Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Flumetsulam

MRID #: 419521-07

Matrix: Soil

Analysis: GC/MS

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STUDY TITLE

DETERMINATION OF RESIDUES OF DE-498 IN SOIL BY CAPILLARY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

DATA REQUIREMENTS

Pesticide Assessment Guidelines - 164-1
FIFRA Phase Technical Guidance- 164-1
Target Crop

AUTHORS

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DATE

April 29, 1991

PERFORMING LABORATORY

North American Environmental Chemistry Laboratories
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LABORATORY PROJECT ID

ACR 91.3

05-1061

Flumetsulam

EPA

MRID

NUMBER

41521-07

STATEMENT OF NO CONFIDENTIALITY CLAIMS

Title: DETERMINATION OF RESIDUES OF DE-498 IN SOIL BY CAPILLARY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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

COMPANY: DowElanco

COMPANY AGENT: Dennis H. Lade

TITLE: Product Registration Manager

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DATE: June 18, 1991

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Title: DETERMINATION OF RESIDUES OF DE-498 IN SOIL BY CAPILLARY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

This method was not developed under Good Laboratory Practice Requirements as identified in 40 CFR 160.12. However it was developed in the spirt of Good Laboratory Practice standards and all records are on file for retrieval. Also the Validation procedure conducted by a second laboratory was conducted under GLP.

Edward L Olberding — June 14 1991

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ACR #: 91.3
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DETERMINATION OF RESIDUES OF DE-498 IN SOIL BY CAPILLARY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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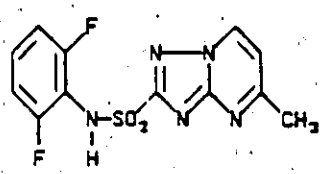
1. Scope

This method is applicable for the quantitative determination of DE-498 (N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo-[1,5a]-pyrimidine-2-sulfonamide) in soil at a validated lower level of quantitation of 2.5 ppb.



N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo-[1,5a]-pyrimidine-2-sulfonamide (DE-498)

2. Principle

DE-498 residues are extracted from soil using a 90% acetone/10% 0.1 N hydrochloric acid solution. Following evaporation of the acetone, the sample is diluted with 0.005 N hydrochloric acid and purified using a C_{18} solid-phase extraction (S-P-E). The eluent from the S-P-E is evaporated to dryness, and the residue reconstituted with acetonitrile. The sample is then derivatized with methyl iodide to form the N-methyl derivative. The derivatized sample solution is evaporated to dryness, reconstituted with toluene containing N-d3-methyl DE-498 as an internal standard and analyzed by capillary gas chromatography/mass spectrometry GC/MS

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3. Safety Precautions

a. Each analyst should be acquainted with the potential hazards of the reagents, products, and solvents used in this method before commencing laboratory work. SOURCES OF INFORMATION INCLUDE: MATERIAL SAFETY DATA SHEETS, LITERATURE, AND OTHER RELATED DATA. Safety information on non-DowElanco products should be requested from the supplier. Disposal of reagents, reactants, and solvents must be in compliance with local, state, and federal laws and regulations.

b. Acetone, acetonitrile, methanol, methyl-t-butyl ether, toluene, and triethylamine are flammable and should be used in well-ventilated areas away from ignition sources.

4. Equipment

a. Gas chromatograph, Model 5890A, Hewlett-Packard, Avondale, PA 19311.

b. Automatic sampler, Model 7673A, Hewlett-Packard, Avondale, PA 19311.

c. Mass selective detector, Model 5971A, Hewlett-Packard, Palo Alto, CA 94304.

d. Mass spectrometer data system, Model 59970, Hewlett-Packard, Palo Alto, CA 94304.

e. Balance, analytical, Model AE200, Mettler Instrument Corp., Hightstown, NJ 08520.

f. Balance, pan, Model BB2440, Mettler Instrument Corp.

g. Centrifuge, with head to accomodate 10-dram vials, Model Centra-8, International Equipment Company, Needham Heights, MA 02194.

h. Desiccator, 250 mm I.D. with Drierite adsorbent, Catalog Number 08-595E, Fisher Scientific, Pittsburg, PA 15219.

i. Evaporator, N-Evap, Model 111, Organomation Associates, Inc., South Berlin, MA 01549.

Set at a water bath temperature of 40°C and a nitrogen flow rate of 200 mL/min.

j. Oven, Model OV-490A-2, Blue M Electric Company, Blue Island, IL 60406.

k. Shaker, variable-speed reciprocating with box carrier, Model 6000, Eberbach Corp., Ann Arbor, MI 48106.

l. Ultrasonic bath, Model 1200, Branson Cleaning Equipment Company, Shelton, CT 06484.

m. Vacuum manifold box, Model spe-21, J. T. Baker Chemical Company, Phillipsburg, NJ 08865.

n. Vial crimper, Part Number 8710-0979, Hewlett-Packard, Avondale, PA 19311.

o. Vortex mixer, Model G-560, Scientific Industries, Inc., Bohemia, NY 11716.

5. Glassware and Materials

a. Column, capillary gas chromatography, Durabond-17 liquid phase, 10 m x 0.18 mm i.d., 0.3 µm film thickness, Catalog Number 121-1713, J&W Scientific, Folsom, CA 95630.

b. Column inlet liner, deactivated, Catalog Number 5181-3315, Hewlett-Packard, Avondale, PA 19311.

c. Column, C_{18} S-P-E, Catalog Number 7020-07, J. T. Baker Chemical Company. (Note 16.b)

d. Cylinder, graduated, 2000 mL, Catalog Number 131-9058, National Scientific Company, Lawrenceville, Georgia 30245.

e. Dish, weighing, Catalog Number 08-732, Fisher Scientific.

f. Gas, helium, 99.995% purity, Scott Specialty Gases, Troy, MI 48083.

g. Gas, nitrogen, technical grade, Scott Specialty Gases.

h. Moisture trap, Catalog Number 7971, Chrompack, Inc., Raritan, NJ 08869. (Note 16.c.)

i. Charcoal scrubber, Catalog Number 7972, Chrompack, Inc. (Note 16.c.)

j. Oxygen trap, Catalog Number 7970, Chrompack, Inc. (Note 16.c.)

k. Syringes, 10, 50, and 500 µL, Model 700 Series, Hamilton Company, Reno, NV 89520.

l. Vials, 2 dram, with poly(tetrafluoroethylene)-lined screw caps, Catalog Number B7800-3, National Scientific Company.

m. Vials, 10 dram, with poly(tetrafluoroethylene)-lined screw caps, Catalog Number B7800-6, National Scientific Company.

n. Vials, autosampler, 2 mL, Catalog Number C4011-2, National Scientific Company.

o. Vial seals, Catalog Number C4011-1A, National Scientific Company.

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6. Reagents and Chemicals

a. Acetone, acetonitrile, methanol, methyl-t-butyl ether, and toluene (Optima Grade), Fisher Scientific.

b. Hydrochloric acid, 0.1 N, reagent grade, certified concentration, Fisher Scientific.

c. Sodium chloride, ACS reagent grade, Fisher Scientific.

d. Hydrochloric acid, 0.005 N.

Prepare by diluting 50 mL of 0.1 N hydrochloric acid to volume in a 1000-mL volumetric flask with distilled/deionized water.

e. Sodium chloride, 5% (w/v).

Prepare by dissolving 50 grams of sodium chloride in distilled/deionized water in a 1000-mL volumetric flask. Adjust to volume with distilled/deionized water.

f. 90% acetone/10% 0.1 N hydrochloric acid solution.

Prepare by pouring 200 mL of 0.1 N hydrochloric acid into a 2000-mL graduated cylinder. Add 1500 mL of acetone, swirl the cylinder, and allow to equilibrate to room temperature. Adjust to volume with acetone.

g. Methyl iodide, minimum 99.5% purity, Catalog Number 28,956-6, Aldrich Chemical Company, Milwaukee, WI 53233.

h. Methyl iodide, stable-isotope labeled, $^{12}CD_3I$, Catalog Number 29,675-9, Aldrich Chemical Company.

i. Triethylamine, minimum 99% purity, Catalog Number 13,206-3, Aldrich Chemical Company.

j. Water, distilled/deionized, Corning MEGA-PURE Still, Model MP-12A, Corning Glass Works, Science Products Division, Corning, NY 14831.

k. Standard

N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo-[1,5a]-pyrimidine-2-sulfonamide (DE-498), analytical standard.[a/]

[a/] Obtain from Sample Coordinator, DowElanco, P.O. Box 1706, Midland, Michigan 48641-1706.

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7. Preparation of Standards

a. Preparation of Calibration Standards/Spiking Solutions

(1) Dissolve 0.1000 gram of DE-498 analytical standard in acetone in a 100-mL volumetric flask. Dilute to volume to obtain a 1000 µg/mL stock solution.

(2) Dilute 5 mL of the above 1000 µg/mL solution to 1000 mL with acetone in a 1000-mL volumetric flask to obtain a 5.0 µg/mL (5.0 ng/µL) initial solution.

(3) Solutions for spiking soil samples are prepared by diluting the initial solution from Section 7.a.(2) above with acetone as follows:

Aliquot of Initial Soln.	Final Soln. Volume	Spiking Soln. Final Conc.	Equivalent Sample Conc.
mL	mL	ng/mL	ppb
5.0	2000	12.5	2.5
10.0	2000	25.0	5.0
20.0	2000	50.0	10.0
50.0	2000	125.0	25.0
10.0	200	250.0	50.0
20.0	200	500.0	100.0
50.0	200	1250.0	250.0

(4) Solutions for calibration standards are prepared by pipeting 1.0 mL of the DE-498 standards in Section 7.a.(3) above into 2-dram vials and derivatizing according to the procedure described in Section 9, steps k through w.

b. Preparation of Internal Standard Solution

(1) Pipet 2.0 mL of the 1000 µg/mL DE-498 stock solution from Section 7.a.(1) into a 2-dram vial.

(2) Evaporate the solution to dryness using an N-Evap evaporator.

(3) Add 1.0 mL of acetonitrile, cap the vial, and sonicate for 5-10 seconds.

(4) Add 50 µL of triethylamine and 50 µL of stable-isotope labeled methyl iodide (Section 6.h), cap the vial, and sonicate for 5-10 seconds.

(5) Allow the mixture to react with the methyl iodide for 30 minutes at room temperature.

(6) Evaporate the solution to dryness using an N-Evap evaporator.

(7) Add 1.0 mL of a 5% sodium chloride solution, cap the vial, and sonicate for 5-10 seconds.

(8) Add 5.0 mL of methyl-t-butyl ether, cap the vial, and vortex the sample for 5-10 seconds.

(9) Centrifuge the vial for 5 minutes at 2500 rpm.

(10) Carefully transfer the methyl-t-butyl ether layer to a clean 10-dram vial.

(11) Repeat Steps 8-9 three additional times, combining the methyl-t-butyl ether layers in the 10-dram vial.

(12) Evaporate the solution to dryness using an N-Evap evaporator.

(13) Add 20 mL of acetone, cap the vial, and sonicate for 5-10 seconds.

(14) Transfer the acetone to a 200-mL volumetric flask.

(15) Rinse the 10-dram vial again with 20 mL of acetone, and transfer the acetone to the 200-mL volumetric flask.

(16) Dilute the solution to volume with acetone. This solution contains 10.0 µg/mL N-d3-methyl DE-498.

(17) Dilute 10.0 mL of the above 10.0 µg/mL solution to 1000 mL with toluene in a 1000-mL volumetric flask to obtain a 0.100 µg/mL (0.100 ng/µL) solution.

8. Gas Chromatography/Mass Spectrometry

a. Column

Install the splitless liner (5.b) and the capillary column (5.a) on the split/splitless injection port of the GC/MS following the manufacturer's recommended procedure.

b. Typical operating conditions for the determination of DE-498 by capillary GC/MS:

Instrumentation: Hewlett-Packard Model 5890A Gas Chromatograph / Model 5971A Mass Selective Detector

Column: J&W Scientific fused silica capillary Durabond-17 liquid phase
10 m x 0.18 mm I.d.
0.30 µm film thickness

Temperatures:

Column	120°C for 1.1 minutes 120°C to 325°C at 20°C/minute 325°C for 5.65 minutes
Injector	320°C
Interface	310°C

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Carrier Gas:	helium
Head Pressure	100 kPa
Linear Velocity	25 cm/sec
Injection Mode:	splitless
Purge Delay	1.0 minutes
Splitter Flow	50 mL/min
Septum Purge:	1.0 mL/min
Injection Volume:	2 µL
Ions Monitored:	N-methyl DE-498
	m/z 134 (base peak ion) (M^+-205; see Figure 1)
	m/z 142 (M^+-197; see Figure 1)
	N-d3-methyl DE-498 (internal standard)
	m/z 145 (M^+-197; see Figure 2)
Electron Multiplier:	1800 volts

c. A typical calibration curve is shown in Figure 3.

d. Typical chromatograms of a standard, control sample, and a 2.5 ppb recovery sample are shown in Figures 4-6, respectively.

9. Recovery of DE-498 from Soil

a. Weigh 5.0-gram portions of control soil into a series of 10-dram vials.

b. For preparing fortified samples, use part of the samples as controls and fortify the remaining samples by adding 1.0-mL aliquots of the appropriate spiking solutions (Section 7.a.(3)) in acetone to obtain concentrations ranging from 2.5 to 250 ppb.

c. Add 25 mL of a 90% acetone/10% 0.1 N hydrochloric acid extracting solution.

d. Cap the vial and sonicate the sample for 30-45 seconds.

e. Shake the sample for a minimum of 2 hours on a reciprocating shaker at approximately 180 excursions/minute.

f. Centrifuge the sample container for 10 minutes at 2500 rpm.

g. Transfer the acetone/hydrochloric acid solution to a clean 10-dram vial.

h. Evaporate the acetone using an N-Evap evaporator.

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i. Add 15.0 mL of 0.005 N hydrochloric acid, cap the vial, and sonicate the sample for 10-15 seconds.

j. The sample is then purified using the following S-P-E procedure (Note 16.b):

(1) Place a C_{18} S-P-E column on the vacuum manifold box.

(2) Rinse the S-P-E column with 5 mL of methanol.

(3) Condition the S-P-E column with 5 mL of 0.005 N hydrochloric acid. (Do not allow the column bed to dry.)

(4) Transfer the sample solution from Step 9.1 to the S-P-E column and, with the aid of vacuum, slowly pull the sample through the column. Without allowing the column bed to dry, wash the sample vial with a 10 mL aliquot of 0.005 N hydrochloric acid and transfer the wash to the S-P-E column.

(5) Thoroughly dry the S-P-E column by drawing air through it for approximately 45 minutes.

(6) Remove the S-P-E column from the vacuum manifold box and elute the DE-498 by passing 2.5 mL of methanol through the S-P-E column, collecting the eluent in a 2-dram vial. (Note 16.d)

k. Evaporate the solution to dryness using an N-Evap evaporator.

l. Add 500 µL of acetonitrile, cap the vial, and sonicate for 5-10 seconds.

m. Add 10 µL of triethylamine and 10 µL of methyl iodide, cap the vial, and sonicate for 5-10 seconds.

n. Allow the sample to react with the methyl iodide for 30 minutes at room temperature.

o. Evaporate the solution to dryness using an N-Evap evaporator.

p. Add 1.0 mL of a 5% sodium chloride solution, cap the vial, and sonicate for 5-10 seconds.

q. Add 5.0 mL of methyl-t-butyl ether, cap the vial, and vortex the sample for 5-10 seconds.

r. Centrifuge the vial for 5 minutes at 2500 rpm.

s. Carefully transfer the methyl-t-butyl ether layer to a 2-dram vial.

t. Evaporate the solution to dryness using an N-Evap evaporator.

u. Add 1.0 mL of toluene containing the N-d3-methyl DE-498 internal standard, cap the vial, and sonicate for 5-10 seconds.

v. Centrifuge the vial for 5 minutes at 2600 rpm.

w. Transfer the solution to a 2-mL autosampler vial. Seal the vial with a cap and crimper.

x. Analyze the sample by capillary gas chromatography/mass spectrometry.

10. Determination of Percent Recovery of DE-498

a. Inject the calibration standards described in Section 7.a.(4) and determine the peak areas at *m/z* 134 and *m/z* 142 for methylated DE-498 and at *m/z* 145 for d3-methylated DE-498.

For each standard calculate the DE-498 confirmation ratio. The average confirmation ratio for all of the calibration standards will be used to confirm the presence of DE-498 in the soil samples.

For example, using the data from Figure 4:

$$\text{Confirmation Ratio} = \frac{\text{peak area at } m/z\ 142}{\text{peak area at } m/z\ 134}$$

$$\text{Confirmation Ratio} = \frac{42425}{76598}$$

$$\text{Confirmation Ratio} = 0.55387$$

Positive confirmation of the presence of DE-498 is indicated when the confirmation ratio for the samples is in the range of ± 10% of the average found for the standards.

b. Prepare a standard curve by plotting the equivalent DE-498 concentration on the abscissa (x-axis) and the *m/z* 134/145 peak peak area ratio on the ordinate (y-axis) as shown in Figure 3. Using regression analysis, determine the equation for the curve with respect to the abscissa.

For example, using power regression with the data from Figure 3

$$Y = \text{constant} \times X^{(\text{exponent})}$$

$$X = \left(\frac{Y}{\text{constant}} \right)^{1/\text{exponent}}$$

$$\text{DE-498 Conc. (ppb)} = \left(\frac{m/z\ 134/145 \text{ peak area ratio}}{\text{constant}} \right)^{1/\text{exponent}}$$

$$\text{DE-498 Conc. (ppb)} = \left(\frac{m/z\ 134/145 \text{ peak area ratio}}{0.089690} \right)^{1/0.963885}$$

c. Determine the net concentration in each recovery sample by first subtracting the average DE-498 peak area ratio in the control samples from that of the recovery sample. Substitute the peak area ratio obtained into the above equation and solve for the concentration.

$$\text{DE-498 Conc. (ppb)} = \left(\frac{\text{net m/z 134/145 peak area ratio}}{0.089690} \right)^{1/0.963885}$$

$$\text{DE-498 Conc. (ppb)} = \left(\frac{0.210707-0.000000}{0.089690} \right)^{1/0.963885}$$

DE-498 Conc. = 2.43 ppb

d. Determine the percent recovery by dividing the net concentration of each recovery sample by the theoretical concentration added.

$$\text{Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100\%$$

$$\text{Recovery} = \frac{2.43 \text{ ppb}}{2.50 \text{ ppb}} \times 100\%$$

Recovery = 97%

11. Determination of DE-498 in Soil Samples

a. Prepare control, recovery, and treated samples as described in Section 9.

b. Prepare a standard curve and determine the DE-498 concentration in the recovery samples as described in Section 10.

c. Determine the concentration in each treated sample by substituting the DE-498 m/z 134/145 peak area ratio obtained into the equation for the standard curve and solve for the concentration.

For example, using the data from Figure 6:

$$\text{DE-498 Conc. (ppb)} = \left(\frac{\text{m/z 134/145 peak area ratio}}{\text{constant}} \right)^{1/\text{exponent}}$$

$$\text{DE-498 Conc. (ppb)} = \left(\frac{0.210707}{0.089690} \right)^{1/0.963885}$$

DE-498 Conc. = 2.43 ppb

12. Determination of Soil Moisture

a. Accurately weigh a 10-gram portion of soil into a tared weighing dish.

b. Place the sample in an oven at 130°C and allow to dry for a minimum of 16 hours.

c. Remove the sample from the oven, place in a desiccator until the sample has cooled to room temperature, and then re-weigh.

d. Calculate the percent moisture (dry weight basis) as follows:

$$\text{Percent Moisture (dry weight basis)} = \frac{\text{water, g}}{\text{dry soil, g}} \times 100$$

$$= \frac{\text{sample weight before drying} - \text{sample weight after drying}}{\text{sample weight after drying}} \times 100$$

13. Determination of Corrected DE-498 in Soil

a. Determine the DE-498 concentration in the soil samples as described in Section 11.

b. Determine the soil moisture as described in Section 12.

c. Determine the corrected DE-498 concentration in soil samples as follows:

$$\text{DE-498 Conc. (corrected ppb)} = \text{DE-498 Conc. (ppb)} \times \frac{100}{\%\ \text{Recovery}} \times \left(1 + \frac{\%\ \text{Moisture}}{100}\right)$$

14. Precision Statement

Recovery values of DE-498 from samples of soil fortified over the concentration range of 2.50 to 250 ppb averaged 90% with one standard deviation equal to 7% (Table I).

15. Discussion

a. Since the mass spectrum of N-methyl DE-498 shows that primarily lower mass fragments are formed, the mass selective detector tuning was optimized for lower mass sensitivity. Using the "USERTUNE" feature of the mass selective detector and the standard perfluorotributylamine tuning compound, the mass spectrometer tuning was conducted at m/z 69, 100, and 131. Although not necessary for obtaining the sensitivity required to analyze samples at the low end of the validated range, tuning the instrument in this manner often doubled the sensitivity for the ions monitored in this method.

b. During the course of analyzing several hundred soil samples using the method described above, it was found that the DE-498 chromatographic peak shape would remain sharper for longer periods of time when sample matrix was present *vs.* the absence of sample matrix (*i.e.* calibration standards). Breaking off a small section (approx. 25-50 cm) of the capillary column from the injection port side of the column was found to remedy the problem. An alternative solution that also worked very well was to fortify control soil sample extracts with the appropriate DE-498 standard after the C_{18} S-P-E (Section 9.j.(6). This technique had the advantage of minimizing any sample matrix effects on the chromatographic process and subsequent quantitation of DE-498.

16. Notes

a. Equipment, glassware, materials, reagents, and chemicals considered to be equivalent to those specified may be substituted with the understanding that their performance must be confirmed by appropriate tests. Common laboratory supplies are assumed to be readily available and are, therefore, not listed here.

b. Variation in the C_{18} S-P-E columns may influence the elution profile of the DE-498. It is necessary to obtain an elution profile of each lot of S-P-E column used to ensure optimum clean-up efficiency.

c. The scrubber/traps are used in the gas supply lines to purify helium entering the gas chromatograph.

d. Depending on the number of samples being prepared, one may elute the DE-498 from each S-P-E column individually, using either gravity-feed or pressurized elution, or as a group, using the vacuum manifold box.

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TABLE I. RECOVERY OF DE-498 FROM SOIL.

ppb		Control Soil Sample Number[a]	Percent Recovery
Added	Found		
2.50	2.29	261252	92
2.50	2.00	261252	80
2.50	2.63	261244	105
2.50	2.46	261244	98
2.50	2.16	261245	86
2.50	2.43	261253	97
5.00	4.28	261252	86
5.00	4.59	261252	92
5.00	4.64	261244	93
5.00	4.49	261244	90
5.00	4.41	261245	88
5.00	4.49	261253	90
10.0	8.47	261252	85
10.0	8.91	261252	89
10.0	9.46	261244	95
10.0	10.0	261244	100
10.0	8.92	261245	89
10.0	9.34	261253	93
25.0	22.0	261252	88
25.0	22.2	261252	89
25.0	18.0	261244	72
25.0	17.3	261244	69
25.0	25.0	261245	100
50.0	45.0	261252	90
50.0	46.8	261252	94
50.0	43.7	261244	87
50.0	42.3	261244	85
100.0	91.6	261252	92
100.0	92.8	261252	93
250.0	235	261252	94
250.0	227	261252	91
			90 ± 7 [b]

[a] Several samples of soil were used for controls. There was no DE-498 detected in any of the soil samples used for spiking.

[b] Mean ± 1 standard deviation (n=31).

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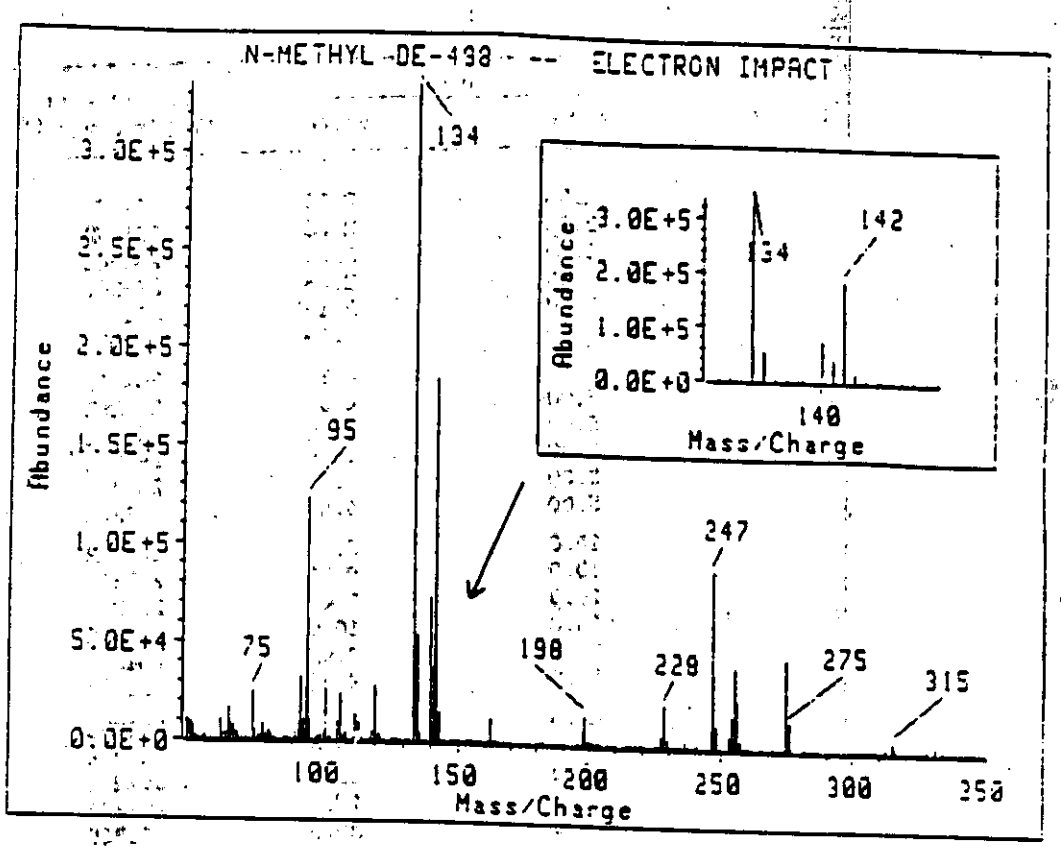
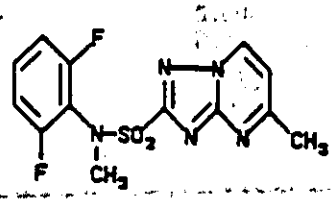
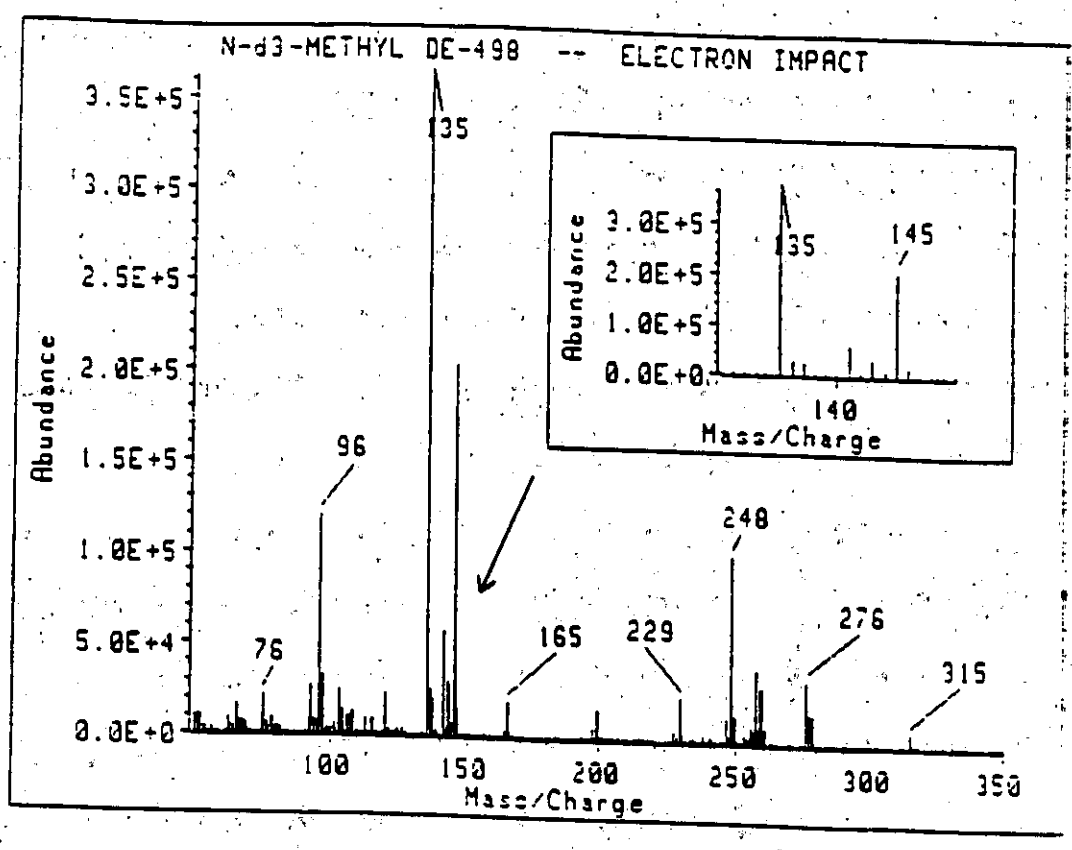
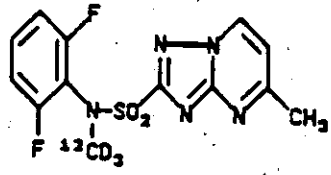




N-Methyl DE-498
Molecular Weight: 339

FIGURE 1. MASS SPECTRUM OF THE N-METHYL DERIVATIVE OF DE-498.





N-d3-Methyl DE-498

Molecular Weight: 342

FIGURE 2. MASS SPECTRUM OF THE N-d3-METHYL DERIVATIVE OF DE-498.

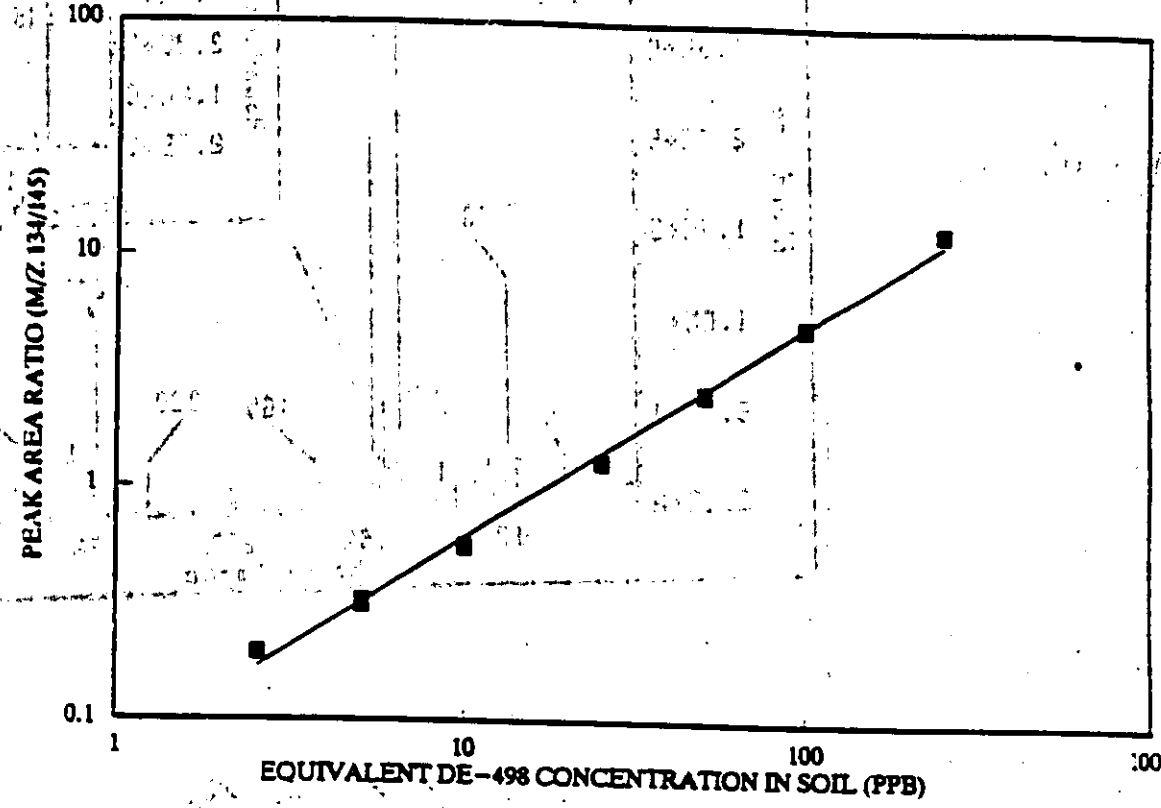
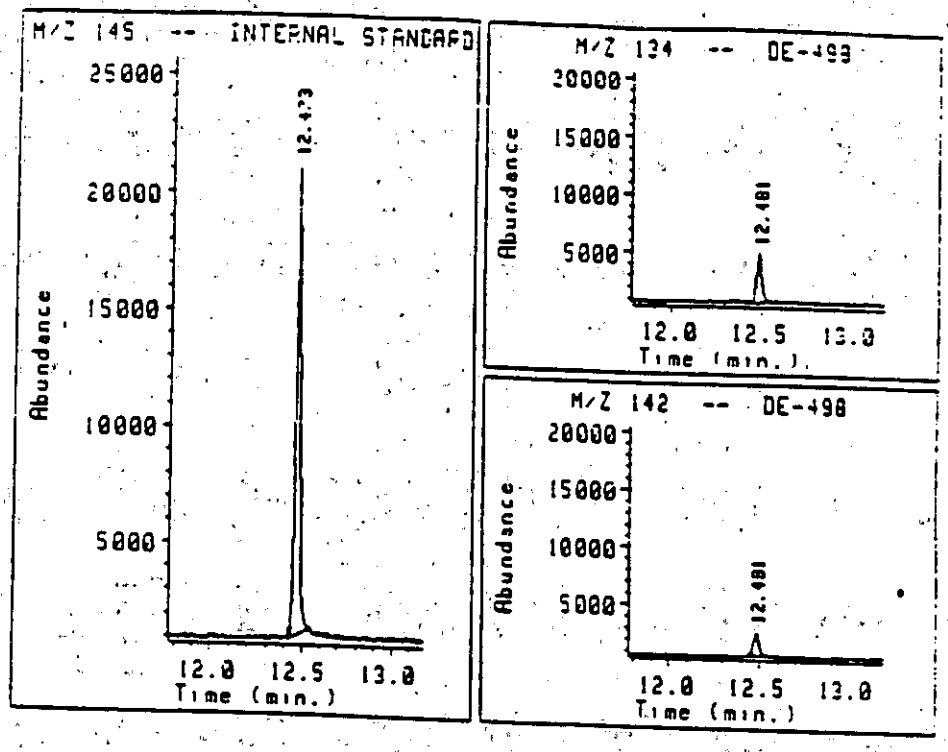


FIGURE 3. TYPICAL CALIBRATION CURVE FOR THE DETERMINATION OF DE-498 IN SOIL.

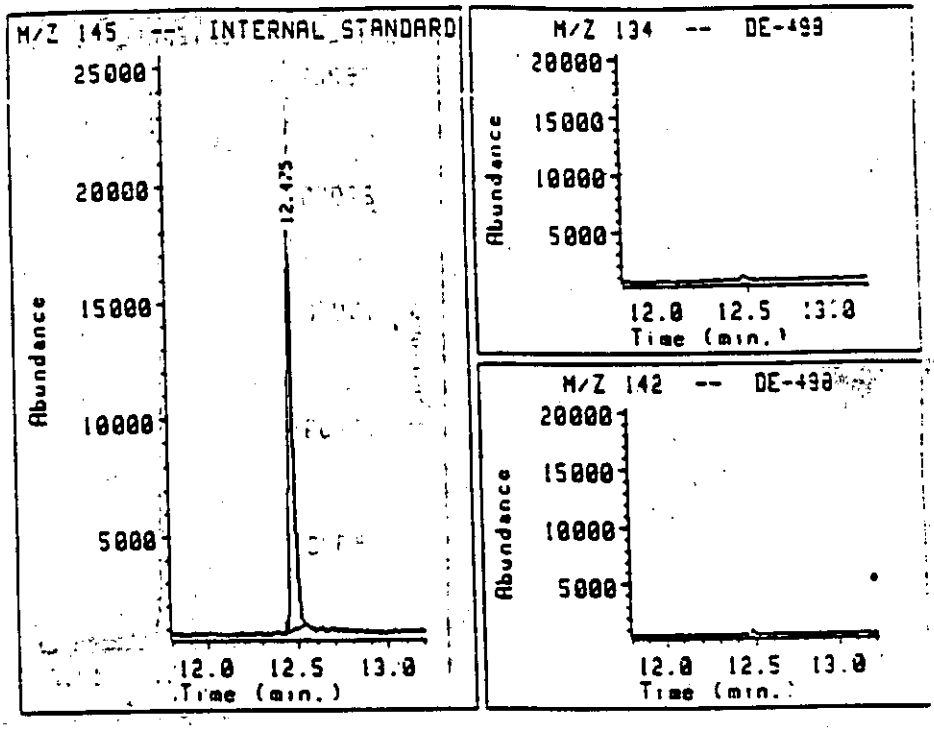


Data File : DATA2:CE17C83A.D
Date : 19 Apr 91 1:23 am
Instrument : MS_5971A - S/N 2749A00149

Sample Name: DE-498 STANDARD - 0.0125 NG/UL - EQUIVALENT TO 2.50 PPB
Sample Info:
Operator : E. L. OLBERDING

INTSTD RETENTION TIME IS :	12.47
PEAK AREA (M/Z 145) :	354123
DE-498 RETENTION TIME IS :	12.48
PEAK AREA (M/Z 134) :	76598
PEAK AREA (M/Z 142) :	42425
DE-498 CONFIRMATION	
RATIO OF M/Z 142/134:	0.5539
DE-498 QUANTITATION	
RATIO OF M/Z 134/145:	0.2163
AVG. RATIO OF M/Z 142/134:	0.5519

FIGURE 4. TYPICAL CHROMATOGRAM OF A 12.5 nG/mL STANDARD EQUIVALENT TO 2.50 ppb IN SOIL.



Data File : DATA2:CE17C86A.D
Date : 19 Apr 91 2:37 am
Instrument : MS_5971A - S/N 2749A00149

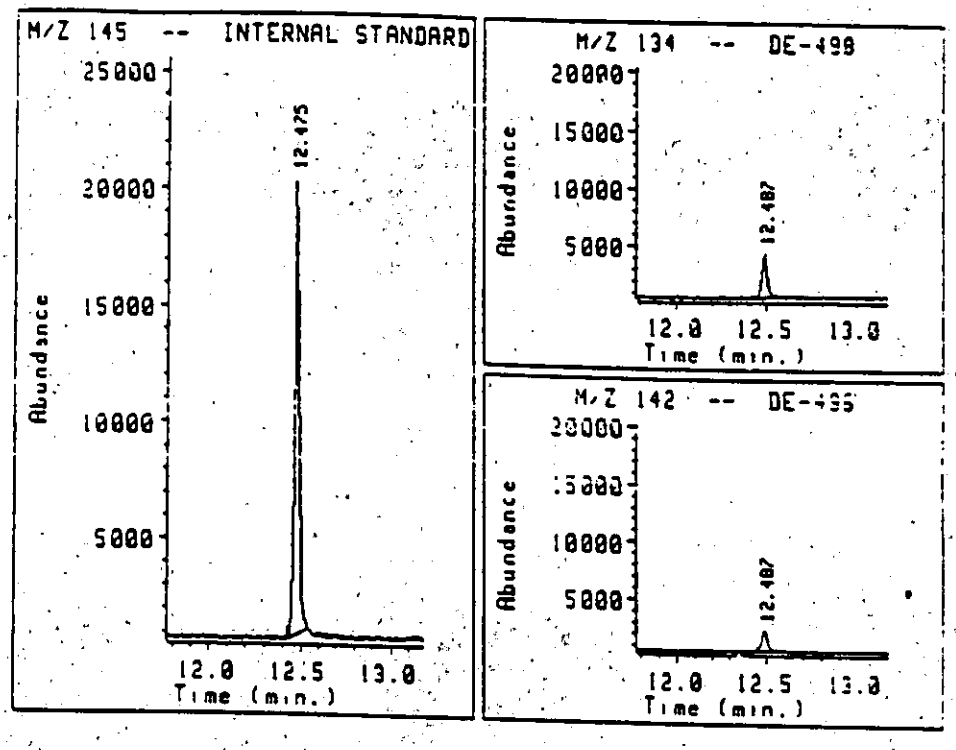
Sample Name: AGR261253 - CONTROL
Sample Info: SOIL FROM BURDETTE, MISSISSIPPI
Operator : E. L. OLBERDING

INTSTD RETENTION TIME IS : 12.47
PEAK AREA (M/Z 145) : 309815

NO DE-498 FOUND

DE-498 CONCENTRATION: 0.00 ppb

FIGURE 5. TYPICAL CHROMATOGRAM OF A CONTROL SAMPLE OF SOIL CONTAINING NO DETECTABLE RESIDUE OF DE-498.



Data File : DATA2:CE17C88A.D
Date : 19 Apr 91 3:25 am
Instrument : MS_5971A - S/N 2749A00149

Sample Name: AGR261253 - SPIKED AT 2.50 PPB - BEFORE PREPARATION
Sample Info: SOIL FROM BURDETTE, MISSISSIPPI
Operator : E. L. OLBERDING

INTSTD RETENTION TIME IS :	12.47
PEAK AREA (M/Z 145) :	343235
DE-498 RETENTION TIME IS :	12.49
PEAK AREA (M/Z 134) :	72322
PEAK AREA (M/Z 142) :	40825
DE-498 CONFIRMATION	
RATIO OF M/Z 142/134:	0.5645
DE-498 QUANTITATION	
RATIO OF M/Z 134/145:	0.2107
AVG. RATIO OF M/Z 142/134:	0.5519

NET DE-498 CONCENTRATION: 2.43 ppb
RECOVERY: 97 %

FIGURE 6. TYPICAL CHROMATOGRAM OF A CONTROL SAMPLE OF SOIL FORTIFIED WITH 2.50 ppb DE-498.

END