ANALYTICAL METHOD

SANDOZ CROP PROTECTION CORPORATION

Location: ● 1300 E. TOUHY AVE. DES PLAINES, IL

● DEVELOPMENT ○ QUALITY CONTROL

Method Number AM-0810
Addendum
Supersedes
Approved BAB Date 10-5-87

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DETERMINATION OF DIENOCHLOR (PENTAC®) IN SOIL

1. Summary

1.1 Fifty gram subsamples of soil are treated with aqueous sodium chloride to break down the soil structure.

1.2 The subsamples are extracted with 1:2 isopropanol/ toluene.

1.3 Aliquots of the extracts are partitioned with deionized water to remove the isopropanol.

1.4 Ten milliliter aliquots of the toluene extracts are dried with anhydrous sodium sulfate.

1.5 The toluene extracts are analyzed by gas chromatography using electron capture detection (ECD).

3. Safety

3.1 The oral LD_{50} of dienochlor in rats is greater than 1000 mg/kg.

3.2 Normal laboratory precautions are required for safe handling of dienochlor.

3.3 Hexane, isopropanol, and toluene are flammable and should not be used near heat, sparks, or open flames.

3.4 All solvents should be used only in well ventilated laboratories.

3.5 Protective gloves should be worn during extraction and analysis.

3.6 Disposal of samples and standards must be done in compliance with on-site safety policies and procedures.

4. Apparatus

4.1 Bottles, screw cap with Polyseal® liner, 8-oz, amber.

4.2 Centrifuge, International Equipment Company, Serial No. 71154-M.

4.3 Distillation receiver, 15-mL.

4.4 Pipets, Pasteur, 9", disposable.

4.5 Platform Shaker, Eberbach Corp., Ann Arbor, MI.

5. Reagents

5.1 Hexane - "Distilled in Glass", Burdick and Jackson, Muskegan, MI 49442.

5.2 Isopropyl Alcohol - Baker Resi-Analyzed, J.T. Baker Chemical Co., Phillipsburg, N.J. 08865.

5.3 Sodium chloride - reagent grade.

5.4 Sodium sulfate - anhydrous, granular, reagent grade.

5.5 Toluene - Baker Resi - Analyzed, J.T. Baker Chemical Co., Phillipsburg, N.J. 08865.

6. Standard

6.1 Dienochlor (1,1',2,2',3,3',4,4',5,5'-decachloro bis-2,4-cyclopentadien-1-yl) - Sandoz Crop Protection Analytical Reference Standard.

6.2 Dienochlor is sensitive to light. Standard solutions must be stored in amber or foil wrapped glassware at 0°C.

7. Procedure

7.1 Extraction

7.1.1 Weigh 50 g of soil subsample into a tared 8-oz amber glass bottle.

7.1.2 To fortify for recovery determination,

add appropriate volume of fortifying solution, e.g. 1.0 mL of a 10^{-8} g/uL solution to 50 g sample (0.2 ppm) and allow solvent to evaporate.

7.1.3 Add 30 mL of 5% NaCl solution and shake for 15 minutes on a platform shaker. Longer shaking may be necessary to breakup the larger clays.

7.1.4 Add 50 mL of isopropanol and 100 mL of toluene and shake for 2 hours on the platform shaker.

7.1.5 Centrifuge sample for 5 minutes.

7.1.6 Transfer a 20-mL aliquot of the organic extract to an 8-oz. amber glass bottle containing 100 mL deionized water and shake for 1 minute.

7.1.7 Centrifuge sample for 5 minutes.

7.1.8 Transfer a 10-mL aliquot of the toluene extract to a Kuderna-Danish receiver and add about 0.1 g of anhydrous sodium sulfate. Shake well. The extracts are now ready for GC analysis.

8. Analysis

8.1 Preparation of Standards

8.1.1 Prepare a stock solution containing 100.0 mg dienochlor/100 mL toluene in a 100-mL volumetric flask (10^{-6} g/uL).

8.1.2 Transfer a 1.0-mL aliquot of the stock solution (10^{-6} g/uL) to a 100-mL volumetric flask and bring to the mark with hexane. This standard (10^{-8} g/uL) will be used for fortifying check samples.

8.1.3 Prepare a range of standards for GC/EC quantitation by diluting aliquots of the appropriate standards to 50 or 100 mL with toluene as follows:

Standard	Aliquot	Final Volume	Concentration of Final Solution
10^{-8} g/uL	1 mL	100 mL	10^{-10} g/uL
10^{-10} g/uL	25 mL	50 mL	5×10^{-11} g/uL
10^{-10} g/uL	10 mL	50 mL	2×10^{-11} g/uL
10^{-10} g/uL	5 mL	50 mL	10^{-11} g/uL

8.2 Gas Chromatographic Conditions

The following gas chromatographic conditions were used during method development. Other conditions may be used provided that dienochlor is separated from sample interferences and the response is linear over the range of interest.

8.2.1 Instrument: Hewlett-Packard, Model 5880, equipped with Electron Capture Detector (^{63}Ni) and H-P model 7671 autosampler.

8.2.2 Column: 30 m x 0.53 mm (I.D.) fused silica with methyl silicone (SE-30) bonded

phase - 0.88 um film thickness (HP-1).

8.2.3 Oven Temperature: 170°C isothermal for 5 minutes.

8.2.4 Injector Temperature: 250°C

8.2.5 Detector Temperature: 350°C

8.2.6 Carrier Gas: helium, inlet pressure: 5 psi (4.5 mL/min).

8.2.7 Make-up Gas: 5% argon/methane at 30 mL/min.

8.2.8 Dienochlor Retention Time: 3.4 min.

8.3 Quantitation

8.3.1 Prepare a standard curve by injecting a fixed volume of standard solutions of ranging concentrations (ng/uL) and plotting peak height versus concentration injected on a log-log graph paper. (Inject 2.0-uL aliquots of 10^{-10}, 5×10^{-11}, 2×10^{-11}, and 10^{-11} g/uL standards).

8.3.2 Determine the concentration of dienochlor in an injected aliquot of sample from the peak height and the standard curve.

8.3.3 Calculate the concentration of

dienochlor in the sample using the following expression:

$$ppm = C_s \times \frac{V_s}{W_s}$$

Where:

ppm = Concentration of dienochlor in the sample in parts per million (ng/mg).

C_s = Concentration of dienochlor in the injected aliquot (ng/uL) - from standard curve.

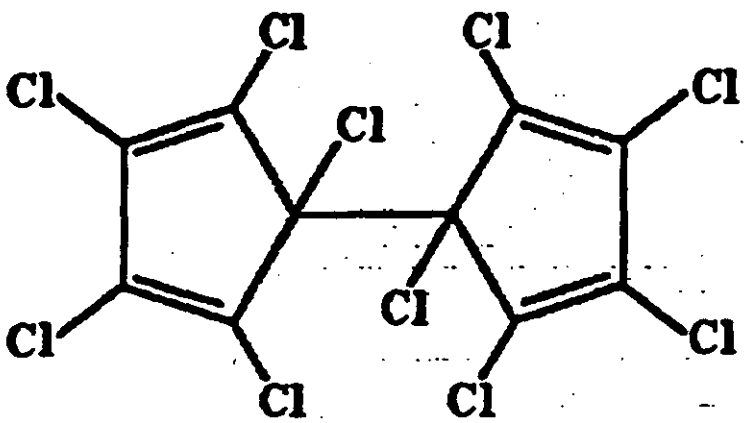
V_s = Volume of final sample extract in milliliters taking into account all dilutions. If not diluted, this volume represents the 10-mL aliquot of the toluene extract from 7.1.8.

W_s = Weight of sample taken for analysis in grams. This weight represents the gram equivalent in the 10-mL aliquot of the toluene extract from 7.1.8.

9. References

9.1 Work was done by L. J. Formanski. This work is recorded in notebook #4931, pp 52-97.

9.2 The structure of dienochlor is presented in Figure 1.



Dienochlor

Figure 1. Molecular structure of Dienochlor.

Appendix VI. Sample Calculations

A. Residue Level Calculation From Gas Chromatographic Results

To determine the concentration of analyte in an injected aliquot of extract, the peak height in the sample chromatogram is compared to the standard curve obtained from a series of standards of known and similar concentration injected during the same GC run. The corresponding concentration of analyte is interpolated from this standard curve.

The concentration of analyte residue in the sample is then determined using this aliquot concentration and the following expression:

$$\text{ppm (ng/mg)} = \frac{C_e\ (\text{ng}/\mu\text{L}) \times V_s\ (\mu\text{L})}{W_s\ (\text{mg})}$$

Where:

ppm = Concentration of analyte in the sample in parts per million (ng of analyte/mg of substrate)

V_s = Volume of final sample extract in microliters taking into account all dilutions and or aliquots used.

W_s = Weight of sample taken for analysis, in milligrams.

C_e = Concentration of residue in extract (ng/μL) determined from the standard curve.

A sample calculation is shown below using a 50 gm sample, 10 ml final volume and a final analyte concentration of 0.50 ng/μl.

V_s = 10 ml, (or 10,000 μl)

W_s = 50 mg, (or 50,000 mg)

C_e = 0.50 ng/μl

$$\text{ppm} = 0.50 \times \frac{10{,}000\ \mu\text{l}}{50{,}000\ \text{mg}}$$

$$\text{ppm} = 0.50 \times 0.20$$

$$\text{ppm} = 0.50$$