Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Fonofos

MRID #: 440062-01

Matrix: Water

Analysis: GC/MS

440062-01

Study Title

Fonofos: Determination of Fonofos, Fonofos Oxon and Methylphenylsulfone in Water by Gas Chromatography and Mass-selective Detection

Data Requirement

Supplemental

Author

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Study Completion Date

April 30, 1996

Performing Laboratory

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Study Number

FONO-95-AM-02

Report Number

RR 96-030B

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No claim of confidentiality is 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).

Company Zeneca Inc.

Submitter Andrew A. Davidson Date 4/29/96

Regulatory Product Manager Andrew Davidson

Title Signature

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This study has been conducted in compliance with the United States of America Environmental Protection Agency's FIFRA Good Laboratory Practice Standards (GLP).

This study meets the requirements of 40 CFR Part 160 (July 1, 1995).

I believe that this study is valid for the purposes for which it was conducted and that this report is a true reflection of the raw data generated.

Study Director Sabrina C. Leung 4-30-96

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Study Number: FONO-95-AM-02

Report Title: FONOFOS: Determination of Fonofos, Fonofos Oxon, and Methylphenylsulfone in Water by Gas Chromatography and Mass-Selective Detection

Report No.: RR 96-030B

QUALITY ASSURANCE STATEMENT

In accordance with ZENECA Inc. policy and procedures for complying with the provisions of the U.S. EPA's FIFRA Good Laboratory Practice Standards (40 CFR PART 160, August 17, 1989), the conduct of this study was inspected/audited by the Quality Assurance Unit at the Western Research Center, Richmond, California, United States of America.

Date	Inspection/Audit	Report Date
Mar 8, 1996	Protocol, Draft	Mar 8, 1996
Mar 8, 1996	Study Conduct	Mar 8, 1996
Apr 29, 1996	Final Report & Data	Apr 29, 1996

So far as can be reasonably established, the methods described and results incorporated in this report accurately reflect the raw data produced during the study.

William Y. Ja
Group Leader, Quality Assurance Unit

4/29/96
Date

Certification of Authenticity

I, the undersigned, declare that this study was performed under my direction and that this report represents a true and accurate record of the results obtained.

Study Director Sabrina C. Leung

Sabrina C. Leung
Principal Research Chemist
Zeneca Inc.

4-30-96
Date

The following person performed work on this study:

Sabrina C. Leung protocol preparation, sample preparation, analysis, and report author

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Diana G. Graham
Group Leader
Dietary Exposure and Environmental Fate
Zeneca Inc.

4/29/96
Date

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Study Title: Fonofos: Determination of Fonofos, Fonofos Oxon and Methylphenylsulfone in Water by Gas Chromatography and Mass-selective Detection

Author: Sabrina C. Leung

1 Summary/Introduction

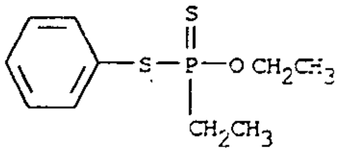
1.1 Scope

A method for the analysis of fonofos, fonofos oxon and methylphenylsulfone in water has been laboratory validated. The concentration range over which this method has been validated extends from 0.1 µg/L to 10 µg/L (parts per billion) in water for each of the compounds listed above. The method detection detection limit (MDL) for each analyte is listed in Table 2.

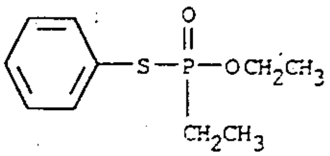
The purpose of this report is to describe an analytical method for the determination of fonofos and its degradates, fonofos oxon and methylphenylsulfone in water. This report also documents a validation study on the method and validation data obtained from the study.

1.2 Principles

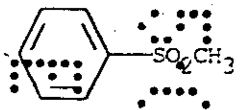
Fonofos is the active insecticidal ingredient in the various formulated products marketed by Zeneca, Inc. under the trademark "DYFONATE®". The Chemical Abstracts Name for fonofos is phosphonodithioic acid, ethyl O-ethyl S-phenyl ester [944-22-9]. The chemical name for fonofos oxon is phosphonothioic acid, ethyl O-ethyl S-phenyl ester [944-21-8]. Methylphenylsulfone is a metabolite that has been identified in a soil-metabolism study. The chemical name for methylphenylsulfone is methylsulfonylbenzene [3112-85-4]. The chemical structures are given below.



Fonofos



Fonofos oxon



Methylphenylsulfone

To determine these compounds, a sample of water is extracted with a solvent mixture of toluene:methylene chloride 5:1. Sodium bisulfite is added to the water to eliminate degradation of the analytes. The toluene-methylene chloride extract is concentrated and analyzed for fonofos, fonofos oxon and methylphenylsulfone by gas chromatography with mass-selective detection.

Method validation was conducted according to Good Laboratory Practice regulations and Zeneca Ag Products standard operating procedures.

2 Materials/Methods

The equipment and reagents described below were used to generate the data and chromatograms presented in this report. Equipment capable of providing equivalent sensitivity and selectivity, and reagents of comparable purity can be used.

2.1 Apparatus

2.1.1 Gas Chromatograph

Hewlett-Packard model 5880A designed for use with capillary columns. The gas chromatograph is equipped with a Hewlett-Packard Model 7673 automatic injector, mass-selective detector (HP Model 59970C MS Chemstation), and data acquisition system (HP Chemserver System).

2.1.2 Gas-Purification Traps

Alltech 8121 Inline Gas Purifier and an Alltech OXY-TRAP® for removing oxygen from the helium carrier gas.

2.1.3 Gas-Chromatographic Column

DB-17 fused silica capillary, 10 m by 0.18 mm i.d. with a 0.3-µm film thickness (J&W catalog no. 121-1713).

2.1.4 Syringes

10-µL capacity (Hamilton 701N) syringe for autosampler; 10-, and 50-µL capacity Gas-Tight syringes (Hamilton 1700 series) for fortification and standard preparation.

2.1.5 Laboratory Shaker

Eberbach 6010 two speed reciprocating shaker with utility box carrier, VWR Cat. No. 57007-101.

2.1.6 Ultrasonic Cleaner

Branson Model No. B-22-4, stainless-steel tank 2.8 liter capacity, tank size 5" L x 9" W x 4"D.

2.1.7 Evaporator

The Meyer N-EVAP analytical evaporator model 112 from Organomation Associates Inc.

2.1.8 Vortex Mixer

Thermolyne mixer model M-16715.

2.1.9 Evaporation Tubes

15 mL capacity, graduated, Kimax catalog No. 45176.

2.1.10 Glass Bottles

8-oz. narrow-mouth bottles equipped with TEFLON-lined screw-caps, and 1-oz narrow-mouth bottle equipped with Polyseal®-lined screw-caps.

2.1.11 Glass Pipettes

1-, 2-, 5- and 10-mL disposable, graduated, glass pipettes for general use.

2.1.12 Calibrated Laboratory Pipettor

Oxford Pipettor Model 10/30, adjustable dispensing range 10 to 30 mL in 1 mL graduations.

2.2 Reagents and Standards

2.2.1 Solvents

Toluene, methylene chloride, methanol and deionized water. Fisher OPTIMA grade toluene, OPTIMA grade methylene chloride, OPTIMA grade methanol, and Millipore Milli-Q® filtered water are acceptable. All solvents must be of high purity and suitable for use in trace organic analyses by gas chromatography.

2.2.2 Helium

High-purity gas supplied to gas chromatograph via lines equipped with gas purification traps and appropriate pressure regulators.

2.2.3 Analytical Reference Standards

The following reference standards are available from Zeneca Ag Products, 1200 South 47th Street, Box Number 4023, Richmond, CA 94804-0023:

FONOFOS :	ASW01094-01R; 99.7% w/w purity
FONOFOS OXON:	ASW01703-01F; 95% w/w purity
METHYLPHENYLSULFONE:	ASW01638-01R; 99.4% w/w purity

2.2.4 Sodium Bisulfite

Analytical grade.

2.2.5 Anhydrous Sodium Sulfate

Analytical grade.

2.2.6 Calibration Solutions

Calibration solutions are used to calibrate the instrument. To prepare a stock calibration solution of an analyte, at nominal concentration of 1.0 mg/mL, place a known quantity (± 0.1 mg) of approximately 25 mg of the analyte of a primary standard of known purity into a clean 20-mL beaker. Add toluene to the beaker to dissolve the analyte. Quantitatively transfer the analyte solution into a clean 25-mL volumetric flask, and dilute to volume with the appropriate solvent. Stopper the volumetric flask, and mix the contents thoroughly. Transfer the contents into a 1-oz bottle, cap the bottle with a Polyseal®-lined cap and keep in a refrigerator when not in use. Calculate the concentration of the stock solution as follows:

$$C = \frac{(W \times P)}{25}$$

where C = concentration of analyte in final solution (mg/mL)

W = weight of primary standard (mg)

P = purity of reference standard (100% = 1.00)

25 = volume of solvent (mL)

To prepare working standard solution for calibration purposes, dilute by volume and combine the calibration stock solution of each analyte, to give a solution mix at concentration of 10 µg/mL each analyte in toluene. This working standard solution is used to prepare daily-use calibration standards in matrix-matched solvent mixture of toluene:methylene chloride 5:1. Matrix-matched calibration standards are recommended to reduce the effects of matrix on the GC/MSD response. Use microliter syringes, clean disposable glassware, and the matrix-matched toluene:methylene chloride 5:1 solvent to make serial dilutions of the working standard solution to give 1.0, 0.5, 0.1, 0.05, 0.01, and 0.005 µg/mL standard solutions in matrix-matched toluene:methylene chloride solvent.

2.2.7 Fortification Solutions

Fortification solutions are used to fortify samples and demonstrate procedural recoveries. To prepare a stock fortification solution of an analyte, at nominal concentrations of 3.0 mg/mL, place a known quantity (± 0.1 mg) of approximately 75 mg of the analyte of a primary standard of known purity into a clean 20-mL beaker. Add methanol to the beaker to dissolve the analyte. Quantitatively transfer the analyte solution into a clean 25-mL volumetric flask, and dilute to volume with the appropriate solvent. Stopper the volumetric flask, and mix the contents thoroughly. Transfer the contents into a 1-oz bottle, cap the bottle with a Polyseal®-

lined cap and keep in a refrigerator when not in use. Calculate the concentration of the stock solution as follows:

$$C = \frac{(W \times P)}{25}$$

where C = concentration of analyte in final solution (mg/mL)

W = weight of primary standard (mg)

P = purity of reference standard (100% = 1.00)

25 = volume of solvent (mL)

To prepare working standard solution for fortification purposes, dilute by volume and combine the fortification stock solution of each analyte, to give a solution mix at concentration of 500 µg/mL each analyte in methanol. Further dilute this fortification solution by volume to give a diluted fortification solution at concentration of 5.0 µg/mL each analyte in methanol.

3 Procedures

3.1 Extraction of Water Samples

3.1.1 Use a graduated cylinder to add 200 mL of water sample into a 8-ounce bottle. Add 20 mg sodium bisulfite (0.01%) to the water sample. Use a calibrated pipettor to add 25.0 mL of toluene, and use a pipette to add 5.0 mL of methylene chloride into the sample bottle. Seal the sample bottle with the Teflon-lined cap. Shake the sample bottle by hand for a few times to mix the solvents, and sonicate the contents in an ultrasonic cleaner for 20 minutes. Extract the sample by shaking on a reciprocating shaker at high speed for 1 hour. After shaking, place the bottle in its upright position for a minimum of 5 minutes. Allow the organic phase of toluene/methylene chloride to separate from the water phase.

3.1.2 Remove the Teflon-lined cap from the sample bottle. Use a disposable Pasteur pipette to transfer at least 15 mL of the top toluene/methylene chloride extract layer into a 1-oz. glass bottle. Dry the extract with ~4 grams of anhydrous sodium sulfate.

3.1.3 Use a disposable pipette to transfer 10.0 mL of the dried toluene/methylene chloride extract into a clean, graduated evaporation tube. Evaporate to 1.0 mL with a gentle stream of nitrogen using the N-EVAP Model 112 evaporator. Do not evaporate the extract to dryness. If the evaporated volume fall below 1.0 mL, add sufficient solvent mixture of toluene:methylene chloride 5:1 to make up to a final volume of 1.0 mL in the evaporation tube.

3.1.4 Vortex mix the final 1.0 mL extract in the evaporation tube for 1 minute and transfer the extract into an autosampler vial for gas chromatographic analysis.

3.2 Preparation of Fortified Water Samples

Analyze unfortified and fortified control samples with each sample set to demonstrate method recovery. For example, place 200 mL of untreated control water into a 8-ounce bottle. Use a µL-syringe to add 4 µL of fortification solution (5.0 µg/mL) to the control sample to produce a fortification level of 0.1 µg/L. Extract as detailed in section 3.1 above. It is recommended that one unfortified and one fortified sample be analyzed for every set of 10 or less field samples.

3.3 GC Conditions

Follow the manufacturer's instructions for operation of the gas chromatograph and mass-selective detector. The specific conditions listed below were used to generate the data and chromatograms presented in this report.

3.3.1 Instrument

Hewlett-Packard model 5880A designed for use with capillary columns and temperature programming of the column oven. The gas chromatograph is equipped with a Hewlett-Packard Model 7673 automatic injector, mass-selective detector and data acquisition system (Hewlett-Packard Model 59970C MS Chemstation and associated software).

3.3.2 Operating Conditions

Column:	DB-17 fused silica capillary, 10 m X 0.18 mm i.d., 0.3 µm film thickness, Cat. 121-1713

Carrier gas: helium

Column head-pressure: ~5 lb/sq. inch

Inlet type: splitless with double restrictor (R&D Separations, Rancho Cordova, CA) with ~ 3 mm silanized borosilicate Pyrex glass wool plug

Inlet temperature: 230°C

Interface temperature: 240°C

Initial oven temperature: 100°C

Initial time: 0.0 min

Oven temperature program rate: 25°C/min

Final oven temperature: 270°C

Final oven temperature time: 5 min

Volume injected: 3 µL

Splitless valve off: 0.5 min

Total run time: 7.0 min

3.3.3 Mass-selective Detector

Mode: low resolution selective-ion-monitoring (s.i.m.)

Dwell time: 100 msec

Tuning: optics optimized for m/z 69, 100 and 131 with perfluorotributylamine using the HP-supplied manual program

Quantitation: Peak height; external standard

Mass monitored: Methylphenylsulfone m/z 94 & 156,
Fonofos oxon m/z 93 & 230,
Fonofos m/z 109 & 246,

Using the above conditions the elution time of methylphenylsulfone is 3.86 min., fonofos oxon is 5.11 min and fonofos is 5.41 min. See Figures 1 and 3 for typical gas chromatograms.

3.4 Calibration

Calibrate the gas chromatograph by using the 1.0, 0.5, 0.1, 0.05, 0.01 and 0.005 µg/mL calibration stadndard solutions. Prepare a calibration curve by plotting response versus standard concentration using the HP Chemserver data acquisition system and an appropriate curve fit program. Figures 4 - 6 show examples of typical calibration curves. After every four to six samples, analyze one or more of the calibration solutions to assure that the calibration curve is still valid. After sample analysis has been completed, re-analyze all calibration solutions to ensure the validity of the entire calibration range.

3.5 Analysis of Sample Extracts

Inject the sample extracts on the same day of extraction and calibration, using the same conditions used for the calibration solutions. The identity of the analyte peak in the sample chromatogram is assigned based upon the coincidence of retention times (within 0.05 min) with those of the calibration chromatogram. Reinject the calibration solution after injection of every four to six sample extracts and at the end of the chromatographic run. Calculate the concentration of the analyte in the sample extract by use of a standard curve program or from the average calibration response factor as described in Section 6.

4 Interferences

Analyze extracts from untreated water samples to demonstrate the absence of interferences from samples matrices, solvent and labware.

5 Confirmatory Techniques

Unexpected positive results, as in control samples, should be confirmed by other means. Confirmation can be achieved by quantitation using a different m/z ion or by using a different type of detector, such as a flame photometric detector with a sulfur bandpass filter.

6 Calculations

The concentration of the analyte in the original sample is calculated by using the external standard method, i.e., the response obtained for the analyte in the sample extract is compared to the response obtained from a separate injection of a calibration solution. To use the calculation methods shown below, the injection volumes for all calibration solutions and sample extracts must be fixed at the same volume.

6.1 Linear Detector Response

6.1.1 Calibration Factor

Calculate the response factor, RF, for injection of a calibration solution as follows:

$$RF = \frac{C_{std}}{R_{std}}$$

Where

C_{std} = concentration in µg/mL of the calibration solution
R_{std} = response units (e.g., peak height or peak area) from detector for the calibration solution

6.1.2 Concentration of Analyte in Water Sample

Determine the concentration of each analyte in the original water sample, C_i, from the corresponding average response factor, RF_{avg}, the extraction volume, and the concentration factor as follows:

$$C_i\ (\mu g/L) = R_i(\text{sample}) * RF_{avg} * V_1 / V_2 / S$$

Where

R_i(sample) = analyte response unit from detector for the sample extract
RF_{avg} = average response factor over the entire range of calibration for the corresponding analyte
V_1 = volume of extract, mL = 30 mL
V_2 = volume of water extracted, L = 0.2 L

S = concentration factor for the extract = 10

6.2 Nonlinear Detector Response

6.2.1 Calibration Curve

Generate a multiple point calibration curve by plotting the concentration of the calibration solution (C_{std}) as the y-axis and the corresponding response units from the detector (R_{std}) as the x-axis for a range of the analyte concentrations. Examples of calibration curves for fonofos, fonofos oxon and methylphenylsulfone are shown in Figures 4 - 6. Use an appropriate curve fit equation such as the power curve equation to best fit the calibration curve. Take the response units from the detector for the analyte in the sample extract, R_i(sample), and determine the concentration of the analyte in the sample extract, C_{ext}, by using the corresponding calibration curve.

6.2.2 Concentration of Analyte in Water Sample

Determine the concentration of each analyte in the original water sample, C_i, from the corresponding concentration of the analyte in the samples extract (C_{ext}), the extraction volume, and the concentration factor as follows:

C_i (µg/L) = $C_{ext} * V_1 / V_2 / S$

Where

C_{ext} = analyte concentration in the sample extract, µg/mL

V_1 = volume of extract, mL = 30 mL

V_2 = volume of water extracted, L = 0.2 L

S = concentration factor for the extract = 10

7 Results and Discussions

7.1 Method Accuracy

Fortified deionized water samples were prepared as described in Section 3.2 and analyzed according to this method to establish accuracy. Seven replicates were prepared at each of the levels of 0.1, 1.0 and 10 µg/L. The fortified samples were then analyzed according to procedure described in Section 3. Recovery data are presented in Table 1. The average recoveries for fonofos, fonofos oxon and methylphenylsulfone were 115.6 %, 109.8 % and 77.2%, respectively.

7.2 Method Precision

The precision of the method depends on variations in sampling, extraction, and instrumental analysis. The variations in extraction and instrumental analysis can be evaluated from the data obtained during analyses of fortified samples. The coefficients of variation, CVs, for 21 analyses as summarized in Table 1 were 9.4%, 9.0 % and 8.5% for fonofos, fonofos oxon and methylphenylsulfone, respectively.

7.3 Freedom from Interferences

An unfortified control water samples was extracted and taken through the entire analytical procedure. No significant interferences were found, and no residues of the test analytes (fonofo, fonofos oxon and methylphenylsulfone) were detected in the control water sample analyzed with the recovery tests. Figures 2 and 3 show sample chromatograms for the analysis of fonofos, fonofos oxon and methylphenylsulfone in control (unfortified) and fortified water samples.

7.4 Method Detection Limit

The method detection limit (MDL) for each test parameter can be determined by using the procedures published in the Code of Federal Regulations (Reference 2). Seven aliquots of reagent water were fortified, as described in Section 3.2, and analyzed according to this method to determine MDL. Table 2 lists the fortification level at 0.1 µg/L and the MDL obtained for each analyte.

7.5 Limit of Determination

The limit of determination (LOD) for the method is 0.1 µg/L for each of the test parameter in a 200-mL water sample as established by fortifications at the 0.1 µg/L level.

8 Safety Considerations

Personnel untrained in the routine safe handling of chemicals must not attempt to use this procedure. Information on any first aid procedures can be found in the Material Safety Data Sheets accompanying the chemical or available from the chemical supplier. In general, always wear safety glasses with side shields. Work in well ventilated areas. Avoid inhaling vapors; and avoid contact of the chemicals with skin and clothing. Keep flammable solvents away from potential sources of ignition.

9 Conclusions

The method is specific for the analysis of fonofos, fonofos oxon and methylphenylsulfone in water down to 0.1 ppb (µg/L) level. Only commercially available laboratory equipment and reagents are required. The analysis can be completed by one person in an 8-hour period. Untreated and fortified untreated samples should be extracted and analyzed with each set of samples to demonstrate absence of interferences and adequate recovery.

The method has been validated at the concentration range of 0.1 to 10 µg/L. If determination of fonofos, fonofos oxon and methylphenylsulfone at a concentration other than 0.1 to 10 µg/L is required, suitably fortified samples must be analyzed to validate the method at that concentration.

10 Tables and Figures

Table 1. Recoveries of Fonofos, Fonofos Oxon and Methylphenylsulfone from Water

Sample Number	Compound µg/L Added	FONOFOS		FONOFOS OXON		METHYLPHENYLSULFONE	
		µg/L Found	% Recovery	µg/L Found	% Recovery	µg/L Found	% Recovery
15738-33-1	0.1	0.1174	117.4	0.1088	108.8	0.0810	81.0
15738-33-2	0.1	0.1190	119.0	0.0980	98.0	0.0766	76.6
15738-33-3	0.1	0.1231	123.1	0.1149	114.9	0.0845	84.5
15738-33-4	0.1	0.1253	125.3	0.1135	113.5	0.0807	80.7
15738-33-5	0.1	0.1228	122.8	0.1111	111.1	0.0772	77.2
15738-33-6	0.1	0.1214	121.4	0.1079	107.9	0.0914	91.4
15738-33-7	0.1	0.1289	128.9	0.1164	116.4	0.0876	87.6
15738-29-1	1.0	1.1395	114.0	1.0845	108.5	0.7960	79.6
15738-29-2	1.0	1.0000	100.0	0.9490	94.9	0.7300	73.0
15738-29-3	1.0	0.9610	96.1	0.9510	95.1	0.7240	72.4
15738-29-4	1.0	1.0780	107.8	1.0700	107.0	0.7890	78.9
15738-29-5	1.0	1.0490	104.9	1.0270	102.7	0.7930	79.3
15738-29-6	1.0	1.0760	107.6	1.0330	103.3	0.7850	78.5
15738-29-7	1.0	1.0990	109.9	1.0860	108.6	0.7950	79.5
15738-24-1	10	10.7250	107.3	10.5210	105.2	6.4950	65.0
15738-24-2	10	12.9710	129.7	12.8760	128.8	7.8500	78.5
15738-24-3	10	10.1210	101.2	10.3530	103.5	7.0100	70.1
15738-24-4	10	11.8720	118.7	11.4170	114.2	6.9530	69.5
15738-24-5	10	11.1000	111.0	10.7500	107.5	6.8120	68.1
15738-24-6	10	12.4370	124.4	12.0290	120.3	6.9860	69.9
15738-24-7	10	13.6400	136.4	13.5080	135.1	7.9360	79.4
15738-24-8	0.0	< 0.1	–	< 0.1	–	< 0.1	–
n =			21		21		21
Mean Recovery:			115.6%		109.8%		77.2%
Std. Deviation :			10.82		9.91		6.53
Coeff. of Variation (CV):			9.4%		9.0%		8.5%

Table 2. d Detection Limits (MDL) for the Analysis of Fonofons, Fonofos Oxon thylphenylsulfone in Water

Sample Number	Compound µg/L Added	FONOFOS µg/L Found	FONOFOS OXON µg/L Found	METHYLPHENYLSULFONE µg/L Found
15738-33-1	0.1	0.1174	0.1088	0.0810
15738-33-2	0.1	0.1190	0.0980	0.0766
15738-33-3	0.1	0.1231	0.1149	0.0845
15738-33-4	0.1	0.1253	0.1135	0.0807
15738-33-5	0.1	0.1228	0.1111	0.0772
15738-33-6	0.1	0.1214	0.1079	0.0914
15738-33-7	0.1	0.1289	0.1164	0.0876
S = std. dev. of the replicate analyses =		0.00384	0.00617	0.00544
MDL = 3.143 x S =		0.0121	0.0194	0.0171

Procedure Reference:

Appendix B to Part 136 of U.S. EPA 40 CFR (July 1, 1995) - Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11

Figure 1. Typical gas chromatogram for fonofos, fonofos oxon, and methylphenylsulfone analysis (0.1 mg/mL calibration standard solution)

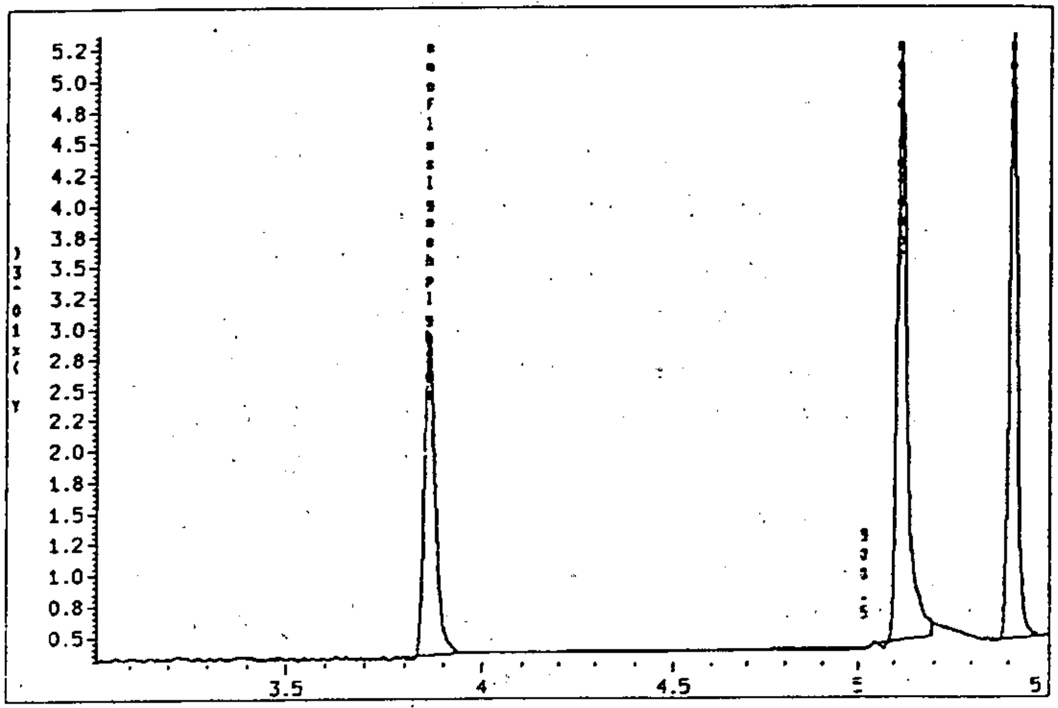


Figure 2. Typical gas chromatogram for fonofos, fonofos oxon, and methylphenyl sulfone analysis (control sample)

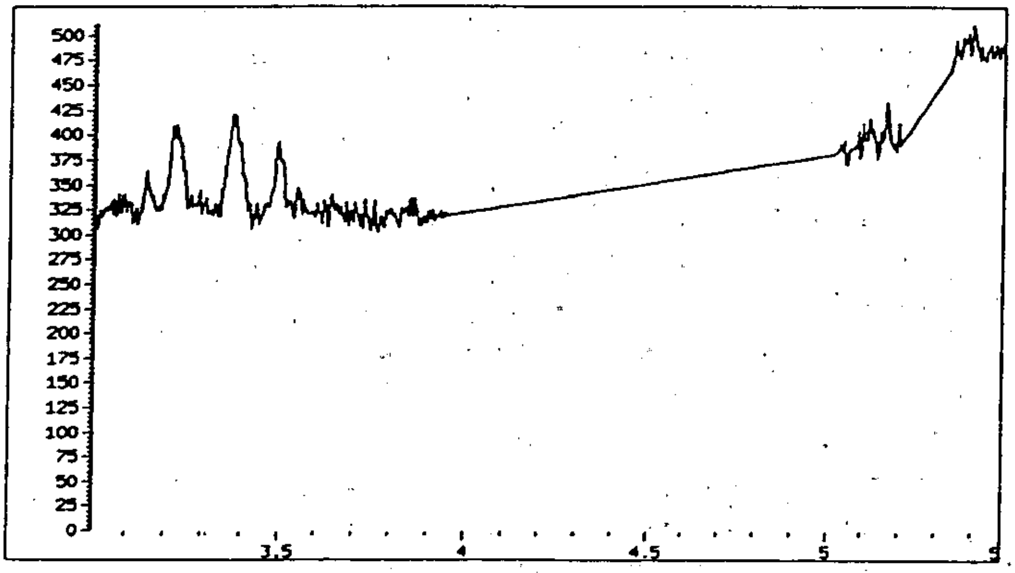


Figure 3. **Typical gas chromatogram for fonofos, fonofos oxon and methylphenylsulfone analysis (water sample fortified at 0.1 µg/L)**

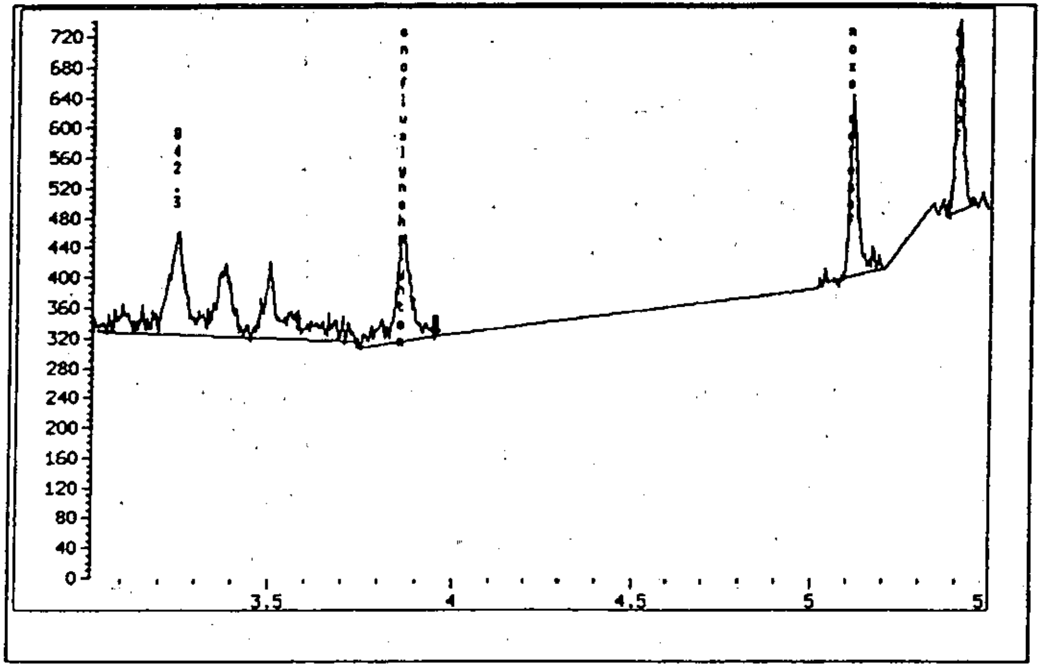


Figure 4. Typical calibration curve for fonofos analysis

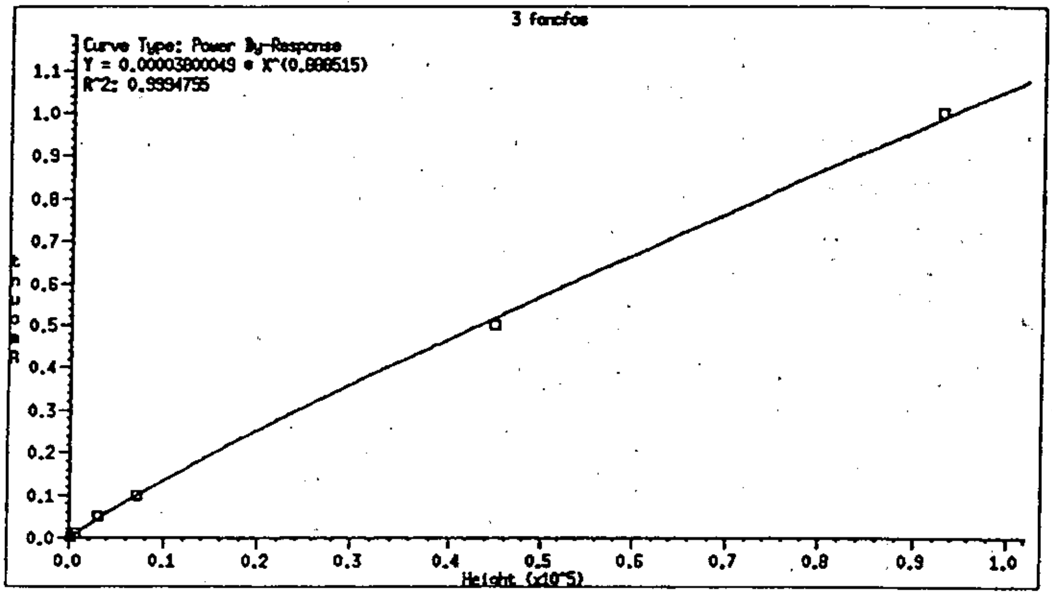


Figure 5. Typical calibration curve for fonofos oxon analysis

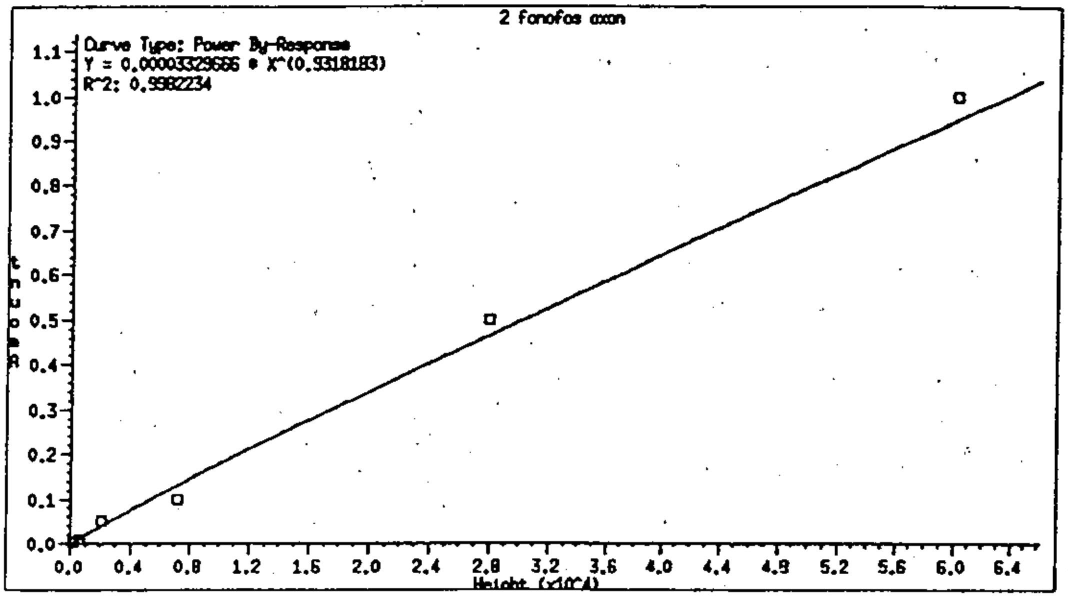
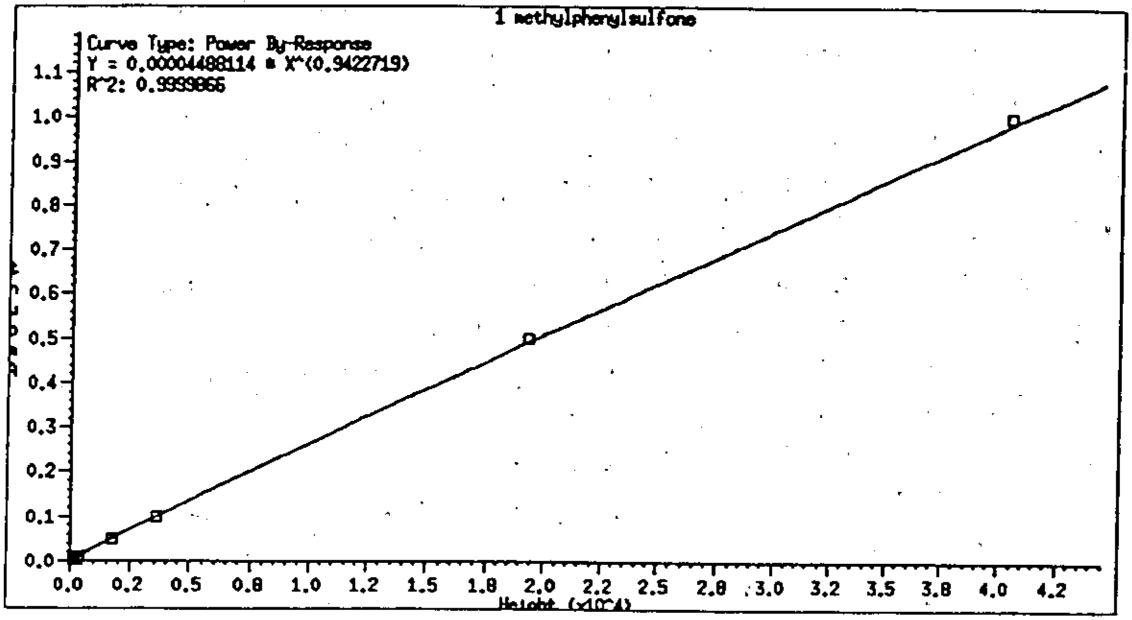


Figure 6 . Typical callibration curve for mehylphenylsulfone analysis



11 Retention of Records

All of the raw data, the protocol, and the final report are located in the GLP Archive of Zeneca Ag Products, Western Research Center, 1200 S. 47th Street, Richmond, CA 94804-0023, U.S.A.

12 References

1. Laboratory Notebook References: 15738-17 to -38.
2. "Definition and Procedure for the Determination of the Method Detection Limit", the <u>Code of Federal Regulations,</u> 40 CFR Part 136, Appendix B (July 1, 1995 revision).