

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Dienochlor

MRID #: 413034-08

Matrix: Soil

Analysis: GC/ECD

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ANALYTICAL METHOD

SANDOZ CROP PROTECTION CORPORATION Location: <input checked="" type="radio"/> 1300 E. TOWRY AVE. DES PLAINES, IL	Method Number <u>AM-0810</u>
	Addendum _____
	Supersedes _____
	Approved <u>TSAB</u> Date <u>10-5-87</u>
<input checked="" type="radio"/> DEVELOPMENT <input type="radio"/> QUALITY CONTROL	Reviewed _____ Date _____
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	Reviewed _____ Date _____

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).

2. Accuracy and Precision

- 2.1 Recoveries from fortified check samples of three soil types are listed in Table 1. Average recoveries ranged from 93.0 ± 8.2% to 107.0 ± 5.0%.

3. Safety

- 3.1 The oral LD₅₀ of dieldrin in rats is greater than 1000 mg/kg.
- 3.2 Normal laboratory precautions are required for safe handling of dieldrin.
- 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 Dieldrin (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 Dieldrin 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/mL 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 dieldrin/100 mL toluene in a 100-mL volumetric flask (10^{-6} g/mL).

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 dieldrin 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:

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

Where:

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

C_s = Concentration of dienochlor in the injected aliquot (ng/ μ L) - 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.

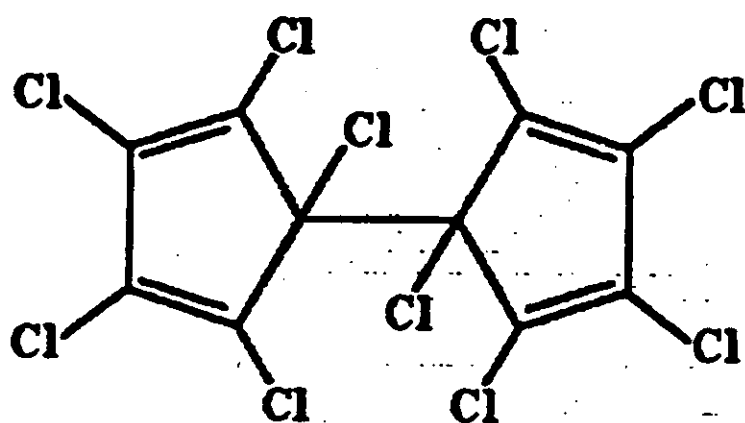
10. Representative Calibration Curve and Chromatograms

Figure 2 Representative Calibration Curve

- 3 Representative Chromatogram of Dieldrin Standard (0.1 ng/uL)**
- 4 Representative Chromatogram of Dieldrin Standard (0.05 ng/uL)**
- 5 Representative Chromatogram of Dieldrin Standard (0.01 ng/uL)**
- 6 Representative Chromatogram of a Silt Clay Soil Check**
- 7 Representative Chromatogram of a Silt Clay Soil Check Fortified at 0.2 ppm Dieldrin**
- 8 Representative Chromatogram of a Sandy Clay Loam Soil Check**
- 9 Representative Chromatogram of a Sandy Clay Loam Soil Check Fortified at 0.2 ppm Dieldrin**
- 10 Representative Chromatogram of a Kenyon Loam Soil Check**
- 11 Representative Chromatogram of a Kenyon Loam Soil Check Fortified at 0.2 ppm Dieldrin**

Table 1. Recoveries of Dieldrin from Soil Fortified at 0.2 ppm.

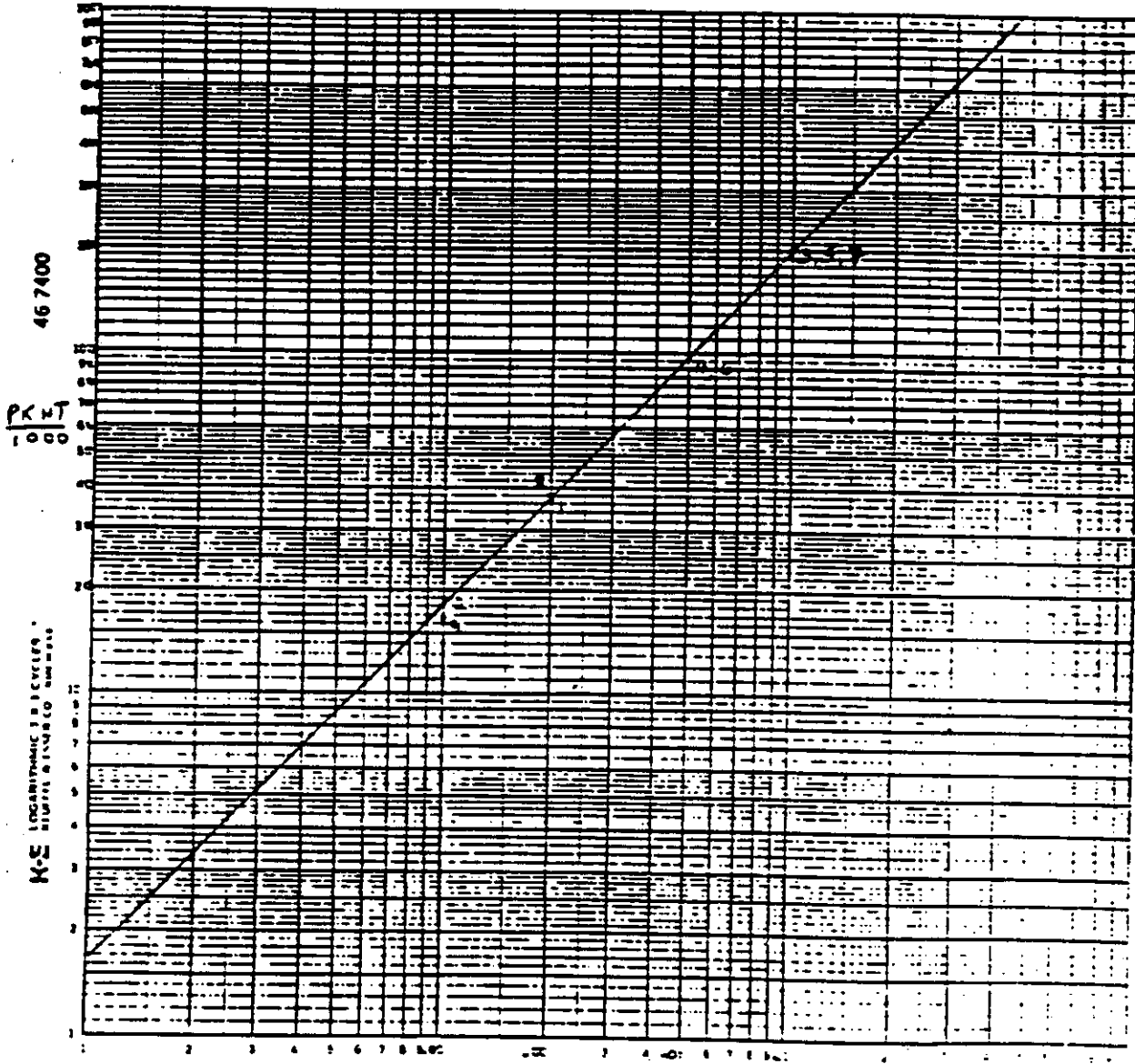
Soil	% Recoveries	Ave. and Std. Dev.
Kenyon Loam	91, 102, 86	93 ± 8.2
Sandy Clay Loam	106, 102, 112	107 ± 5.0
Silt Clay	106, 111, 109, 98, 104, 98	104 ± 5.5



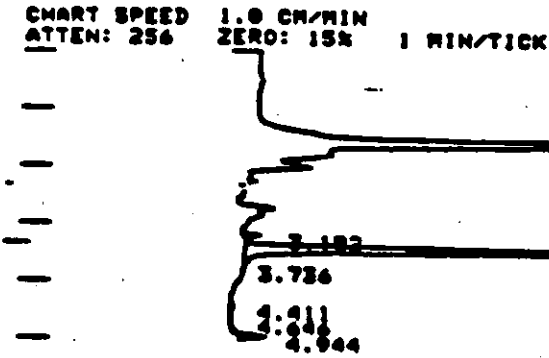
Dienochlor

Figure 1. Molecular structure of Dienochlor.

Figure 2. Representative calibration curve for the determination of dieldrin in soil. The numbers by the data points represent the order of injection of the standards. The standards were interspersed with the study samples.



ng, /ml dieldrin



TITLE: DIENOCHLOR IN SOIL

10:42 12 JUN 87

CHANNEL NO: 4 SAMPLE: E-10

METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.004940	3.392	-0.000	202437	BB	3.95
TOTALS:				-0.000	202437		

UNIDENT AREA: 13119

DETECTED PKS: 6 REJECTED PKS: 0

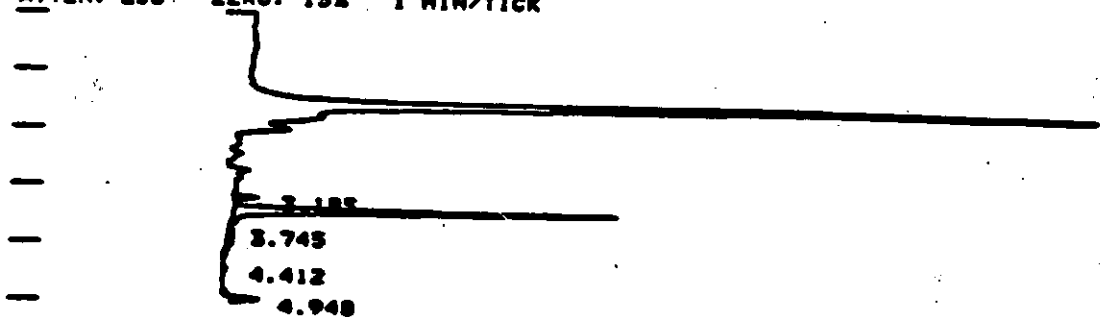
AMT STD: 0.10000

NOISE: 105.6 OFFSET: -020

ERRORS:
FACTOR NOT UPDATED

Figure 3. Representative GC/EC/PSOT chromatogram of a 0.10 ng/ul dienochlor standard solution; 2.0ul injected.

CHART SPEED 1.0 CM/MIN
ATTEN: 256 ZERO: 152 1 MIN/TICK



TITLE: DIENOCHLOR IN SOIL

10:15 12 JUN 87

CHANNEL NO: 4 SAMPLE: SE-11

METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.005007	3.394	-0.006	90207	00	3.85

TOTALS: -0.006 90207

UNIDENT AREA: 15663

DETECTED PKS: 6 REJECTED PKS: 0

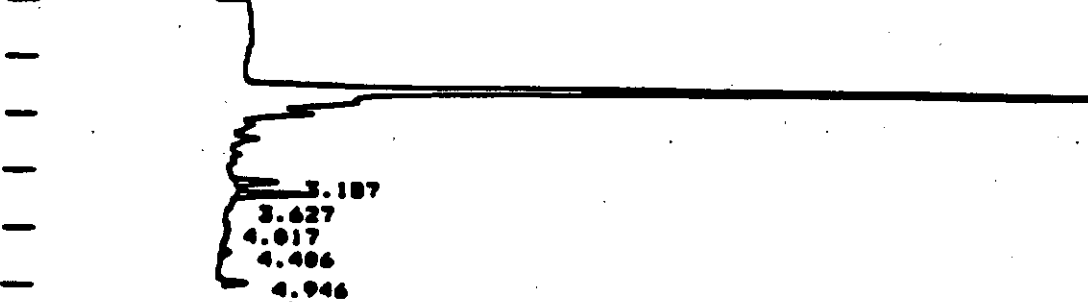
AMT STD: 0.05000

ISE: 105.6 OFFSET: -660

ERRORS:
FACTOR NOT UPDATED

Figure 4. Representative GC/EC/FSOT Chromatogram of a 0.050 ng/uL dienochlor standard solution; 2.0 uL injected.

CHART SPEED 1.0 CM/MIN
ATTEN: 256 ZERO: 15% 1 MIN/TICK



140

TITLE: DIENOCHLOR IN SOIL

17:21 12 JUN 87

CHANNEL NO: 4

SAMPLE: E-11

METHOD: DIENOCHLOR

PK NO	PEAK NAME	RESULT FACTOR	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.005468	3.393	-0.007	10287	BB	4.00

TOTALS: -0.007 10287

UNIDENT AREA: 20682

DETECTED PKS: 0 REJECTED PKS: 1

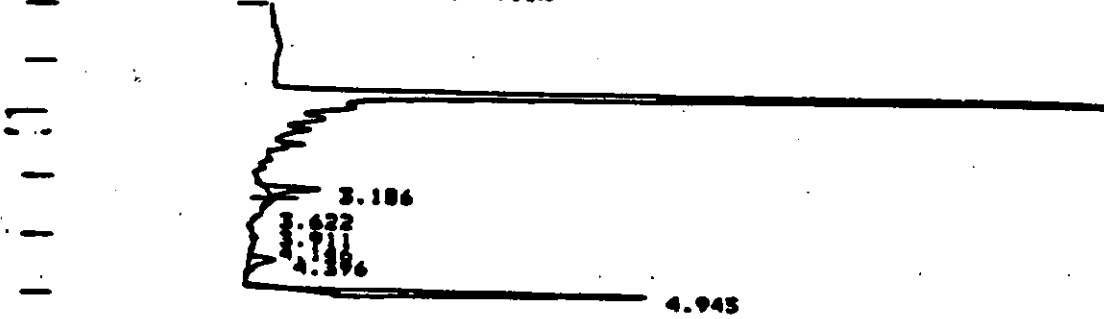
ANT STD: 9.01000

NOISE: 105.6 OFFSET: -473

ERRORS:
FACTOR NOT UPDATED

Figure 5. Representative GC/EC/FSOT chromatogram of a 0.010 ng/ul dienochlor standard solution; 2.0 ul injected.

CHART SPEED 1.0 CM/MIN
ATTEN: 256 ZERO: 15% 1 MIN/TICK



141.

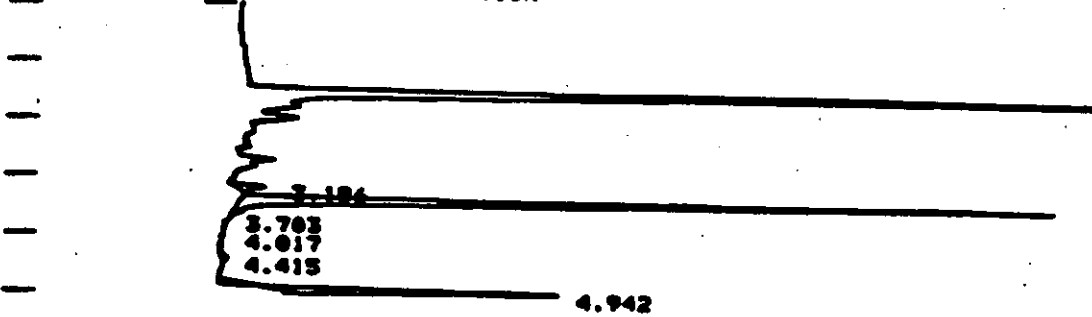
TITLE: DIENOCHLOR IN SOIL 17:07 12 JUN 87
 CHANNEL NO: 4 SAMPLE: SCK 6/12 METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.0019	3.359	-0.041	366	BB	1.10
TOTALS:		0.0019		-0.041	366		

UNIDENT AREA: 104999
 DETECTED PKS: 8 REJECTED PKS: 0
 DIVISOR: 2.00000 MULTIPLIER: 10.0000
 NOISE: 105.6 OFFSET: -277

Figure 6. Representative GC/EC/FSOT chromatogram of a silt clay soil check sample; 0.5 mg equiv./uL injected; <0.005 ng/uL detected; <0.01 ppm dieldrin.

ART SPEED 1.0 CM/MIN
ATTEN: 256 ZERO: 15N 1 MIN/TICK



TITLE: DIENOCHLOR IN SOIL 19:23 12 JUN 87

CHANNEL NO: 4 SAMPLE: CK-11 6/12 METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	1.0951	3.392	-0.008	211002	BB	3.85
TOTALS:		1.0951		-0.008	211002		

UNIDENT AREA: 01389

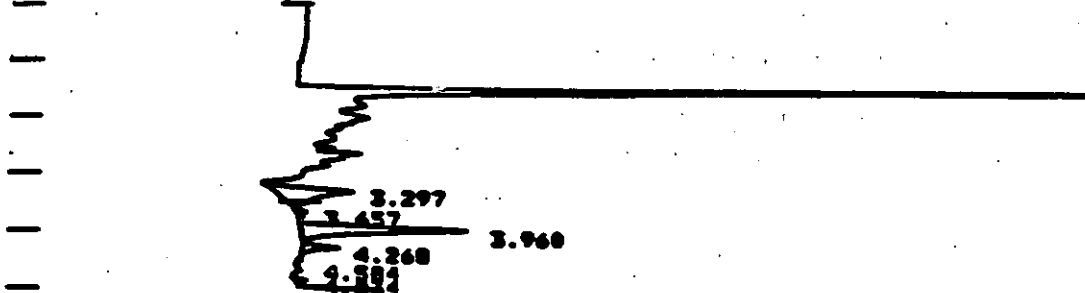
DETECTED PKS: 9 REJECTED PKS: 2

DIVISOR: 2.00000 MULTIPLIER: 10.0000

NOISE: 105.6 OFFSET: -1294

Figure 7. Representative GC/EC/FSOT chromatogram of a silt clay soil check sample fortified at 0.2 ppm dieldrin; 0.5 ng equiv./uL injected; 0.104 ng/uL detected; 0.208 ppm dieldrin (104% recovery).

CHART SPEED 1.0 CM/MIN
ATTEN: 512 ZERO: 15% 1 MIN/TICK



TITLE: DIENOCHLOR IN SOIL

8:51 24 JUN 87

CHANNEL NO: 4

SAMPLE: SCL CK

METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	M1/2 (SEC)
1	DIENOCHLO	0.2109	3.297	-0.133	40627	33	15.75

TOTALS:		0.2109		-0.133	40627		
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UNIDENT AREA: 121971

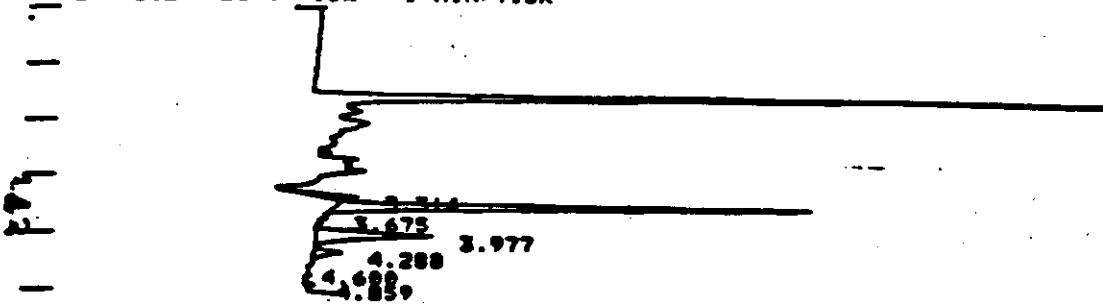
DETECTED PKs: 9 REJECTED PKs: 0

DIVISOR: 2.00000 MULTIPLIER: 10.0000

NOISE: 247.7 OFFSET: 8365

Figure 8. Representative GC/EC/FSOT chromatogram of a sandy clay loam soil check sample; 0.5 mg equiv./uL injected; < 0.005 ng/uL detected; < 0.01 ppm dieldrin.

CHART SPEED 1.0 CM/MIN
ATTEN: 512 ZERO: 15% 1 MIN/TICK



TITLZ: DIENOCHLOR IN SOIL

10:00 24 JUN 87

CHANNEL NO: 4

SAMPLE: SCL CK+111

METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	1.2730	3.464	0.034	245278	BB	4.65
TOTALS:		1.2730		0.034	245278		

UNIDENT AREA: 102573

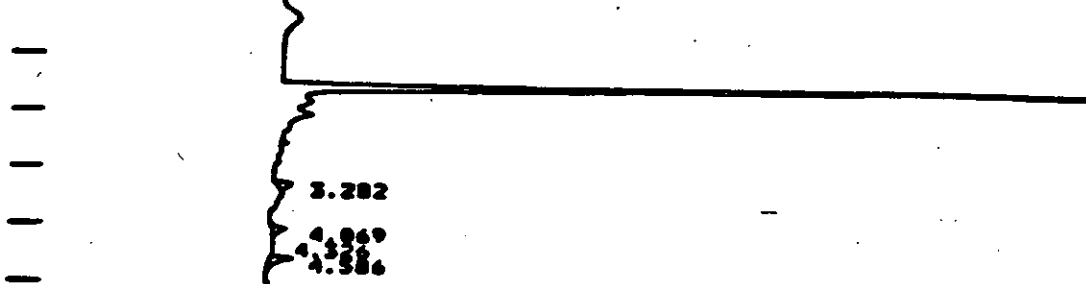
DETECTED PKS: 10 REJECTED PKS: 0

DIVISOR: 2.0000 MULTIPLIER: 10.0000

NOISE: 247.7 OFFSET: 8666

Figure 9. Representative GC/EC/FSOT chromatogram of a sandy clay loam soil check sample fortified at 0.2 ppm dienochlor; 0.5 mg equiv./ul injected; 0.112 ng/uL detected; 0.226 ppm dienochlor (112% recovery).

CHART SPEED 1.0 CM/MIN
ATTEN: 512 ZERO: 15% 1 MIN/TICK



TITLE: DIENOCHLOR IN SOIL

15:31 23 JUN 87

CHANNEL NO: 4 SAMPLE: KLS CK

METHOD: DIENOCHLOR

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.0069	3.467	-0.013	1331	BB	15.65
TOTALS:		0.0069		-0.013	1331		

UNIDENT AREA: 27570

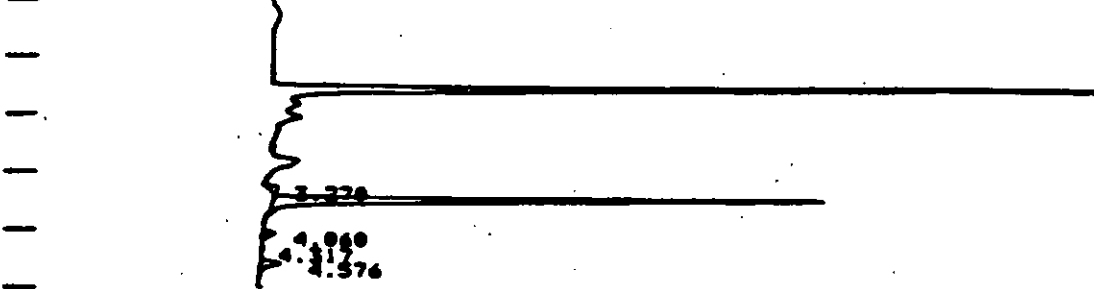
DETECTED PKS: 6 REJECTED PKS: 0

DIVISOR: 2.00000 MULTIPLIER: 10.0000

NOISE: 165.1 OFFSET: 3991

Figure 10. Representative GC/EC/PSOT chromatogram of a kenyon loam soil check sample; 0.5 mg equiv./uL injected; < 0.005 ng/uL detected; < 0.01ppm dieldrin.

CHART SPEED 1.0 CM/MIN
ATTEN: 512 ZERO: 15% 1 MIN/TICK



TITLE: DIENOCHLOR IN SOIL 17:55 23 JUN 87
 CHANNEL NO: 4 SAMPLE: KLS CK+11 METHOD: DIENOCHLOR

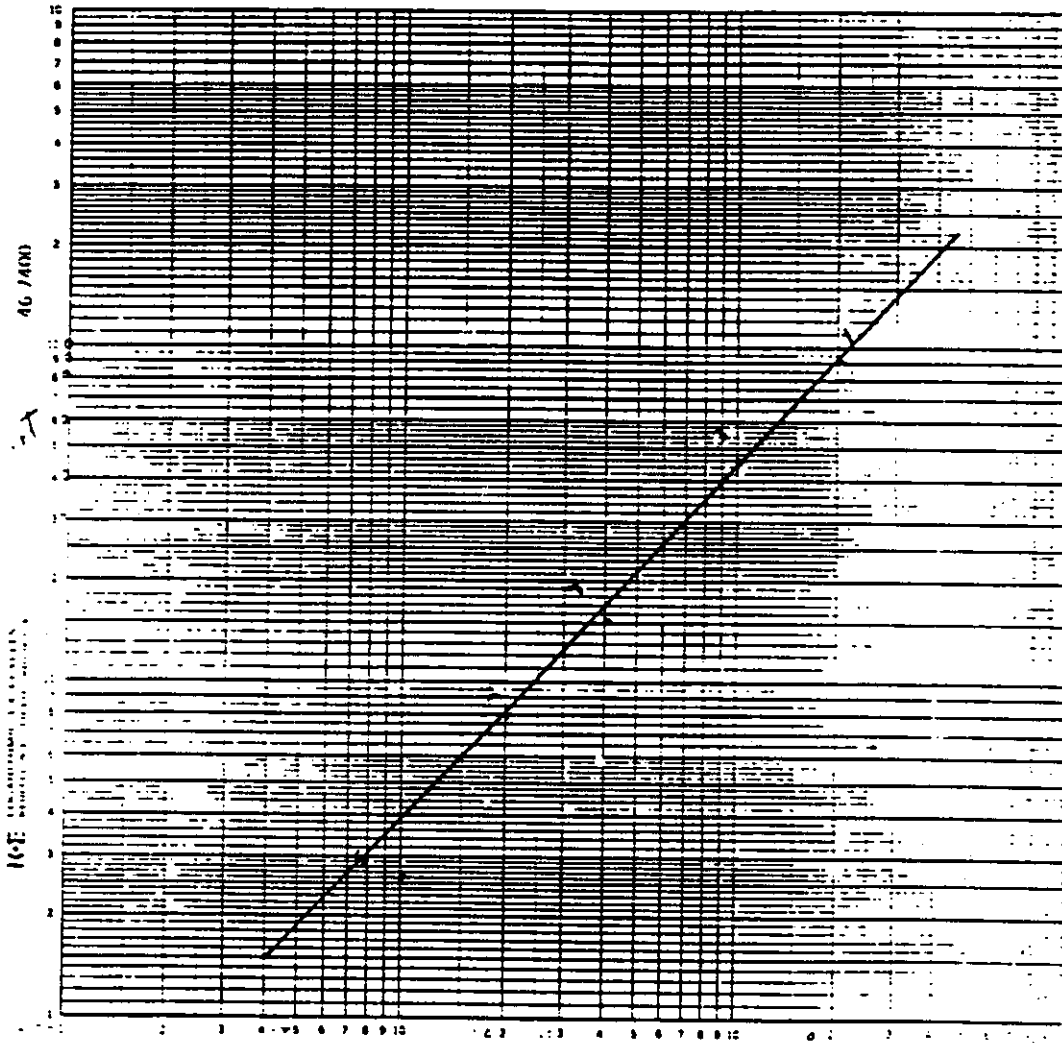
PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	1.4760	3.479	-0.001	284397	BB	3.90
TOTALS:		1.4760		-0.001	284397		

UNIDENT AREA: 21697
 DETECTED PKS: 5 REJECTED PKS: 0
 DIVISOR: 2.00000 MULTIPLIER: 10.0000
 NOISE: 165.1 OFFSET: 2685

Figure 11. Representative GC/EC/FSOT chromatogram of a kenyon loam soil check sample fortified at 0.2 ppm dieldrin; 0.5 ng equiv./ul injected; 0.102 ng/ul detected; 0.204 ppm dieldrin (102% recovery).

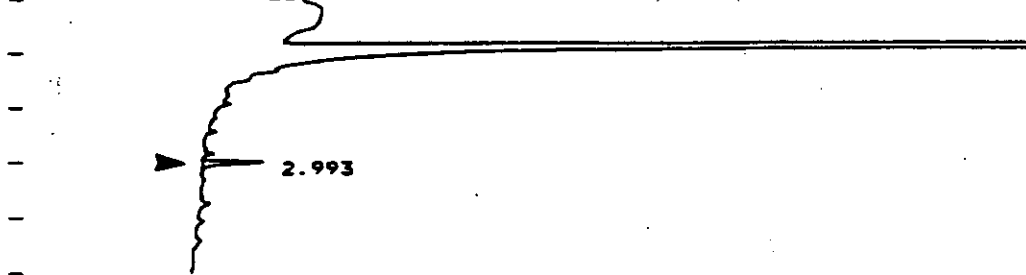
APPENDIX V

**REPRESENTATIVE STANDARD CURVE AND
SAMPLE CHROMATOGRAMS**



Typical standard curve for dieldrin GC analysis.
The numbers by the data points represent the order of injection of the standards. Standards were interspersed with samples during analysis.

HART SPEED 1.0 CM/MIN
TTEN: 120 ZERO: 10% 1 MIN/TICK



TITLE: DIENOCHLOR/SOIL/GEORGIA

13:57 23 JUN 80

CHANNEL NO: 1

SAMPLE: E-11

METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
1	DIENOCHLOR	100.0000	2.993	0.003	7953	BB	3.20
TOTALS:		100.0000		0.003	7953		

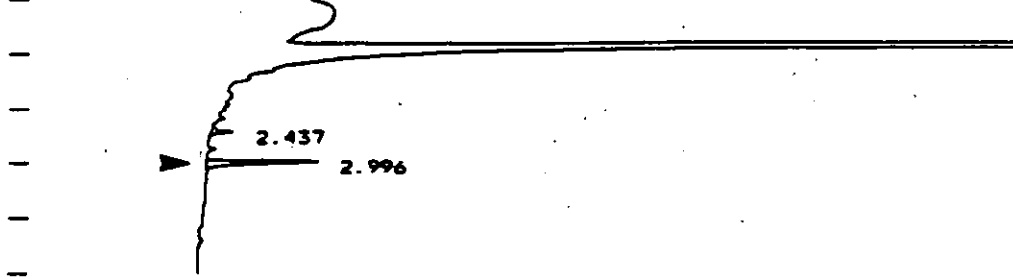
DETECTED PKS: 1 REJECTED PKS: 0

MULTIPLIER: 1.00000

NOISE: 4792.9 OFFSET: 5522

Typical chromatogram of dienochlor standard; 2.0 ul injection of a 0.01 ng/ul standard.

START SPEED 1.0 CM/MIN
ATTEN: 120 ZERO: 10X 1 MIN/TICK



TITLE: DIENOCHLOR/SOIL/GEORGIA

16:37 23 JUN 88

CHANNEL NO: 1 SAMPLE: 2711

METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLOR	84.5937	2.996	0.006	15336	BB	3.20
TOTALS:		84.5937		0.006	15336		

INTEGRATED AREA: 2793

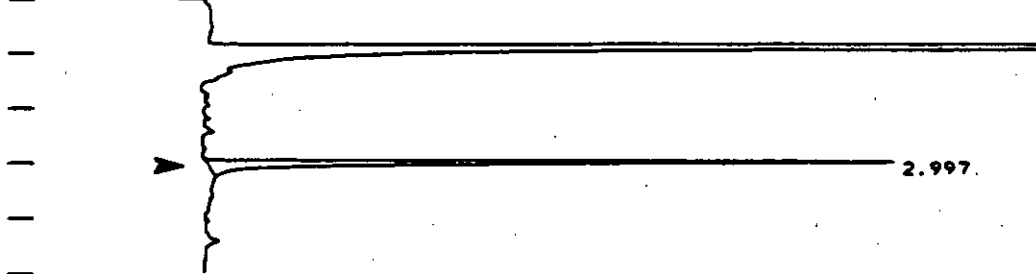
DETECTED PKS: 2 REJECTED PKS: 0

MULTIPLIER: 1.00000

NOISE: 1790.6 OFFSET: 5894

Typical chromatogram of dieldrin standard; 2.0 ul injection of a 0.02 ng/ul standard.

CHART SPEED 1.0 CM/MIN
ATTEN: 128 ZERO: 10% 1 MIN/TICK



TITLE: DIENOCHLOR SOIL GEORGIA

12:57 23 JUN 68

CHANNEL NO: 1 SAMPLE: E-10

METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W:2 (SEC)
1	DIENOCHLOR	100.0000	2.997	0.007	93337	BB	3.35
TOTALS:		100.0000		0.007	93337		

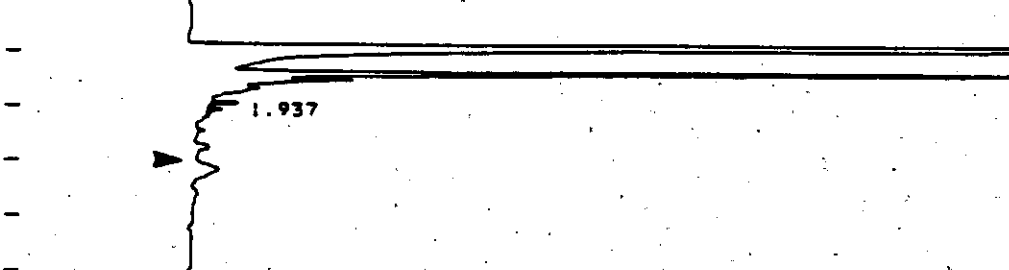
DETECTED PKs: 1 REJECTED PKs: 0

MULTIPLIER: 1.00000

NOISE: 4752.9 OFFSET: 475

Typical chromatogram of dieldrin standard; 2.0 ul injection of a 0.1 ng/ul standard.

HART SPEED 1.0 CM/MIN
TTEN: 100 ZERO: 10% 1 MIN/TICK



TITLE: DIENOCHELOR/SOIL/GEORGIA 13:05 23 JUN 88

CHANNEL NO: 1 SAMPLE: 75-77C(0-10) METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1-W2 (SEC)
1		100.0000	1.937		3690	88	2.33
TOTALS:		100.0000			3690		

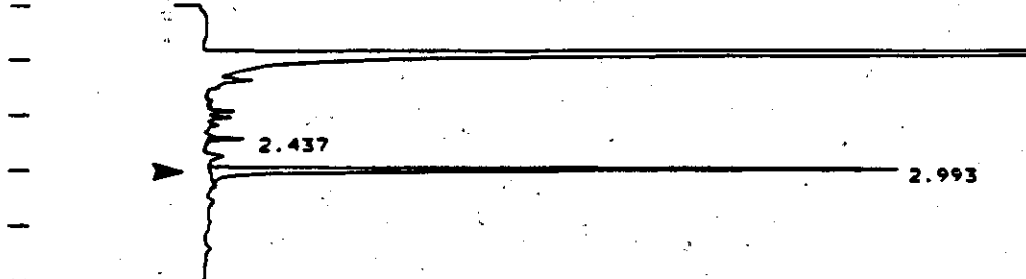
DETECTED PKS: 1 REJECTED PKS: 0

MULTIPLIER: 1.00000

NOISE: 4792.9 OFFSET: 488

Typical chromatogram of check soil (75-77).

HART SPEED 1.0 CM/MIN
ATTEN: 128 ZERO: 10% 1 MIN/TICK



TITLE: DIENOCHLOR/SOIL/GEORGIA

13:14 23 JUN 68

CHANNEL NO: 1 SAMPLE: 75-77CK+

METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1:2 (SEC)
1		4.7446	2.437		4670	BB	2.25
2	DIENOCHLOR	95.2554	2.993	0.003	93757	BB	3.15
TOTALS:		100.0000		0.003	98427		

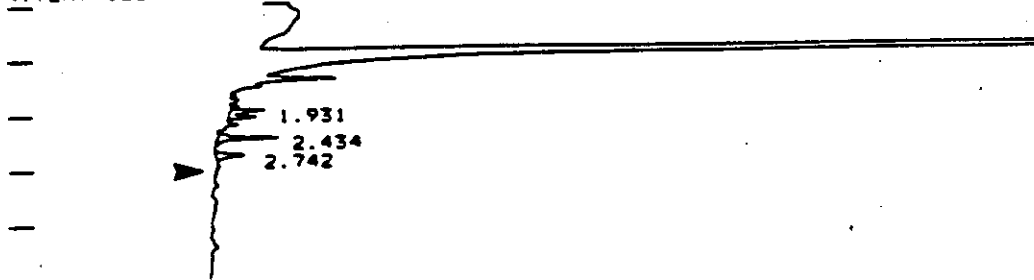
DETECTED PKS: 2 REJECTED PKS: 0

MULTIPLIER: 1.00000

NOISE: 4792.9 OFFSET: 510

Typical chromatogram of ck soil (75-77, 20-30 cm); fortified at 0.20 ppm dienochlor.

CHART SPEED 1.0 CM/MIN
ATTEN: 128 ZERO: 10% 1 MIN/TICK



TITLE: DIENOCHLOR SOIL GEORGIA

17:27 23 JUN 88

CHANNEL NO: 1

SAMPLE: 78-80(20-30)

METHOD: DI

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	WIDE (SEC)
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TOTALS:		0.0000			0		
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UNIDENT AREA: 204.4

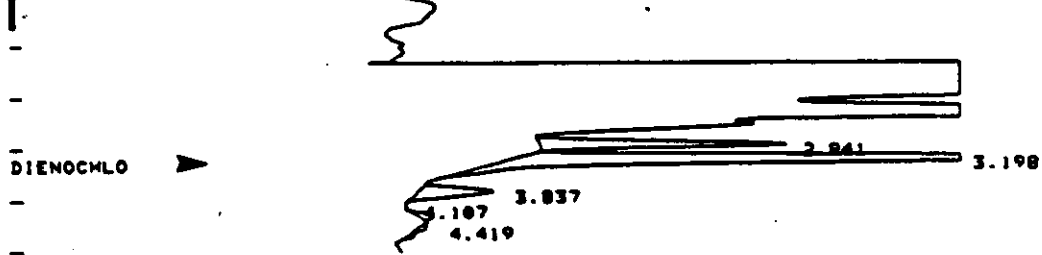
DETECTED PPS: 4 REJECTED PPS: 0

MULTIPLIER: 1.00000

NOISE: 1790.6 OFFSET: 4462

Typical chromatogram of treated soil (78-80, 20-30 cm); 2.0 mg eq./ul injected, no dienochlor was detected.

START SPEED 1.0 CM/MIN
ATTEN: 120 ZERO: 15% 1 MIN/TICK



TITLE: DIENOCHLOR/SOIL(TUFF):GEORGIA
CHANNEL NO: 3 SAMPLE: ~~24-26(0-10)~~ TRT 24-26 (0-10) 15:29 2 MAR 88
METHOD: D

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	TIME OFFSET	HEIGHT COUNTS	SEP CODE	W1/2 (SEC)
2	DIENOCHLO	0.4585	3.198	0.000	113842	BB	8.35
TOTALS:		0.4585		0.000	113842		

IDENT AREA: 49911
DETECTED PKS: 6 REJECTED PKS: 1
DIVISOR: 2.00000 MULTIPLIER: 12.5000
NOISE: 338.7 OFFSET: 9544
NOTES:

Typical chromatogram of treated soil (24-26, 0-10 cm); 0.067 ppm dieno-chlor detected.

APPENDIX VI

SAMPLE CALCULATIONS

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:

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

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/ μ L) 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/ μ l.

$$V_s = 10 \text{ ml, (or } 10,000 \mu\text{l)}$$

$$W_s = 50 \text{ mg, (or } 50,000 \text{ mg)}$$

$$C_e = 0.50 \text{ ng}/\mu\text{l}$$

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

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

$$\text{ppm} = 0.50$$

B. Calculation of Residue Per Soil Core From a PPM Measurement

Residue per soil core was calculated from the ppm values obtained from the 0-10 cm, 10-20 cm, 20-30 cm etc. soil core segments for each sampling interval and the volume and weight of the soil contained within the core segments.

Dimensions of a soil core, (0.9 inch diameter by 10 cm length).

$$\text{Volume of a cylinder (V)} = \text{Pi } r^2 \text{ H}$$

$$\text{Pi} = 3.14$$

r = radius of the cylinder

$$= 0.9 \text{ inch diameter} / 2 \times 2.54 \text{ cm/in}$$

$$= 1.143 \text{ cm}$$

H = height of the cylinder

$$= 10 \text{ cm for each soil core}$$

$$\begin{aligned} V &= 3.14 \times (1.143 \text{ cm})^2 \times 10 \text{ cm} \\ &= 41.04 \text{ cm}^3 \end{aligned}$$

The bulk density of the soil was 1.52 grams/cm³.

Using a ppm value determined for day 3 (0.022 ppm) in the 0-10 cm core segment gives a value of 0.022 µg per gram of soil.

$$\mu\text{g/soil core} = \text{ppm } (\mu\text{g/g}) \times \text{Bulk Density} \times \text{Volume of Core Segment}$$

$$\begin{aligned} &= 0.022 \mu\text{g/g} \times 1.52 \text{ g/cm}^3 \times 41.04 \text{ cm}^3/\text{core} \\ &= 1.37 \mu\text{g}/10 \text{ cm core segment} \end{aligned}$$

This value is the total µg detected at day 3 (see Table III, page 15 this report). No residue was seen in the 10-20 cm or 20-30 cm segments.