate	20.0	20.7	104
	0.40	0.430	107
	0.40	0.420	105(1)
EPTC Sulfoxide	20.0	19.7	96
	0.40	0.433	108(1)
	0.40	0.352	88
Butylate Sulfoxide	20.0	23.6	118
	0.40	0.556	139
	0.40	0.422	106(1)
Fonofos Oxon	20.0	19.6	98
	0.40	0.463	116
	0.40	0.390	97(1)
Desethyl Napropamide	20.0	20.0	100
	0.40	0.427	107
	0.40	0.412	103(1)
Phosmet Oxon	20.0	20.5	102
	0.40	0.585	146
	0.40	0.416	104(1)

(1) Analyzed using calibration solutions prepared in extracts of untreated soil to avoid response enhancement in soil extract matrix.

Recoveries for all analytes ranged from 98% to 118% at 0.5 ppm (20 µg). At 0.01 ppm (0.4 µg), recoveries ranged from 107% to 146% for analyses in which calibration solutions were not prepared in sample matrix. When analyses were done with calibration solutions prepared in sample matrix, recoveries at 0.01 ppm ranged from 88% to 106%. For the two analytes that showed a clear response enhancement in sample matrix at 0.01 ppm, butylate sulfoxide and phosmet oxon, mean recoveries dropped from 139% to 106% and from 146% to 104%, respectively. The response enhancement may be a function of the particular soil; calibration solutions should be prepared in untreated control sample extract as described in Section II.B.3 only if high recoveries (>120%) are obtained with calibration solutions prepared in toluene.

B. Precision

The precision of the method depends on variations in extraction and in instrumental analysis. The precision of the instrumental analysis was evaluated by triplicate injections of the extracts of samples fortified at 0.5 ppm. As the data in Table 1 show, the average relative variance, $100(X-\bar{X})/\bar{X}$, for triplicate injections of all the analytes was 3.8%. The mean coefficient of variation, including extraction variation, for 6 determinations at 0.5 ppm and 8 determinations at 0.01 ppm of each analyte was 2.2%.

C. Limit of Detection

The detection limit of the method is 0.01 ppm as determined by fortifications at the 0.01 ppm level with 2-3 cm peak heights.

D. Dry Weight Basis

This method determines the residues on an as-received basis. If it is desired to express the values on a dry-weight basis, compensation is necessary for water present in the sample. Percent moisture can be determined by drying a subsample at 105°C for 24 hours.

E. Safety Precautions

Personnel untrained in the routine safe-handling of chemicals and good laboratory practices should not attempt to use this procedure. In general, always wear safety glasses, work in a well ventilated area, avoid inhaling vapors, and avoid contact of any chemical with skin and clothing. Flammable solvents should be kept away from potential sources of ignition.

Toluene, Acetone
Flammable.
Avoid contact with skin and clothing.
Avoid breathing vapor; work in well ventilated area.

S-Methyl Molinate, EPTC Sulfoxide, Butylate Sulfoxide, Fonofos Oxon, Desethyl Napropamide, and Phosmet Oxon
Avoid contact with skin and clothing.
Work in well ventilated area.
Wash with soap and water after any accidental contact.

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IV. CONCLUSIONS

The method is specific for the analysis of S-methyl molinate, EPTC sulfoxide, butylate sulfoxide, fonofos oxon, desethyl napropamide, and phosmet oxon residues in soil. Only readily available laboratory equipment and reagents are required. The analysis can be completed by one person in an 8-hour period if an adequately homogenized sample is available. Untreated and fortified untreated samples should be extracted and analyzed with each set of samples to demonstrate absence of interferences and adequate recovery. If determination of any analyte at a concentration other than 0.01 ppm to 0.50 ppm is required, suitably fortified samples must be analyzed to validate the method at that concentration.

V. CERTIFICATION

This is to certify that this is a complete and unaltered report prepared by the Environmental Science Department of ICI Americas Inc., Western Research Center.

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VI. TABLES AND FIGURES

A. Table 1. Recoveries of S-Methyl Molinate, EPTC Sulfoxide, Buty..te Sulfoxide, Fonofos Oxon, Desethyl Napropamide, and Phosmet Oxon from Soil Fortified with 20 µg (0.5 ppm) of Analyte.

B. Table 2a. Recoveries from Soil Fortified with 0.4 µg (0.01 ppm) of Analyte; Toluene Calibration Solutions.

C. Table 2b. Recoveries from Soil Fortified with 0.4 µg (0.01 ppm) of Analyte; Matrix Calibration Solutions.

Table 1

Recoveries of S-Methyl Molinate, EPTC Sulfoxide, Butylate Sulfoxide, Fonofos Oxon, Desethyl Napropamide, and Phosmet Oxon from Soil Fortified with 20 µg (0.5 ppm) of Analyte

Analyte	Sample Number	µg Found			Mean	RV[1]	Percent Recovery
		Run #1	Run #2	Run #3			
S-methyl molinate	65	20.9	20.5	20.6	20.7	1.0	103
	67	21.4	20.3	20.8	20.8	2.6	104
	78	21.1	20.6	19.8	20.5	3.2	103
	87	21.5	20.8	19.9	20.7	3.9	104
	94	21.2	20.8	19.4	20.5	4.6	102
	99	22.6	21.2	19.8	21.2	6.6	106
				MEAN	20.7	3.7	104
				CV	1.2		
EPTC sulfoxide	65	20.5	21.0	18.6	20.0	6.3	100
	67	21.4	21.5	20.5	21.1	2.6	106
	78	20.0	20.6	18.4	19.7	5.8	98
	87	20.4	18.0	18.9	19.1	6.3	95
	94	20.8	19.4	19.4	19.9	4.1	99
	99	19.3	18.1	18.5	18.6	3.3	93
				MEAN	19.7	4.7	99
				CV	4.3		
Butylate sulfoxide	65	24.0	24.4	22.3	23.6	4.7	118
	67	24.2	24.8	23.0	24.0	3.8	120
	78	24.0	24.8	22.1	23.6	5.9	118
	87	24.1	23.0	23.1	23.4	2.6	117
	94	24.0	23.6	23.6	23.7	1.3	119
	99	24.0	23.0	22.7	23.2	2.9	116
				MEAN	23.6	3.5	118
				CV	1.1		
Fonofos oxon	65	19.6	19.7	19.6	19.6	0.3	98
	67	20.0	19.6	19.9	19.8	1.0	99
	78	19.9	19.5	19.1	19.5	2.1	98
	87	19.8	19.5	19.0	19.4	2.1	97
	94	20.2	19.1	18.4	19.2	4.7	96
	99	20.9	20.1	18.9	20.0	5.3	100
				MEAN	19.6	2.5	98
				CV	1.4		

Table 1
(cont.)

Analyte	Sample Number	µg Found Run #1	Run #2	Run #3	Mean	CV	Percent Recovery
Desethyl napropamide	65	20.3	19.4	19.7	19.8	2.3	99
	67	20.4	20.2	19.7	20.1	1.8	101
	78	20.8	20.0	19.5	20.1	3.3	101
	87	20.9	19.4	20.1	20.1	3.7	101
	94	21.2	19.5	18.8	19.8	6.2	99
	99	21.8	20.4	18.7	20.3	7.6	102
				MEAN	20.2	4.2	100
				CV	1.0		
Phosmet Oxon	65	20.4	20.5	18.9	19.9	4.5	100
	67	21.1	21.5	19.7	20.8	4.6	104
	78	21.6	21.7	19.5	20.9	5.9	105
	87	20.8	19.3	20.4	20.2	3.9	101
	94	20.9	20.6	20.2	20.6	1.7	103
	99	21.3	20.2	19.7	20.4	4.0	102
				MEAN	20.5	4.1	102
				CV	1.8		

1) RV = relative variance for three injections of the same sample extract. $100(X-\bar{X})/\bar{X}$.

2) CV = Coefficient of variation for analyses of six samples. $100s/\bar{x}$.

Table 2a

Recoveries from Fortified Soil with 0.4 µg (0.01 ppm) of Analyte
Toluene Calibration Solution

Analyte	Fortification	Found µg	Percent Recovery
S-methyl molinate	A	0.424	106
	B	0.432	108
	C	0.424	106
	D	0.440	110
	E	0.436	109
	F	0.436	109
	G	0.424	106
	H	0.420	105
	MEAN	0.430	107
	CV	1.7	
EPTC sulfoxide	A	0.408	102
	B	0.440	110
	C	0.436	109
	D	0.448	112
	E	0.420	105
	F	0.455	114
	G	0.428	107
	H	0.424	106
	MEAN	0.433	108
	CV	3.6	
Butylate sulfoxide	A	0.540	135
	B	0.556	139
	C	0.552	138
	D	0.572	143
	E	0.568	142
	F	0.572	143
	G	0.552	138
	H	0.536	134
	MEAN	0.556	139
	CV	2.5	
Fonofos oxon	A	0.452	113
	B	0.460	115
	C	0.456	114
	D	0.476	119
	E	0.464	116
	F	0.468	117
	G	0.452	113
	H	0.472	118
	MEAN	0.463	116
	CV	2.0	

Table 2a
(cont.)

Analyte	Fortification	Found µg	Percent Recovery
Desethyl napropamide	A	0.420	105
	B	0.420	105
	C	0.420	105
	D	0.424	106
	E	0.440	110
	F	0.436	109
	G	0.420	105
	H	0.436	105
	MEAN	0.427	107
	CV	2.0	
Phosmet oxon	A	0.580	145
	B	0.612	153
	C	0.576	144
	D	0.580	145
	E	0.592	148
	F	0.596	149
	G	0.556	139
	H	0.584	146
	MEAN	0.585	146
	CV	2.8	

Table 2b

Recoveries from Fortified Soil with 0.4 µg (0.01 ppm) of Analyte; Matrix Calibration Solution

Analyte	Fortification	Found µg	PPM	Percent Recovery
S-methyl molinate	A	0.404	0.010	101
	B	0.412	0.010	103
	C	0.420	0.010	105
	D	0.432	0.011	108
	E	0.428	0.011	107
	F	0.424	0.011	106
	MEAN	0.420	0.011	105
	CV	2.3		
EPTC sulfoxide	A	0.368	0.009	92
	B	0.384	0.010	96
	C	0.372	0.009	93
	D	0.356	0.009	89
	E	0.320	0.008	80
	F	0.312	0.008	78
	MEAN	0.352	0.009	88
	CV	8.2		
Butylate sulfoxide	A	0.444	0.011	111
	B	0.440	0.011	110
	C	0.440	0.011	110
	D	0.444	0.011	111
	E	0.384	0.010	99
	F	0.380	0.010	95
	MEAN	0.422	0.011	106
	CV	6.5		
Fonofos oxon	A	0.396	0.010	99
	B	0.404	0.010	101
	C	0.400	0.010	100
	D	0.400	0.010	100
	E	0.368	0.009	92
	F	0.372	0.009	93
	MEAN	0.390	0.010	97
	CV	3.9		

Table 2b
(cont.)

Analyte	Fortification	Found µg	PPM	Percent Recovery
Desethyl-naprcpamide	A	0.428	0.011	107
	B	0.420	0.011	105
	C	0.404	0.010	101
	D	0.444	0.011	111
	E	0.424	0.011	106
	F	0.412	0.010	103
	MEAN	0.412	0.010	103
	CV	3.4		
Phosmet oxon	A	0.400	0.010	100
	B	0.404	0.010	101
	C	0.400	0.010	100
	D	0.404	0.010	101
	E	0.420	0.010	105
	F	0.400	0.010	100
	MEAN	0.416	0.010	104
	CV	7.1		

VII. REFERENCES

WRC Laboratory Notebook

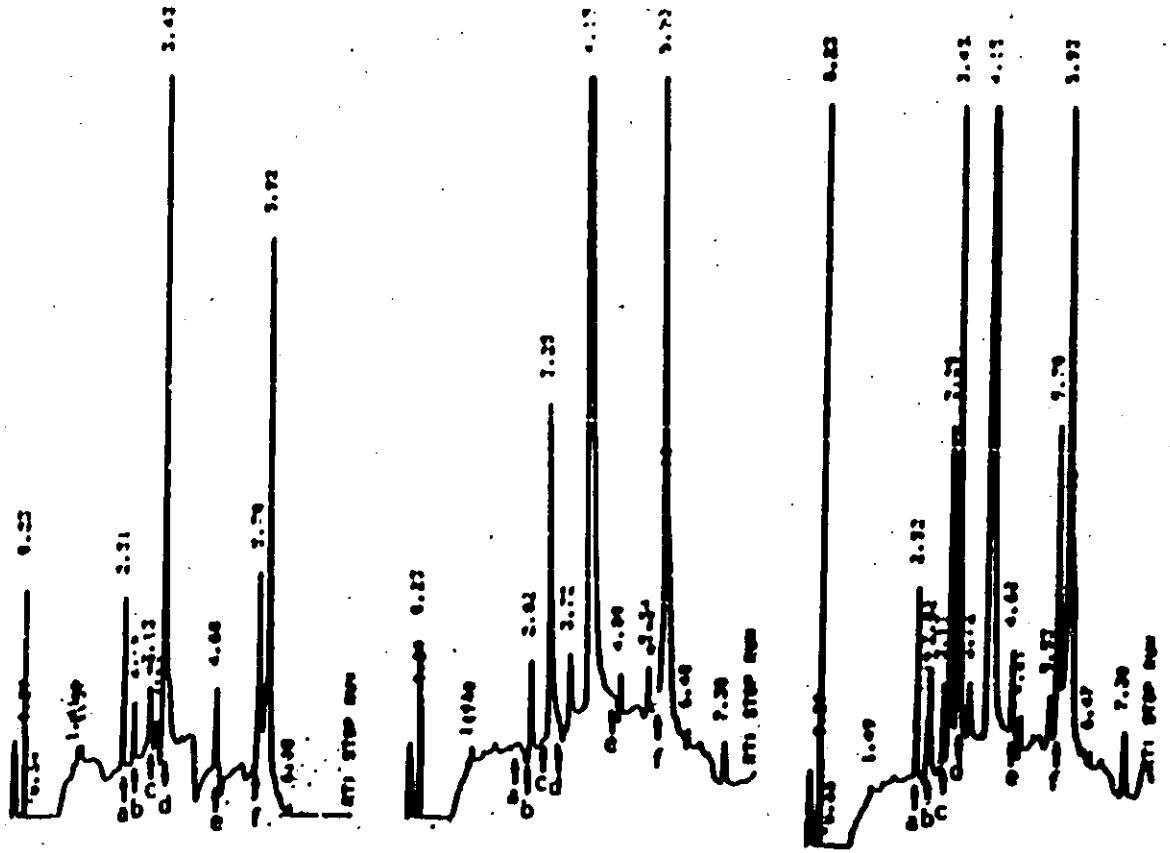
11735
11853
11903

M14:89-07

APPENDIX A

Representative Chromatograms

a = S-methyl Molinate
b = LPTC Sulfoxide
c = Butylate Sulfoxide
d = Fonofos Oxon
e = Desethylnapropamide
f = Phosmet Oxon



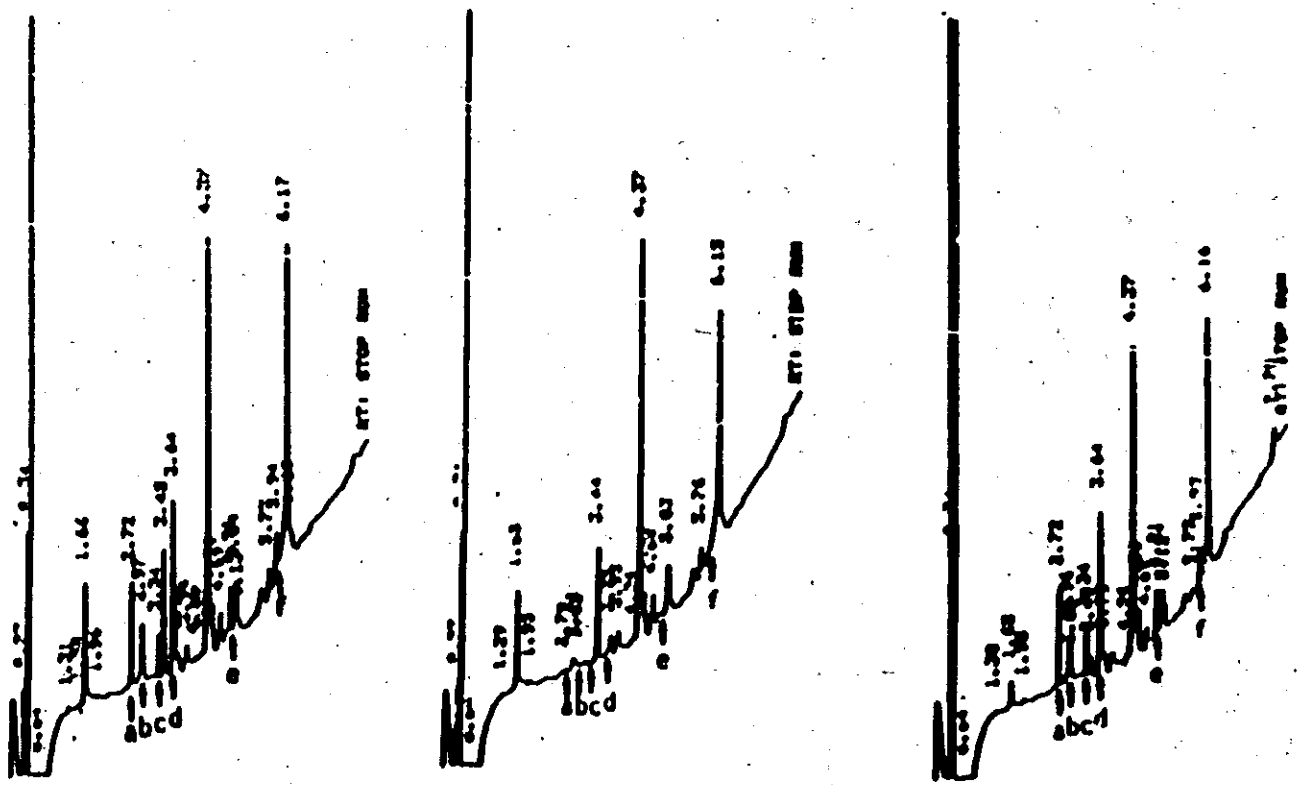
Calibration Solution
0.01 ug/mL
Each Analyte

Untreated Control

Fortification A
0.01 ppm
Each Analyte

Figure 1. Typical Chromatograms, Toluene Calibration Solution

a = S-methyl Molinate
b = EPTC Sulfoxide
c = butylate Sulfoxide
d = Fonofos Oxon
e = Desethylnapropamide
f = Phosmet Oxon



Calibration Solution
0.01 ug/mL
Each Analyte

Untreated Control

Fortification A
0.01 ppm
Each Analyte

Figure 2. Typical Chromatograms, Matrix Calibration Solution