# ORGANICS LABORATORY

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| Method No. | Edition | Revision |
|---|---|---|
| CHR-12111671 | 03/28/89 | 1.0 |

Subject:

Determination of Prometryn and GS-11354 in Soils

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References: CIBA-GEIGY Corporation

1.0 SCOPE

This method describes the procedure for extraction, cleanup and quantitation of Prometryn and GS-11354 residues in soil. This method is sensitive to 0.01 ppm.

2.0 PRINCIPLE

Prometryn and GS-11354 are extracted from soil by refluxing with acetonitrile/water (80:20). The extract is concentrated to aqueous and cleaned up by use of an Extrelut column. The analytes are eluted with ethyl acetate/hexane (25:75) and the eluate is dried by rotary evaporation. The analytes are brought up in ethyl acetate/methanol (95:5) and analyzed by capillary gas chromatography with flame thermionic detection.

3.0 CHEMICALS AND SOLUTIONS

Prometryn Standard Reference Material (CIBA-GEIGY Corporation)
GS-11354 Standard Reference Material (CIBA-GEIGY Corporation)
Acetonitrile
Ethyl Acetate

3.0 Chemicals and Solutions (continued)

Glass Wool
Hexane
Methanol
Water, Reverse Osmosis/Deionized
Extrelut

4.0 APPARATUS

4.1 Equipment

Heating Mantle or Hot Plate
Glass Microfibre Filter
Buchner Funnel
Teflon Boiling Chips
Nitrogen Atmosphere Evaporator
Extrelut Reservoir Tube, 75 mL capacity

4.2 Glassware

Graduated Cylinder, 200 mL
Boiling Flask, 500 mL
Liebig Condenser
Erlenmeyer Sidearm Suction Flask, 500 mL
Pipets, Class A Volumetric
Pipettes, Dispo Pastuer
Vial, Solvent Saver (7 mL)
Vial, Auto Sampler (1.5 mL) with screw cap

4.3 Gas Chromatograph

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|---|---|
| Instrument: | Shimadzu GC-9A with Flame Thermionic Detector |
| Autosampler: | Shimadzu AOC-9 |
| Column: | 15 x 0.53 mm ID, 5% Phenyl methyl silicon, 1 um DF |
| Data System: | Maxima Data System, Dynamic Solutions or equivalent |

5.0 ANALYTICAL PROCEDURE

5.1 Extraction

5.1.1 Weigh 50 g (to 0.001g) of soil into a 500 mL boiling flask and record the weight on the Raw Data Summary Sheet.

5.1.2 Add 250 mL of acetonitrile:water (80:20) and assemble the flask, water cooled Liebig condenser, and heating mantle for refluxing.

5.1 Extraction (continued)

5.1.3 Reflux for one hour.

5.1.4 Remove heat from the flask and allow it to cool to approximately room temperature.

5.1.5 Filter sample solution through Buchner Funnel fitter with a microfibre filter. Rinse boiling flask and filtrate with approximately 50 mL of 80/20 acetonitrile/water. Collect all of the filtered extract.

5.1.6 Quantitatively transfer the filtered extract to a 500 mL boiling flask and rotary evaporate until approximately 20 mL aqueous remains.

5.1.7 Add one package of Extrelut to the sample flask and mix well. Allow the sample to adsorb onto the Extrelut for a minimum of 20 minutes.

5.1.8 Place a plug of glass wool in the tip of a polyethylene Extrelut reservoir tube and transfer the adsorbed sample.

5.1.9 Elute the analytes of concern into a 500 mL boiling flask with 225 mL of ethyl acetate:hexane (25:75).

5.1.10 Rotary evaporate the sample to dryness and bring up with approximately 5 mL of ethyl acetate:methanol (95:5).

5.1.11 Transfer the sample to a saver vial and dry on a nitrogen evaporator.

5.1.12 Bring up in 2 mL of ethyl acetate: methanol (95:5 ) and transfer to an autosampler vial for analysis by capillary gas chromatography.

6.0 GAS CHROMATOGRAPHIC ANALYSIS

6.1 Instrument Set Points

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|---|---|---|
| 6.1.1 | Column: | 15 m x 0.53 mm ID, 5% Phenyl methyl silicon capillary column, 1 um DF |
| 6.1.2 | Temperature: | Column oven-160 degrees C Injector/Detector Block-225 degrees C |

6.1 Instrument Set Points (continued)

6.1.3 Times: Run Time-12 minutes
Approximate Elution Times:
GS-11354 - 5.6 minutes
Prometryn - 9.0 minutes

6.1.4 Gases: Carrier - Helium at 0.8kg/cm2
Detector - Air and Hydrogen at appropriate setting to achieve optimum sensitivity

6.1.5 Miscellaneous: Range 2
Injection Volume - 6 uL

6.2 Calibration and Quantitation

6.2.1 Set up instrument and detector as per Shimadzu GC-9A I and II Instruction Manuals. Verify performance and stability as per manuals.

6.2.2 Inject standards and samples in a ratio of at least one standard for every three samples. Be sure standards cover the entire concentration range of the samples. Have at least four different concentrations of standards across the concentration range.

6.2.3 Use standard peak heights and concentrations to calculate the linear regression and interpolate residue concentration in sample extracts.

6.2.4 Calculation:

$$ppm = \frac{\text{ng residue found}}{\text{mg sample extracted}}$$