Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Dichloropropene

MRID #: 445365-11

Matrix: Soil

Analysis: GC/MS

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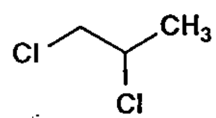
EFFECTIVE: July 26, 1995
SUPERSEDES: New

Determination of Residues of 1,2-Dichloropropane and *cis*- and *trans*-1,3-Dichloropropene in Soil by Purge and Trap Extraction, Capillary Gas Chromatography and Mass Selective Detection

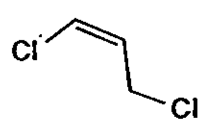
S. C. Dolder, C. E. Kubitschek and H. E. Dixon-White
North American Environmental Chemistry Laboratory
DowElanco
Indianapolis, Indiana 46268 - 1053

A. Scope

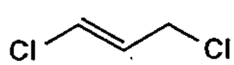
This method is applicable for the quantitative determination of residues of 1,2-dichloropropane (1,2-D) and *cis*- and *trans*-1,3-dichloropropene (1,3-D) in soil over the concentration range of 0.200-160,000 µg/kg with a validated limit of quantitation of 0.200 µg/kg for each compound.



1,2-D
CAS No. 78-87-5



cis-1,3-D
CAS No. 10061-01-5



trans-1,3-D
CAS No. 10061-02-6

B. Principle

This analytical method is based on established EPA purge-and-trap methodology for volatile organic analytes (VOAs), such as Method 8260 (1). Initial extraction of the analytes from soil is by one of two related methods depending on the levels at which the analytes are present in the sample. For analysis in the low-level range (0.20-200 µg/kg), a slurry of soil and water is heated and stirred. The volatile chlorinated hydrocarbons are purged from the sample by sparging with helium, and captured on a sorbent-containing trap. For analysis in the high-level range (200-160,000 µg/kg), the soil sample is first extracted with methanol. An aliquot of the methanol is diluted with water and the resulting water sample is then sparged with helium. The analytes purged from the sample are captured on a sorbent-containing trap. Subsequent steps for both analyses are identical. When purging is complete, the trap is heated and backflushed with helium, and the VOAs are desorbed and transferred to a gas chromatograph (GC). The analytes are then separated on a capillary column and quantitated using mass selective detection (MSD).

C. Safety Precautions

1. Each analyst must 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 obtained from the container label or from the supplier. Disposal of reagents, reactants, and solvents must be in compliance with local, state, and federal laws and regulations.

2. The analytes covered by this method can cause severe and possibly fatal respiratory distress at air concentrations in the ppm range. All operations involving the neat analytes, or concentrated solutions of these compounds, must be carried out in a fume hood. In addition, the effluent from the GC splitter should be routed into an exhaust vent or through a carbon trap to prevent the release of the analytes into the laboratory air.

D. Equipment (Note R.1.)

1. Balance, analytical, Model AE200, Mettler Instrument Corporation, Hightstown, NJ 08520.
2. Balance, pan, Model PM600, Mettler Instrument Corporation.
3. Gas chromatograph, Model 5890 Series II, Hewlett-Packard, Wilmington, DE 19808.
4. Mass selective detector, Model 5971, Hewlett-Packard, Palo Alto, CA 94304.
5. Mass selective detector data system, Model G1034B, Hewlett-Packard.
6. Purge and trap autosampler, Model 2016, Tekmar Company, Cincinnati, OH 45249.
7. Purge and trap autosampler/concentrator, Dynatrap, Dynatech Precision Sampling Corp., Baton Rouge, LA 70895.
8. Purge and trap concentrator, Model 3000, Tekmar Company.
9. Water purification system, Model Milli-Q UV Plus, Millipore Corporation, Milford, MA 01757.

E. Glassware and Materials (Note R.1.)

1. Column, capillary gas chromatography, DB-VRX, 30 m x 0.25 mm i.d., 1.4 μm film thickness, catalog number 122-1534, J&W Scientific, Folsom, CA 95630.
2. Column inlet liner, deactivated, catalog number 5181-8818, Hewlett-Packard, Kennett Square, PA 19348.
3. Filter, charcoal, catalog number 7972, Chrompack, Inc., Raritan, NJ 08869. (Note R.2.)
4. Filter, moisture, catalog number 7971, Chrompack, Inc. (Note R.2.)
5. Filter, oxygen, catalog number 7970, Chrompack, Inc. (Note R.2.)

6. Fritted sparge glassware, 25 mL, catalog number 14-3022000, Tekmar Company.
7. Gas, helium, 99.995% purity, Airco, Murray Hill, NJ 07974.
8. Gas-tight syringes, fixed needle, 10, 100 and 500 µL, catalog numbers 1701, 1710, and 1750, Hamilton Company, Reno, NV 89520.
9. Gas-tight syringe, Leur Lock, 25 mL, catalog number 1025, Hamilton Company.
10. Magnetic stir bars, 3 mm x 13 mm, catalog number 14-511-61, Fisher Scientific, Pittsburgh, PA 15219.
11. Syringe valve, catalog number 2-0940M, Supelco, Inc., Bellefonte, PA 16823.
12. Trap, Tenax, catalog number 2-1075, Supleco, Inc.
13. Trap, Tenax, catalog number 12-0083-003, Tekmar Company.
14. Vials, 11-dram, catalog number 033395D, with polyethylene-lined screw caps, catalog number ALP5026, Fisher Scientific.
15. Vials, 40 mL pre-cleaned amber for volatile analysis, with Teflon-lined septa and screw caps, catalog number 2V42MHSE2BS, Fisher Scientific, Pittsburgh, PA 15219. (Note R.3.)

F. Reagents and Chemicals (Note R.1.)

1. Reagents

a. Internal standard, 2-bromo-1-chloropropane, 95%, compound number 23,127-4, Aldrich Chemical Company, Milwaukee, WI 53233.

b. Methyl alcohol, purge and trap grade, catalog number 41,481-6, Sigma-Aldrich, St. Louis, MO 63178.

c. Standards

(1) 1,2-dichloropropane

The 1,2-D standard used for generating the validation data contained in this method was Lot Number AGR277102, with a purity of 99.2% (2).

(2) *cis*-1,3-dichloropropene

The *cis*-1,3-D standard used for generating the validation data contained in this method was Lot Number AGR164301, with a purity of 97.1% (3).

(3) *trans*-1,3-dichloropropene

The *trans*-1,3-D standard used for generating the validation data contained in this method was Lot Number TSN100232, with a purity of 97.2% (4).

Obtain standards from Test Substance Coordinator, DowElanco, Indianapolis, IN 46268.

d. Water, distilled/deionized, purified using a Milli-Q UV Plus purification system (Section D.9.).

G. Preparation of Standards

1. Preparation of VOA Standard Solutions

NOTE: CARRY THIS PROCEDURE OUT IN A FUME HOOD. When mixing standard solutions do not shake them excessively, as loss of analytes may occur. Mix by capping and gently inverting the solutions approximately five times. Store all standard solutions under frozen conditions in vials with Teflon lined screw cap lids. Allow standard solutions to warm to room temperature prior to use.

a. Tare a 100-mL volumetric flask containing approximately 80 mL of methanol. Add 0.1100-0.1300 g of 1,2-D dropwise via Pasteur pipette (approximately eight to ten drops). Make sure that the liquid falls directly into the methanol and does not run down the inside walls of the volumetric flask. Stopper the flask. Reweigh the flask and calculate the exact weight of the analyte added (Note R.4.). Bring the solution to volume with methanol to yield a stock solution of approximately 1.2 g/L 1,2-D. Calculate the exact concentration of the stock solution; if the standard is less than 97% pure, make the correction for percent purity as follows (Assume a purity of 96% for the 1,2-D standard for the purpose of this example.):

   0.1100 g 1,2-D/0.1 L x 96/100 = 1.056 g/L

b. Repeat step G.1.a. for *cis*-1,3-D and *trans*-1,3-D, preparing a stock solution of approximately 1.2 g/L for each analyte.

c. Using gas-tight syringes, transfer 1/C mL of each stock solution (where C = the concentration of the stock solution in g/L) into a single 10 mL volumetric flask containing approximately 8 mL methanol. Dilute to volume with methanol to obtain a standard solution containing all three analytes, each at a concentration of 100.0 mg/L.

d. Using gas-tight syringes, transfer 1/C mL of each stock solution (where C = the concentration of the stock solution in g/L) into a single 100 mL volumetric flask containing approximately 80 mL methanol. Dilute to volume with methanol to obtain a standard solution containing all three analytes each at a concentration of 10.0 mg/L. For example, a stock solution of 1.056 g/L 1,2-D would be diluted as follows:

$$1/1.056 = 0.947 \text{ mL}$$
$$(947\ \mu L)(1\times10^{-6} L/\mu L)(1.056\ g/L)(1/0.100\ L) = 0.0100\ g/L = 10.0\ mg/L$$

e. Transfer 10.0 mL of the 10.0 mg/L standard solution into a 100 mL volumetric flask. Dilute to volume with methanol to obtain a standard solution containing all three analytes each at a concentration of 1.00 mg/L.

f. Transfer 10.0 mL of the 1.00 mg/L standard solution into a 100 mL volumetric flask. Dilute to volume with methanol to obtain a standard solution containing all three analytes each at a concentration of 0.100 mg/L.

g. Tare a 100-mL volumetric flask containing approximately 80 mL of methanol. Add 11.0-13.0 g of 1,2-D dropwise via Pasteur pipette (approximately eighty drops). Make sure that the liquid falls directly into the methanol and does not run down the inside walls of the volumetric flask. Stopper the flask. Reweigh the flask and calculate the exact weight of the analyte added (Note R.4.). Bring the solution to volume with methanol to yield a stock solution of approximately 120 g/L 1,2-D. Calculate the exact concentration of the stock solution; if the standard is less than 97% pure, make the correction for % purity as follows (Assume a purity of 96% for the 1,2-D standard for the purpose of this example.):

   11.0 g 1,2-D/0.100 L x 96/100 = 105.6 g/L

h. Repeat step G.1.g. for *cis*-1,3-D and *trans*-1,3-D, preparing a stock solution of approximately 120 g/L for each analyte.

i. Using gas-tight syringes, transfer 1/(C/100) mL of each stock solution from Section G.1.g. and G.1.h. (where C = the concentration of the stock solution in g/L) into a single 10 mL volumetric flask containing approximately 6 mL methanol. Dilute to volume with methanol to obtain a standard solution containing all three analytes each at a concentration of 10,000 mg/L. For example, a stock solution of 105.6 g/L 1,2-D would be diluted as follows:

$$1/(105.6/100) = 0.947 \text{ mL}$$
$$(947\ \mu L)(1 \times 10^{-6} L/\mu L)(105.6\ g/L)(1/0.010\ L) = 10.0\ g/L = 10000\ mg/L$$

j. Using gas-tight syringes, transfer 1/(C/100) mL of each stock solution G.1.g. and G.1.h. (where C = the concentration of the stock solution in g/L) into a single 100 mL volumetric flask containing approximately 80 mL methanol. Dilute to volume with methanol to obtain a standard solution containing all three analytes each at a concentration of 1000 mg/L.

2. Preparation of the Internal Standard

Following the procedure outlined above in Sections G.1.a. and d., prepare a 10 mg/L solution of the internal standard (IS), 2-bromo-1-chloropropane. (It is not necessary to correct for percent purity of the IS.)

3. Preparation of the Calibration Standard Solutions for the Low-level Dynatrap Analysis

Prepare aqueous calibration standards of the VOAs by transferring aliquots of the standard solutions as outlined below into empty 40-mL volatile analysis vials (Section E.15.). Use an appropriately-sized gas-tight syringe to transfer the standard solution into the vial, add a magnetic stir bar (Section E.10.) to each vial, and cap immediately. The Dynatrap purge and trap will add 10 mL of distilled/deionized water to each standard prior to analysis. **Calibration standards should be prepared just prior to loading onto the Dynatrap purge and trap for analysis.**

a. Low range calibration standards (Section K.1.):

Initial Solution Concentration mg/L[a]	Aliquot of Initial Solution µL	Calibration Std. Final Conc. µg/L	Equivalent Soil Conc. µg/kg[b]
0.100	5.0	0.050	0.10
0.100	10	0.10	0.20
0.100	25	0.25	0.50
0.100	50	0.50	1.0
1.00	10	1.0	2.0
1.00	25	2.5	5.0

[a] Section G.1.e.-f.
[b] Equivalent soil concentration based upon a 5.0 g sample size.

A 10.0 µL aliquot of the 10.0 mg/L IS solution (Section G.2.) must be added to each calibration standard manually using a gas-tight syringe and transferring the IS to the vial containing the standard just prior to sealing, to obtain an IS concentration of 10.0 µg/L. IS addition can be done automatically using the Dynatrap IS addition feature, in which case a 1.0 µL aliquot of a 100 mg/L IS solution (prepared as described in G.1.c.) must be added to the sample.

b. High range calibration standards (Section K.1.):

Initial Solution Concentration	Aliquot of Initial Solution	Calibration Std. Final Conc.	Equivalent Soil Conc.
mg/L[a]	µL	µg/L	µg/kg[b]
1.00	25	2.5	5.0
1.00	50	5.0	10
1.00	100	10	20
10.0	20	20	40
10.0	40	40	80
10.0	100	100	200

[a] Section G.1.d.-e.
[b] Equivalent soil concentration based upon a 5.0 g sample size.

IS must be added to each calibration standard as described in G.3.a.

4. Preparation of the Calibration Standard Solutions for the High-level Tekmar Analysis

Prepare aqueous calibration standards of the VOAs by transferring aliquots of the standard solutions into 100-mL volumetric flasks containing distilled/deionized water, as outlined below. Use an appropriately-sized gas-tight syringe to transfer the standard solution and inject it into the water below the narrow neck of the flask. **Because aqueous solutions of VOAs are not stable in any container with headspace, each calibration standard should be prepared just prior to purge and trap analysis.**

Calibration standards (Section L.1.):

Initial Solution Concentration	Aliquot of Initial Solution	Calibration Std. Final Conc.	Equivalent Soil Conc.
mg/L[a]	µL	µg/L	µg/kg[b]
1.00	5	0.05	100
1.00	10	0.10	200
1.00	20	0.20	400
1.00	40	0.40	800
1.00	80	0.80	1600
10.0	10	1.0	2000
10.0	50	5.0	10000
100	10	10	20000
100	20	20	40000
100	40	40	80000
100	80	80	160000
100	100	100	200000

[a] Section G.1.c.-e.
[b] Equivalent soil concentration based upon a 5.0 g sample size.

Transfer a 10.0 µL aliquot of the 10.0 mg/L IS solution (Section G.2.) to each calibration standard, to obtain an IS concentration of 1.0 µg/L.

H. Low-level Dynatrap Purge and Trap

1. Purge and Trap Concentrator

Install the Tenax trap (Section E.12.) on the Dynatrap purge and trap concentrator (Section D.7.) following the manufacturer's recommended procedure.

2. Typical Operating Conditions

Instrumentation:	Dynatrap Autosampler/Concentrator
Purge/Carrier Gas:	helium
Purge Flow	40 mL/min
Desorb Flow	20 mL/min
Dynatrap Controller Menu Settings:	
GC cycle time:	23 min
Trap purge ready temperature:	42 °C
Prepurge	0 min
Soil Preheat	70 °C
Preheat stir	Enabled
Preheat time	2 min
Purge time	11 min
Dry purge time	2 min
Desorb preheat	160 °C
Desorb temperature	180 °C
Desorb time	2.0 min
Bake temperature	220 °C
Bake time	6 min
Transfer line temperature	130 °C
Water trap	Enabled
WTKO Cool	45 °C
WTKO Bake	150 °C
Valve oven	110 °C
GC Start	Desorb start
Data start	Desorb start
GC ready	Closed

I. High-level Tekmar Purge and Trap

1. Purge and Trap Concentrator

Install the Tenax trap (Section E.13.) and sparge glassware (Section E.6.) on the Tekmar purge and trap concentrator (Section D.8.) and autosampler (Section D.6.) following the manufacturer's recommended procedure.

2. Typical Operating Conditions

Instrumentation:	Tekmar 3000 Concentrator Tekmar 2016 Autosampler
Purge/Carrier Gas:	helium
Purge Flow	40 mL/min
Desorb Flow	20 mL/min
Trap Pressure	5 psi
Tekmar Controller Menu Settings:	
3000 Transfer Line	140 °C
3000 Valve	140 °C
2016 Transfer Line	130 °C
2016 Valve	130 °C
Moisture control system temp.	130 °C
Trap purge ready temp.	30 °C
Trap purge temp. setting	20 °C (i.e. not heated above ambient temp. during purge)
Sample heater	Off
Prepurge	0.00 min
Preheat time	0.00 min
Purge time	11.00 min
Dry purge time	2.00 min
Moisture control system desorb temp.	45 °C
GC Start	Desorb start
Cryo Focuser	Not applicable
GC cycle time	23 min
Desorb preheat	170 °C
Desorb time	2.00 min
Desorb temp.	180 °C
Sample drain	off
Bake time	10 min
Bake temp.	185 °C
Bake gas bypass	On
Bake gas bypass delay	2.00 min
Moisture control system bake	180 °C

J. Gas Chromatography/Mass Spectrometry (High and Low-level)

1. Column (Note R.5.)

Install the column inlet liner (Section E.2.) and the capillary column (Section E.1.) in the split/splitless injection port of the gas chromatography/mass spectrometer (GC/MSD) following the manufacturer's recommended procedure.

2. Typical Operating Conditions

Instrumentation:	Hewlett-Packard Model 5890 Series II GC Hewlett-Packard Model 5971 Mass Selective Detector Hewlett-Packard Model G1034B Data System Software
Column:	J&W Scientific fused silica capillary DB-VRX liquid phase 30 m x 0.25 mm i.d. 1.4 µm film thickness
Temperatures:	
Column	35 °C for 1.0 min 35 °C to 140 °C at 9 °C/min 140 °C for 0.10 min 140 °C to 210 °C at 20 °C/min 210 °C for 2.0 min
Injector	200 °C
Interface	230 °C
Carrier Gas:	helium
Head Pressure	6 psi
Linear Velocity	approximately 30 cm/sec at 35 °C
Injection Mode:	Split (Note R.5.)
Splitter Flow	20 mL/min
Septum Purge	Off (capped)
Detector:	electron impact selected ion monitoring
Calibration Program	midmass autotune (Note R.6.)
Electron Multiplier	1600 volts

Ions Monitored:

Compound	*m/z*, Quantitation	*m/z*, Confirmation
1,2-D	63	76
cis-1,3-D	75	112
trans-1,3-D	75	112
2-bromo-1-chloropropane	77	--

Dwell Time 75 msec

Full scan mass spectra of the above analytes and IS are shown in Figures 1-4.

3. Typical Chromatograms

Typical chromatograms of a standard, control sample, and a 0.2 µg/kg recovery sample for soil are illustrated in Figures 5-13, respectively.

K. Calibration - Low-level Dynatrap

1. General Approach

The Dynatrap purge and trap which has the capacity to analyze soil samples directly, eliminating the need for an initial solvent extraction, is used for the low-level analysis. Because of the wide range of calibration standard concentrations (0.05-100 µg/L) used in this analysis, it is unlikely that any single calibration curve will provide accurate quantitation over the entire range. In general, deviations from the calculated curve will be most severe at the low end of the curve, which affects measurements near the limit of quantitation. To improve quantitation, it is typically useful to divide the calibration range into two subranges and to produce a separate calibration curve for each. Thus, the low range will encompass VOA standard concentrations of 0.05-2.50 µg/L (equivalent to 0.10-5.0 µg/kg soil sample concentrations), and the high range 2.5-100 µg/L (equivalent to 5.0-200 µg/kg soil sample concentrations).

A calibration check (Section K.4.) must be carried out at the beginning of each 12-hr period during which samples are analyzed in order to confirm calibration of the instrumentation.

2. Initial Calibration

a. Prior to analyzing any standards, bake out the trap at approximately 185 °C and set the GC oven temperature to 220 °C for 10 minutes.

b. Select either the low or high standard range for calibration based upon anticipated levels of the VOAs in the samples being analyzed. After the system has been returned to its starting conditions, load the vials containing the calibration standards for the selected range (Section G.3.) into the Dynatrap autosampler tray. Set the autosampler to add 1.0 µL of a 100 mg/L IS solution (prepared as described in G.1.c.) to each vial (if not added manually during standard preparation) and 10.0 mL of distilled/deionized water. Analyze the calibration standards by purge and trap using GC/MSD as described in Sections H. and J.

3. Calibration Curve

a. Following analysis of the range of calibration standards described in G.3.a. (low range) or G.3.b. (high range), determine the peak areas for 1,2-D (*m/z* 63), *cis*-1,3-D (*m/z* 75), *trans*-1,3-D (*m/z* 75), and 2-bromo-1-chloropropane (IS) (*m/z* 77).

b. For each analyte, prepare a standard curve by plotting the standard concentration (µg/L) on the abscissa (x-axis) and the standard/IS peak area ratio (Quantitation Ratio) on the ordinate (y-axis) as shown in Figures 14-16. Using regression analysis, determine the equation for the curve with respect to the abscissa.

For example, using power regression (5) with the 1,2-D data from Figure 14:

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

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

where: Y = Quant. Ratio; X = Conc. (µg/L)

$$\frac{\text{1,2-D Conc.}}{(\mu g/L)} = \left(\frac{\text{Quant. Ratio}}{\text{constant}}\right)^{(1/\text{exponent})}$$

$$\frac{\text{1,2-D Conc.}}{(\mu g/L)} = \left(\frac{\text{Quant. Ratio}}{0.06498}\right)^{(1/1.02715)}$$

c. Typical calibration curves for the determination of each of the analytes in soil for the low-level, low range analysis are shown in Figures 14-16.

4. <u>Calibration Check</u>

a. For the low calibration range, prepare a 0.25 µg/L calibration standard (Section G.3.a.); for the high range, prepare a 10.0 µg/L calibration standard (Section G.3.b.).

b. Analyze the calibration check standard and calculate the amount of each analyte present using the equation for the current calibration curve for each analyte. (Section K.3.)

c. Calculate the absolute value of the percent difference in the calculated and the theoretical standard concentration as follows:

|(1 - (Calculated/Theoretical))| x 100

If the difference in the calculated and theoretical value for each analyte in the calibration check standard is less than 10%, the existing curves are considered to be valid. Sample analysis may proceed.

d. If the difference in the calculated and theoretical value for any analyte exceeds 10%, the original calibration is no longer considered valid, and a new initial calibration (Section K.2.) must be carried out prior to continuing with sample analysis. A new initial calibration must always be carried out prior to sample analysis whenever major maintenance (column change, source cleaning, filament or multiplier replacement, trap replacement, etc.) is performed on the purge and trap GC/MSD system. Minor maintenance (replacing GC septum or inlet liner, removing a portion of the upper end of the column, etc.) does not automatically necessitate recalibration; however, the system should be evaluated on a case by case basis.

L. <u>Calibration - High-level Tekmar</u>

1. <u>General Approach</u>

The Tekmar autosampler/concentrator (Sections D.6. and 8.), fitted with fritted U-shaped sparge tubes (Section E.6.), is used for the high-level analysis. Soil samples are subjected to an initial solvent extraction, and the resulting extract is diluted with water and analyzed on the Tekmar instrument. A single calibration curve is used in the high-level analysis, covering the range of standard concentrations from 0.05-160 µg/mL (equivalent to 100-320000 µg/kg sample concentration). If problems are encountered in obtaining good correlation of the calibration curve over this range, splitting the curve into two ranges, as done for the low-level method, (Section K.1.) should improve the fit.

A calibration check must be carried out at the beginning of each twelve-hour period during which, samples are analyzed in order to confirm calibration of the instrumentation.

2. Initial Calibration

   a. Prior to analyzing any standards, bake out the trap at approximately 185 °C and set the GC oven temperature to 220 °C for 10 minutes.

   b. After the system has been returned to its starting conditions, load the first aqueous calibration standard (Section G.4.) by removing the plunger from the 25-mL gas-tight Luer Lock syringe (Section E.9.), attaching the closed syringe valve (Section E.11.) to the Luer lock, and pouring the standard into the open end of the syringe until it is nearly full. (Do not draw the standard up into the syringe.) Replace the syringe plunger in the barrel, open the valve, expel any air from the syringe, and adjust the volume to exactly 25.0 mL. Open the loading valve on the Tekmar purge and trap device and load the standard into the sparge tube. Analyze the standard by purge and trap using gas chromatography/mass spectrometry, as described in Sections I. and J.

   c. Repeat L.2.b. for each standard in the calibration range. After each standard has been analyzed, rinse the sparging tube with two approximatley 25-mL aliquots of distilled/deionized water before loading the next standard. Standards should always be analyzed from lowest to highest concentration to minimize carryover. (Note R.7.)

3. Calibration Curve

   a. Following analysis of the range of calibration standards described in G.4.a., determine the peak areas for 1,2-D (*m/z* 63), *cis*-1,3-D (*m/z* 75), *trans*-1,3-D (*m/z* 75), and 2-bromo-1-chloropropane (IS) (*m/z* 77).

   b. For each analyte, prepare a standard curve by plotting the concentration (µg/L) on the abscissa (x-axis) and the standard/IS peak area Quantitation Ratio on the ordinate (y-axis) as shown in Figures 17-19. Using regression analysis, determine the equation for the curve with respect to the abscissa. (See Section K.3.b. for an example calculation.)

   c. Typical calibration curves for the determination of each of the analytes in soil for the high-level analysis are shown in Figures 17-19.

4. Calibration Check

   a. Prepare 0.20 and 20 µg/L calibration check standards (Section G.4.).

   b. Analyze the calibration check standards (Section L.2.b.) and calculate the amount of each analyte present, using the equation for the current calibration curve for each analyte (Section L.3.).

   c. If the difference in the calculated and theoretical value for each analyte in the calibration check standards is less than 10%, the existing curves are considered to be valid. Sample analysis may proceed. (See Section K.4.c. for an example calculation.)

   d. If the difference in the calculated and theoretical value for any analyte exceeds 10%, the original calibration is no longer considered valid, and a new initial calibration (Section L.2.) must be carried out prior to continuing with sample analysis. A new initial calibration must always be carried out prior to sample analysis whenever major maintenance (column change, source cleaning, filament or multiplier replacement, trap replacement, etc.) is performed on the purge and trap GC/MSD system. Minor maintenance (replacing GC septum or inlet liner, removing a portion of the upper end of the column, etc.) does not automatically necessitate recalibration; however, the system should be evaluated on a case by case basis.

M. Determination of Recovery of 1,2-D, *cis*-1,3-D and *trans*-1,3-D in Soil Using the Low-level Dynatrap Analysis

1. Preparation of Recovery Samples

a. Following analysis of the last calibration standard or check standard, analyze a blank (empty) volatile analysis vial containing a magnetic stir bar. To each vial, add 10 mL distilled/deionized water (automatically added to the vial by the Dynatrap) and 10.0 µL of the 10.0 mg/L IS solution (G.2.) (added manually prior to sealing the vial; if added automatically using the Dynatrap, add 1.0 µL of a 100 mg/L IS solution). Analyze the blank by purge and trap using gas chromatography/mass spectrometry as described in Sections H. and J. Calculate the levels of analytes detected using the current calibration curve for each analyte (Section K.3.). If the concentration of any analyte exceeds the limit of detection or 30 % of the targeted limit of quantitation (Section Q.1.c.), additional blanks should be analyzed until a clean blank is obtained.

b. Obtain a control soil sample from the field sampling location. Weigh a 5.0-g aliquot of the control into an amber volatile analysis vial. Add a magnetic stir bar, IS and 10 mL distilled, deionized water to the sample and analyze it as described for the blank in Section M.1.a., to demonstrate that none of the analytes of interest are detectable in the control sample.

c. Prepare fortified samples at appropriate concentrations by weighing 5.0± 0.05 g aliquots of control soil into amber volatile analysis vials (Note R.8.). Fortify the soils over the range of 0.20-200 µg/kg using the standard solutions, as indicated below:

Low range calibration

Standard Solution Concentration mg/L[a]	Aliquot of Standard Solution µL	Soil Sample Conc. µg/kg	Equivalent Calibration Std. Conc. µg/L
0.10	10	0.2	0.10
0.10	25	0.5	0.25
0.10	50	1.0	0.50
1.0	10	2.0	1.0
1.0	25	5.0	2.5

[a]Section G.1.e.-f.

IS must be added to each calibration standard as described in G.3.a.

High range calibration

Standard Solution Concentration mg/L[a]	Aliquot of Standard Solution µL	Soil Sample Conc. µg/kg	Equivalent Calibration Std. Conc. µg/L
1.0	25	5.0	2.5
1.0	50	10.0	5.0
10.0	10	20.0	10.0
10.0	20	40.0	20.0
10.0	40	80.0	40.0
100.0	10	200.0	100.0

[a]Section G.1.c.-e.

IS must be added to each calibration standard as described in G.3.a.

d. Analyze the fortified recovery samples by purge and trap using gas chromatography/mass spectrometry as described in Sections H. and J.

2. Calculation of Percent Recovery

a. Determine the peak areas for 1,2-D (*m/z* 63, 76), *cis*-1,3-D (*m/z* 75, 112), *trans*-1,3-D (*m/z* 75, 112), and IS (*m/z* 77) for each calibration standard analyzed as part of the current calibration curve (Section K.2.).

b. For each standard, calculate the confirmation ratio for each of the three analytes. The average standard confirmation ratio for each analyte will be used to confirm the presence of that analyte in the soil samples.

For example, using the data for 1,2-D from Figure 5:

Confirmation Ratio = peak area of quantitation ion/peak area confirmation ion

Confirmation Ratio = peak area *m/z* 63 / peak area *m/z* 76

Confirmation Ratio = 1023/538

Confirmation Ratio = 1.90

Positive confirmation of the presence of each analyte in a soil sample is indicated when the confirmation ratio for the sample is in the range of ± 15% of the average found for the standards in the current calibration curve.

c. Prepare a standard curve for each of the analytes and determine the equations for the calibration curves using regression analysis, as described in K.3.

d. Calculate the net concentration (µg/kg) of 1,2-D in each recovery sample by first subtracting the quantitation ratio in the control sample (average ratio if more than one control was analyzed) from that of the recovery sample. Substitute the net quantitation ratio obtained into the calibration curve equation and solve for the concentration (µg/L). Multiply the µg/L purged by the method factor to determine the soil concentration (µg/kg).

For example, using power regression and the 1,2-D data from Figures 8, 11 and 14:

$$\frac{\text{1,2-D Conc.}}{(\mu g/L)} = \left(\frac{((\text{Sample Quant. Ratio}) - (\text{Control Quant. Ratio})}{\text{constant}}\right)^{1/\text{exponent}}$$

$$\frac{\text{1,2-D Conc.}}{(\mu g/L)} = \left(\frac{(0.00615 - 0)}{0.06498}\right)^{1/1.02715}$$

1,2-D Conc. = 0.10073 µg/L

1,2-D Conc. (µg/kg) = 0.10073 µg/L x Method Factor (L/kg)

where: Method Factor = extraction volume (L) /sample weight (kg)
=0.01 L / 0.00502 kg
=1.99 L/kg

1,2-D Conc. (µg/kg) = 0.10073 µg/L x 1.99 L/kg

1,2-D Conc. = 0.201 µg/kg

e. Determine the percent recovery by dividing the net concentration found for each recovery sample by the theoretical concentration added.

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

$$\text{Recovery} = \frac{0.201\ \mu g/kg}{0.199\ \mu g/kg} \times 100$$

Recovery = 101%

f. For each analyte, determine the net concentration and corresponding percent recovery as described for 1,2-D in Section M.2.d.-e.

The average recovery for each analyte in a given sample set will be used to correct for daily method efficiency.

N. Determination of VOAs in Soil using the Low-level Dynatrap Analysis

1. Analysis of samples

a. Determine the desired calibration range for sample analysis based upon anticipated sample residue levels. Calibrate the instrument/check calibration over the appropriate range for the sample set as described in Section K.

b. Prepare and analyze blank, control, and recovery samples as described in Section M.1. Prior to commencing with analysis of soil samples, analyze a blank volatile analysis vial (Section M.1.a.) to determine that the system is uncontaminated.

c. Weigh 5.0 ± 0.05-g portions of soil samples into amber volatile analysis vials (Note R.8.). Add a magnetic stir bar to each vial. Add 10.0 µL of the 10.0 mg/L IS solution (G.2.) to each sample. (The IS can also be added automatically using the Dynatrap as described in Section M.1.a.) Seal the vials and load them into the carousel of the Dynatrap.

d. Analyze the samples by purge and trap using gas chromatography/mass spectrometry, as described in Sections H. and J.

2. Determination of Soil Moisture

a. Accurately weigh an approximately 10-g portion of soil into a tared weighing dish and record the weight to the nearest 0.01 g.

b. Place the sample in an oven at approximately 110 °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 reweigh.

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

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

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

3. Calculation of analyte concentration in soil

a. Determine the soil concentration of each analyte by substituting the quantitation ratio into the equation for the corresponding standard calibration curve (Section K.3.), and calculating the µg/L purged. Calculate the uncorrected (gross) µg/kg in the soil by multiplying the µg/L by the method factor.

For example, using the 1,2-D data from Figures 11 and 14, the uncorrected concentration is calculated as follows:

$$\frac{\text{1,2-D Conc.}}{(\mu g / L)} = \left(\frac{\text{(Quant. Ratio)}}{0.06498}\right)^{1/1.02715}$$

$$\frac{\text{1,2-D Conc.}}{(\mu g / L)} = \left(\frac{0.00615}{0.06498}\right)^{1/1.02715}$$

$$\text{1,2-D Conc.} = 0.10073\ \mu g/L$$

$$\text{1,2-D Conc. } (\mu g/kg) = 0.10073\ \mu g/L \times \text{Method Factor (L/kg)}$$

$$\text{1,2-D Conc. } (\mu g/kg) = 0.10073 \times (0.01\ L/0.00502\ kg)$$

$$\text{1,2-D Conc.} = 0.201\ \mu g/kg$$

Correct the amount found for percent moisture of the soil sample as follows:

$$\mu g/kg = (\mu g/kg) \times (1 + (\% \text{ Moisture}/100)$$

b. The uncorrected results as determined in Section N.3.a. must be reported. In addition, the results can be corrected for method recovery using the following procedure:

Calculate the mean % recovery of each analyte for the recovery samples analyzed with the treated samples on the same day.

Determine the corrected analyte concentration in the soil samples as follows (Assume a 1,2-D average percent recovery of 115 for purpose of the example.):

$$\frac{1,2\text{-D Conc.}}{(\text{corrected } \mu g / L)} = \left(\frac{\text{uncorrected } 1,2\text{-D}(\mu g / kg) \times 100}{\% \text{ Recovery}}\right)$$

$$\frac{1,2\text{-D Conc.}}{(\text{corrected } \mu g / L)} = \left(\frac{0.201 \times 100}{115}\right)$$

$$1,2\text{-D Conc.} = 0.175\ \mu g/kg$$

c. Any sample giving a quantitation ratio response for one or more of the analytes, which is greater than 10 percent above that of the highest standard in the current calibration range, must be reanalyzed following recalibration of the instrument in the appropriate concentration range. Samples with estimated residues greater than 200 µg/kg should be analyzed using the high-level analysis (Section O.).

d. When analyzing samples using the high range calibration of the low-level analysis, any sample giving a quantitation ratio response for one or more of the analytes, which is greater than 10 percent below that of the lowest standard in the current calibration range, must be reanalyzed following recalibration of the instrument in the appropriate concentration range.

O. Determination of Recovery of 1,2-D, *cis*-1,3-D and *trans*-1,3-D in Soil Using the High-level Tekmar Analysis

1. Preparation of Recovery Samples

a. Following analysis of the last calibration standard/check standard, rinse the sample sparge tube(s) twice with distilled/deionized water. Analyze a blank distilled/deionized water sample by removing the plunger from the 25 mL gas-tight Luer Lock syringe, attaching the closed syringe valve to the Leur Lock, and pouring the water into the open barrel of the syringe until it is almost full. (Do not draw the water up into the syringe.) Replace the syringe plunger in the barrel, open the valve, expel any air from the syringe and adjust the volume to exactly 25.0 mL. Remove the syringe valve, pull the plunger back slightly, and inject 2.5 µL of the 10.0 mg/L IS (Section G.2.) through the Luer Lock tip into the sample. Open the loading valve on the Tekmar purge and trap device, load the sample into the sparge tube, and begin the analysis. (Sections I. and J.) Calculate the levels of analytes detected using the current calibration curve for each analyte (Section L.3.). If the concentration of any analyte exceeds the limit of detection or 30% of the targeted limit of quantitation, additional blanks should be run until a clean blank is obtained.

b. Obtain a control soil sample from the field sampling location. Weigh 5.0-g portions of the control into 11-dram vials with poly-seal caps (Section E.14.) (Note R.8.). Use one sample as a control and fortify the remaining samples at appropriate levels according to the table below:

Standard Solution Concentration mg/L[a]	Aliquot of Standard Solution µL	Soil Sample Conc. µg/kg	Equivalent Calibration Std. Conc. µg/L
100	10	200	0.1
100	50	1000	0.5
1000	25	5000	2.5
10000	10	20000	10
10000	40	80000	40
10000	80	160000	80

[a]Section G.1.c., i.-j.

c. Add 10.0 mL methanol to each soil sample, seal the vial, vortex for approximately 15 seconds, and centrifuge at 2500 rpm for 3 minutes.

d. Analyze each control and fortified recovery sample by loading the Luer Lock syringe with distilled/deionized water and adjusting the volume to exactly 25.0 mL, as described in section O.1.a. Remove the syringe valve and using a gas-tight syringe transfer 25.0 µL of the soil methanol extract (Section O.c.) into the water through the Luer Lock tip. Inject 2.5 µL of the 10.0 mg/L IS (Section G.2.) through the Luer Lock tip into the water sample. Open the loading valve on the purge and trap device, load the sample into the sparge tube, and begin the analysis. (Sections I. and J.)

2. Calculation of Percent Recovery

a. Calculate the average standard confirmation ratio and standard curves for each analyte as described in M.2.a.-c.

b. Determine the net concentration (µg/kg) of 1,2-D in each recovery sample by first subtracting the quantitation ratio for the control sample (average ratio if more than one control was analyzed) from that of the recovery sample. Substitute the net quantitation ratio obtained into the calibration curve equation and solve for the concentration (µg/L), as shown in Section M.2.d.

Multiply the µg/L purged by the method factor to determine the soil concentration.

$$\underset{(\mu g/kg)}{\text{1,2-D Conc.}} = \mu g/L \times \text{Method Factor (L/kg)}$$

where:

$$\text{Method Factor} = \left(\frac{\text{extraction vol. (L)}}{\text{Sample wt. (kg)}}\right) \times \left(\frac{\text{final aqueous vol. (L)}}{\text{methanol aliquot vol. (L)}}\right)$$

$$= \left(\frac{0.01\ \text{L}}{0.005\ \text{kg}}\right) \times \left(\frac{0.025\ \text{L}}{0.000025\ \text{L}}\right)$$

$$= 2000\ \text{L/kg}$$

c. Determine the percent recovery by dividing the net concentration of each recovery sample by the theoretical concentration added, as shown in Section M.2.e.

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

d. For each analyte, determine the net concentration and corresponding percent recovery as described for 1,2-D in Section O.2.b.-c.

The average percent recovery for each analyte in a given sample set will be used to correct for daily method efficiency.

P. Determination of VOAs in Soil using the High-level Tekmar Method

1. Calibrate the instrument/check calibration over the range indicated in section G.4.
2. Prepare and analyze blank, control, and recovery samples as described in Section O.1. Rinse the sample sparge tube(s) with distilled/deionized water. If standards or recovery samples fortified at 10 µg/L or higher were analyzed in any sparge tubes prior to initiating the analysis of a set of samples, analyze a blank as described in section O.1.a. in order to ensure that carryover in each of the tubes is not a problem.
3. Weigh 5.0-g portions of soil samples into 11-dram vials and analyze, as described in Sections O.1. c.-d. (Note R.8.)
4. Calculate the soil concentration (µg/kg) of each analyte by substituting the quantitation ratio into the equation for the corresponding standard calibration curve (Section L.3.), calculating the µg/L purged as shown on Section N.3.a. Calculate the uncorrected (gross) µg/kg in the soil by multiplying the µg/L by the method factor, as follows:

$$(\mu\text{g/kg}) = \mu\text{g/L} \times \text{method factor (L/kg)}$$

where:

$$\text{Method Factor} = \left(\frac{\text{extraction vol. (L)}}{\text{Sample wt. (kg)}}\right) \times \left(\frac{\text{final aqueous vol. (L)}}{\text{methanol aliquot vol. (L)}}\right)$$

$$= \left(\frac{0.01\ \text{L}}{0.005\ \text{kg}}\right) \times \left(\frac{0.025\ \text{L}}{0.000025\ \text{L}}\right)$$

$$= 2000\ \text{L/kg}$$

5. The uncorrected results as determined in section P.4. must be reported. In addition, the results can be corrected for method recovery using the following procedure (See Section N.3.b. for example.):

a. Calculate the mean % recovery of each analyte for the recovery samples analyzed with the treated samples.

b. Determine the corrected analyte concentration in the soil samples as follows:

$$\frac{\text{1,2 - D Conc.}}{(\text{corrected } \mu g / L)} = \left(\frac{\text{uncorrected 1,2 - D}(\mu g / kg) \times 100}{\% \text{ Recovery}} \right)$$

6. Any sample giving a quantitation ratio response, for one or more of the analytes, which is greater than 10 percent below that of the lowest standard in the current calibration range must be reanalyzed using the low-level analysis (Section N.). Any sample giving a quantitation ratio response greater than 10 percent above that of the highest standard in the calibration range must be reanalyzed. A fresh soil aliquot must be extracted with methanol, and the resulting extract diluted appropriately with water to bring the sample response into the calibration range. The additional dilution must then be accounted for in the calculation of the result (Section P.4.) as follows:

1,2-D Conc. (µg/kg) = µg/L x method factor (L/kg) x additional dilution factor

Q. Results and Discussion

1. Method Validation

a. Recovery Levels and Precision

A method validation study was conducted to determine the recovery levels and the precision of the method for 1,2-D, *cis*-1,3-D, and *trans*-1,3-D in soil; the results from the low-level analysis are summarized in Tables I-III, respectively. The results from the high-level analysis of 1,2-D, *cis*-1,3-D, and *trans*-1,3-D are summarized in Tables IV-VI, respectively.

Recovery values of 1,2-D from soil samples fortified over the concentration range of 0.20-200 µg/kg and analyzed using the low-level method, averaged 111% with one standard deviation equal to 8% (Table I). Recovery values of 1,2-D from soil samples fortified over the concentration range of 200-160000 µg/kg and analyzed using the high-level method, averaged 83% with one standard deviation equal to 8% (Table II).

Recovery values of *cis*-1,3-D from soil samples fortified over the concentration range 0.20-200 µg/kg averaged 100% with one standard deviation equal to 8% (Table III). Recovery values of *cis*-1,3-D from soil samples fortified over the concentration range of 200-160000 µg/kg and analyzed using the high-level method, averaged 78% with one standard deviation equal to 8% (Table IV).

Recovery values of *trans*-1,3-D from soil samples fortified over the concentration range 0.20-200 µg/kg averaged 88% with one standard deviation equal to 9% (Table V). Recovery values of *trans*-1,3-D from soil samples fortified over the concentration range of 200-160000 µg/kg and analyzed using the high-level method, averaged 80% with one standard deviation equal to 12% (Table VI).

b. Standard Curve

The average coefficient of determination (r^2) for the power regression equations describing the detector response as a function of the standard calibration curve concentration were greater than 0.99 for all three analytes in all calibration ranges.

c. Calculated Limits of Quantitation and Detection

Following established guidelines (6), the Limits of Quantitation (LOQ) and Limits of Detection (LOD) were calculated using the standard deviation from the 0.20 µg/kg recovery results for each of the three analytes. The LOQ was calculated as ten times the standard deviation (10*s*), and the LOD was calculated as three times the standard deviation (3*s*) of the results of the analysis of eight samples. The results are summarized in Tables VII-IX.

The calculated statistics support an LOQ between 0.068 and 0.13 µg/kg for the three analytes, lower than the targeted method LOQ of 0.20 µg/kg; however, results should not be quantified at levels below which no recovery samples have been analyzed.

2. Confirmation of Residue Identity

Confirmation of the presence of residues is described in Section M.2.b. For each of the three analytes, confirmation is by comparison of the retention time (gas chromatography) as well as the confirmation ratios resulting from selected ion monitoring (mass spectrometry). Positive confirmation of the presence of each analyte is indicated when the confirmation ratio for the sample is in the range of ± 15% of the average found for the corresponding standards. If additional confirmation is required beyond that discussed in this method, the mass spectrum of each of the three analytes contain additional ions that may be used for confirmation.

3. Assay Time

A typical analytical run would consist of a minimum of six calibration standards for the low-level analysis or 12 for the high-level analysis, a reagent blank, a control (a non-fortified sample), a minimum of two fortified controls (one of which must be at the LOQ when analyzing a sample set in the low range of the low-level analysis), and sixteen samples. This typical analytical run could be prepared in approximately 8 hours, with the purge and trap/chromatographic analysis continuing into the same evening.

As indicated in Sections K.1. and L.1., once the instrument has been calibrated, a calibration check can be done every twelve hours by analyzing one or two standards to verify that the existing calibration curve is still valid. This reduces the amount of time required for daily calibration, and allows the analysis of a greater number of treated samples per day. In addition, the Dynatech can be calibrated in both the low and high range prior to analysis of samples. The sample results can then be calculated using the appropriate curve, increasing the acceptable range for sample responses and ultimately reducing the number of reassays required of samples which fall out of the standard curve range.

R. Notes

1. 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.

2. The filters are used in the carrier gas supply lines to purify the helium entering the gas chromatograph and sparging apparatus.

3. Vials (40 mL) appropriate for volatile organic analysis are available from a variety of vendors. Regardless of which vendor the vials are purchased from, a check should be done to evaluate the vials and septa for potential interferences in the purge and trap analysis. This is easily done by analyzing three empty vials using the Dynatrap/GC/MSD conditions described in this method and evaluating the resulting chromatograms for interferences at the retention times of the analytes of interest.

4. An alternate method for transferring the neat standards to the methanol contained in the volumetric flask is to tare a gas-tight syringe, draw an amount of the neat standard into the syringe, reweigh the syringe determining the weight of the standard, and transfer the standard from the syringe to the flask by injecting it below the surface of the methanol contained in the flask. Rinse the syringe into the flask to be certain that the analyte contained in the syringe is completely transferred to the flask.

5. The purge and trap concentrator is interfaced with the gas chromatograph by cutting the GC helium inlet line approximately 4 cm from its entry into the injection port. The cut end of the helium line is routed to the concentrator, and the concentrator's transfer line is connected to the remaining short section of tubing leading to the injector. The flow from the purge and trap concentrator is 20 mL/min and the flow through the column is approximately 1 mL/min, therefore a 19:1 split will occur. The injector should be run in the split mode at all times. While this technique appears to sacrifice sensitivity by splitting off 95% of the analytes, it permits the use of normal-dimension capillary columns (0.18 or 0.25 mm i.d.), rather than megabore columns which necessitate the use of jet separators or cryogenic interfaces. By virtue of the superior resolution possible with the 0.25 mm i.d. capillary column, the desired limits of quantitation (0.20 μg/kg) are readily attained for all analytes.

6. The mass spectrometer should be tuned prior to analysis of a set of calibration standards and generation of a calibration curve. Once the instrument has been calibrated it should not be tuned again until just prior to analysis of a fresh set of calibration standards for generation of a new calibration curve.

7. It is possible to load all standards in a calibration curve onto the Tekmar 2016 autosampler prior to beginning the analysis; however, this then requires rinsing of all sparge tubes used and confirming the absence of carryover prior to analyzing samples. Using a single sparge tube to analyze consecutively loaded standards confines the potential for contamination and need for cleaning to a single tube.

8. If soil aliquots are weighed to 5.0 ± 0.05 g, a value of 5.0 may be used to calculate the soil sample concentration. If less precision is used in weighing the soil aliquot, the exact weight must be recorded and used in the calculation of soil concentration (fortified and/or actual).

S. References

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Table I. Recovery of 1,2-Dichloropropane from Soil Using the Low-level Method

Sample Number	Date of Analysis	1,2-D, µg/kg Added	1,2-D, µg/kg Found	Percent Recovery[a]
M465 A	04-Oct-94	0.00	0.00	--
M465 B	04-Oct-94	0.00	0.00	--
M465 E	04-Oct-94	0.00	0.00	--
M465 A	20,21-Oct-94	0.00	0.00	--
M465 B	20,21-Oct-94	0.00	0.00	--
M465 E	20,21-Oct-94	0.00	0.00	--
M465 A	20,21-Oct-94	0.100	<0.20	NA[b]
M465 B	20,21-Oct-94	0.099	<0.20	NA[b]
M465 E	20,21-Oct-94	0.100	<0.20	NA[b]
M465 A	20,21-Oct-1994	0.201	0.229	114
M465 B	20,21-Oct-1994	0.202	0.214	106
M465 E	20,21-Oct-1994	0.199	0.201	101
M465 A	20,21-Oct-1994	0.200	0.233	116
M465 B	20,21-Oct-1994	0.201	0.233	116
M465 E	20,21-Oct-1994	0.200	0.217	109
M465 A	20,21-Oct-1994	0.200	0.238	119
M465 B	20,21-Oct-1994	0.200	0.237	118
M465 E	20,21-Oct-1994	1.00	1.18	117
M465 A	20,21-Oct-1994	1.00	1.25	125
M465 A	04-Oct-94	4.95	4.83	98
M465 B	04-Oct-94	5.00	4.74	95
M465 B	20,21-Oct-1994	4.96	5.94	120
M465 E	20,21-Oct-1994	4.99	5.74	115
M465 E	04-Oct-94	20.0	23.8	119
M465 A	04-Oct-94	19.8	21.4	108
M465 B	04-Oct-94	79.4	82.9	104
M465 E	04-Oct-94	79.2	85.8	108
M465 A	04-Oct-94	198	220	111
M465 B	04-Oct-94	199	196	98
			$\bar{x}$ =	111
			s =	8
			n =	20

[a] Calculations performed using exact weights and more digits than displayed.
[b] NA=not applicable. The residue was below the 0.20 µg/kg limit of quantitation.

Table II. Recovery of 1,2-Dichloropropane from Soil Using the High-level Method

Sample Number	Date of Analysis	1,2-D, µg/kg Added	1,2-D, µg/kg Found	Percent Recovery[a]
M465 A	12,13-Oct-94	0.00	0.00	--
M465 B	12,13-Oct-94	0.00	0.00	--
M465 E	12,13-Oct-94	0.00	0.00	--
M465 A	12,13-Oct-94	199	155	78
M465 B	12,13-Oct-94	199	147	74
M465 E	12,13-Oct-94	200	158	79
M465 A	12,13-Oct-94	200	152	76
M465 B	12,13-Oct-94	198	164	83
M465 E	12,13-Oct-94	198	172	87
M465 A	12,13-Oct-94	199	209	105
M465 B	12,13-Oct-94	200	177	88
M465 E	12,13-Oct-94	988	985	100
M465 A	12,13-Oct-94	998	853	85
M465 B	12,13-Oct-94	5000	3808	76
M465 E	12,13-Oct-94	4970	4066	82
M465 A	12,13-Oct-94	19841	15158	76
M465 B	12,13-Oct-94	19920	15982	80
M465 E	12,13-Oct-94	79523	64819	82
M465 A	12,13-Oct-94	79208	64050	81
M465 B	12,13-Oct-94	160000	122631	77
M465 E	12,13-Oct-94	159046	134853	85
			$\bar{x}$ =	83
			s =	8
			n =	18

[a] Calculations performed using exact weights and more digits than displayed.

Table III. Recovery of *cis*-1,3-Dichloropropene from Soil Using the Low-level Method

Sample Number	Date of Analysis	*cis*-1,3-D, µg/kg Added	*cis*-1,3-D, µg/kg Found	Percent Recovery[a]
M465 A	04-Oct-94	0.00	0.00	--
M465 B	04-Oct-94	0.00	0.00	--
M465 E	04-Oct-94	0.00	0.00	--
M465 A	20,21-Oct-1994	0.00	0.00	--
M465 B	20,21-Oct-1994	0.00	0.00	--
M465 E	20,21-Oct-1994	0.00	0.00	--
M465 A	20,21-Oct-1994	0.100	<0.20	NA[b]
M465 B	20,21-Oct-1994	0.099	<0.20	NA[b]
M465 E	20,21-Oct-1994	0.100	<0.20	NA[b]
M465 A	20,21-Oct-1994	0.201	0.203	101
M465 B	20,21-Oct-1994	0.202	0.187	93
M465 E	20,21-Oct-1994	0.199	0.190	95
M465 A	20,21-Oct-1994	0.200	0.203	101
M465 B	20,21-Oct-1994	0.201	0.206	102
M465 E	20,21-Oct-1994	0.200	0.201	100
M465 A	20,21-Oct-1994	0.200	0.203	101
M465 B	20,21-Oct-1994	0.200	0.200	100
M465 E	20,21-Oct-1994	1.00	1.14	113
M465 A	20,21-Oct-1994	1.00	1.12	112
M465 A	20,21-Oct-1994	4.96	4.62	93
M465 B	20,21-Oct-1994	4.99	5.22	105
M465 B	04-Oct-94	4.95	4.48	91
M465 E	04-Oct-94	5.00	4.41	88
M465 E	04-Oct-94	20.0	22.6	113
M465 A	04-Oct-94	19.8	20.0	101
M465 B	04-Oct-94	79.4	73.9	93
M465 E	04-Oct-94	79.2	82.6	104
M465 A	04-Oct-94	198	197	99
M465 B	04-Oct-94	199	175	88
			$\bar{x}$ =	100
			s =	8
			n =	20

[a] Calculations performed using exact weights and more digits than displayed.
[b] NA=not applicable. The residue was below the 0.20 µg/kg limit of quantitation.

Table IV. Recovery of *cis*-1,3-Dichloropropene from Soil Using the High-level Method

Sample Number	Date of Analysis	*cis*-1,3-D, µg/kg Added	*cis*-1,3-D, µg/kg Found	Percent Recovery[a]
M465 A	12,13-Oct-94	0.00	0.00	--
M465 B	12,13-Oct-94	0.00	0.00	--
M465 E	12,13-Oct-94	0.00	0.00	--
M465 A	12,13-Oct-94	199	141	71
M465 B	12,13-Oct-94	199	143	72
M465 E	12,13-Oct-94	200	159	79
M465 A	12,13-Oct-94	200	142	71
M465 B	12,13-Oct-94	198	147	74
M465 E	12,13-Oct-94	198	149	75
M465 A	12,13-Oct-94	199	186	93
M465 B	12,13-Oct-94	200	192	96
M465 E	12,13-Oct-94	988	932	94
M465 A	12,13-Oct-94	998	786	79
M465 B	12,13-Oct-94	5000	3537	71
M465 E	12,13-Oct-94	4970	3760	76
M465 A	12,13-Oct-94	19841	13852	70
M465 B	12,13-Oct-94	19920	14780	74
M465 E	12,13-Oct-94	79523	61460	77
M465 A	12,13-Oct-94	79208	61373	77
M465 B	12,13-Oct-94	160000	117664	74
M465 E	12,13-Oct-94	159046	125021	79
			$\bar{x}$ =	78
			s =	8
			n =	18

[a] Calculations performed using exact weights and more digits than displayed.

Table V. Recovery of *trans*-1,3-Dichloropropene from Soil Using the Low-Level Method

Sample Number	Date of Analysis	*trans*-1,3-D, µg/kg Added	*trans*-1,3-D, µg/kg Found	Percent Recovery[a]
M465 A	04-Oct-94	0.00	0.00	--
M465 B	04-Oct-94	0.00	0.00	--
M465 E	04-Oct-94	0.00	0.00	--
M465 A	20,21-Oct-1994	0.00	0.00	--
M465 B	20,21-Oct-1994	0.00	0.00	--
M465 E	20,21-Oct-1994	0.00	0.00	--
M465 A	20,21-Oct-1994	0.100	<0.20	NA[b]
M465 B	20,21-Oct-1994	0.099	<0.20	NA[b]
M465 E	20,21-Oct-1994	0.100	<0.20	NA[b]
M465 A	20,21-Oct-1994	0.201	0.169	84
M465 B	20,21-Oct-1994	0.202	0.157	77
M465 E	20,21-Oct-1994	0.199	0.167	84
M465 A	20,21-Oct-1994	0.200	0.173	86
M465 B	20,21-Oct-1994	0.201	0.167	83
M465 E	20,21-Oct-1994	0.200	0.181	90
M465 A	20,21-Oct-1994	0.200	0.172	86
M465 B	20,21-Oct-1994	0.200	0.159	79
M465 E	20,21-Oct-1994	1.00	1.07	106
M465 A	20,21-Oct-1994	1.00	0.987	98
M465 A	20,21-Oct-1994	4.96	3.89	78
M465 B	20,21-Oct-1994	4.99	4.82	97
M465 B	04-Oct-94	4.95	4.08	82
M465 E	04-Oct-94	5.00	3.91	78
M465 E	04-Oct-94	20.0	21.0	105
M465 A	04-Oct-94	19.8	18.4	93
M465 B	04-Oct-94	79.4	66.5	84
M465 E	04-Oct-94	79.2	81.4	103
M465 A	04-Oct-94	198	184	93
M465 B	04-Oct-94	199	162	81
			$\bar{x}$ =	88
			s =	9
			n =	20

[a] Calculations performed using exact weights and more digits than displayed.
[b] NA=not applicable. The residue was below the 0.20 µg/kg limit of quantitation.

Table VI. Recovery of *trans*-1,3-Dichloropropene from Soil Using the High-Level Method

Sample Number	Date of Analysis	*trans*-1,3-D, µg/kg Added	*trans*-1,3-D, µg/kg Found	Percent Recovery[a]
M465 A	12,13-Oct-94	0.00	0.00	--
M465 B	12,13-Oct-94	0.00	0.00	--
M465 E	12,13-Oct-94	0.00	0.00	--
M465 A	12,13-Oct-94	199	140	70
M465 B	12,13-Oct-94	199	142	71
M465 E	12,13-Oct-94	200	155	77
M465 A	12,13-Oct-94	200	139	70
M465 B	12,13-Oct-94	198	148	75
M465 E	12,13-Oct-94	198	148	75
M465 A	12,13-Oct-94	199	199	100
M465 B	12,13-Oct-94	200	226	113
M465 E	12,13-Oct-94	988	946	96
M465 A	12,13-Oct-94	998	788	79
M465 B	12,13-Oct-94	5000	3555	71
M465 E	12,13-Oct-94	4970	3832	77
M465 A	12,13-Oct-94	19841	14022	71
M465 B	12,13-Oct-94	19920	15005	75
M465 E	12,13-Oct-94	79523	63642	80
M465 A	12,13-Oct-94	79208	64833	82
M465 B	12,13-Oct-94	160000	122182	76
M465 E	12,13-Oct-94	159046	129035	81
			$\bar{x}$ =	80
			s =	12
			n =	18

[a] Calculations performed using exact weights and more digits than displayed.

Table VII. Calculated Limits of Detection and Quantitation for the Determination of 1,2-Dichloropropane in Soil

Sample Number	Date of Analysis	1,2-D, µg/kg Added	1,2-D, µg/kg Found
M465 A	20,21-Oct-1994	0.201	0.229
M465 B	20,21-Oct-1994	0.202	0.214
M465 E	20,21-Oct-1994	0.199	0.201
M465 A	20,21-Oct-1994	0.200	0.233
M465 B	20,21-Oct-1994	0.201	0.233
M465 E	20,21-Oct-1994	0.200	0.217
M465 A	20,21-Oct-1994	0.200	0.238
M465 B	20,21-Oct-1994	0.200	0.237
		$\bar{x}$ =	0.225
		s =	0.013
		LOD[a] ($3s$) =	0.039
		LOQ[b] ($10s$) =	0.13

[a] LOD = Limit of Detection.
[b] LOQ = Limit of Quantitation.

Table VIII. Calculated Limits of Detection and Quantitation for the Determination of *cis*-1,3-Dichloropropene in Soil

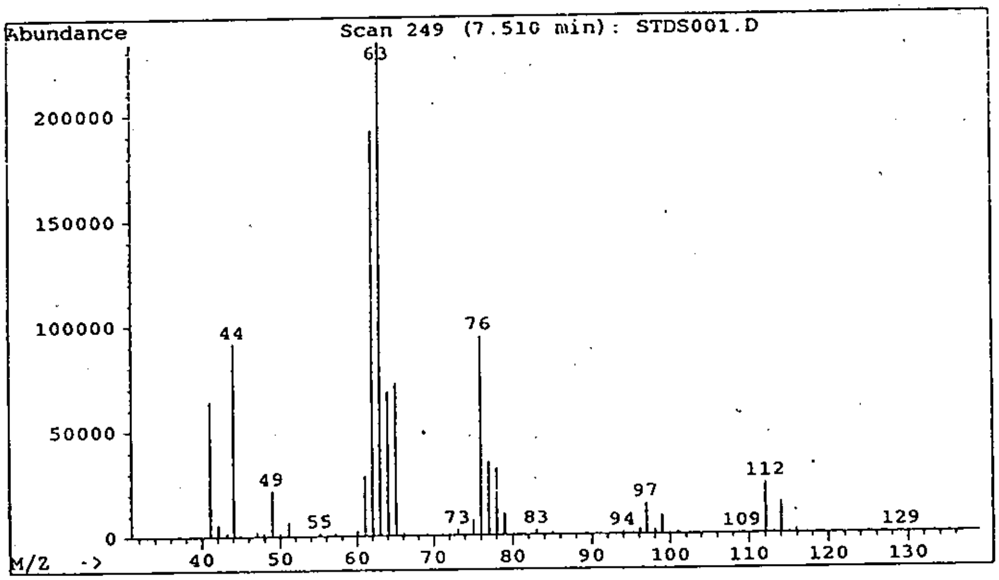
Sample Number	Date of Analysis	*cis*-1,3-D, µg/kg Added	Found
M465 A	20,21-Oct-1994	0.201	0.203
M465 B	20,21-Oct-1994	0.202	0.187
M465 E	20,21-Oct-1994	0.199	0.190
M465 A	20,21-Oct-1994	0.200	0.203
M465 B	20,21-Oct-1994	0.201	0.206
M465 E	20,21-Oct-1994	0.200	0.201
M465 A	20,21-Oct-1994	0.200	0.203
M465 B	20,21-Oct-1994	0.200	0.200
		$\bar{x}$ =	0.199
		s =	0.0068
		LOD[a] ($3s$) =	0.020
		LOQ[b] ($10s$) =	0.068

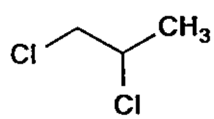
[a] LOD = Limit of Detection.
[b] LOQ = Limit of Quantitation.

Table IX. Calculated Limits of Detection and Quantitation for the Determination of *trans*-1,3-Dichloropropene in Soil

Sample Number	Date of Analysis	*trans*-1,3-D, µg/kg Added	Found
M465 A	20,21-Oct-1994	0.201	0.169
M465 B	20,21-Oct-1994	0.202	0.157
M465 E	20,21-Oct-1994	0.199	0.167
M465 A	20,21-Oct-1994	0.200	0.173
M465 B	20,21-Oct-1994	0.201	0.167
M465 E	20,21-Oct-1994	0.200	0.181
M465 A	20,21-Oct-1994	0.200	0.172
M465 B	20,21-Oct-1994	0.200	0.159
		$\bar{x}$ =	0.168
		s =	0.0077
		LOD[a] ($3s$) =	0.023
		LOQ[b] ($10s$) =	0.077

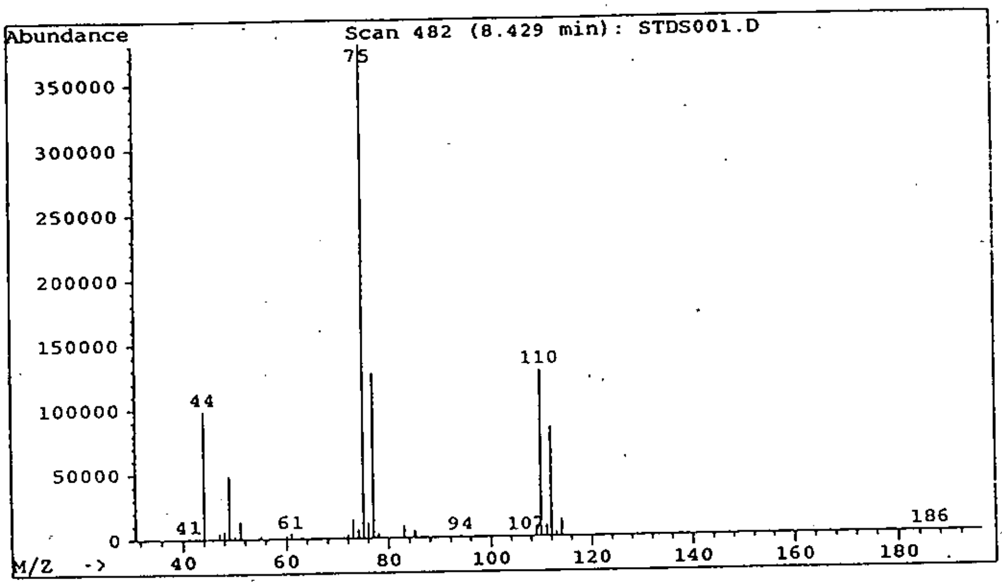
[a] LOD = Limit of Detection.
[b] LOQ = Limit of Quantitation.

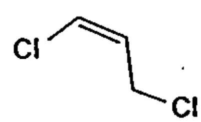




1,2-D
Formula: $C_3H_6Cl_2$
Molecular Weight: 112

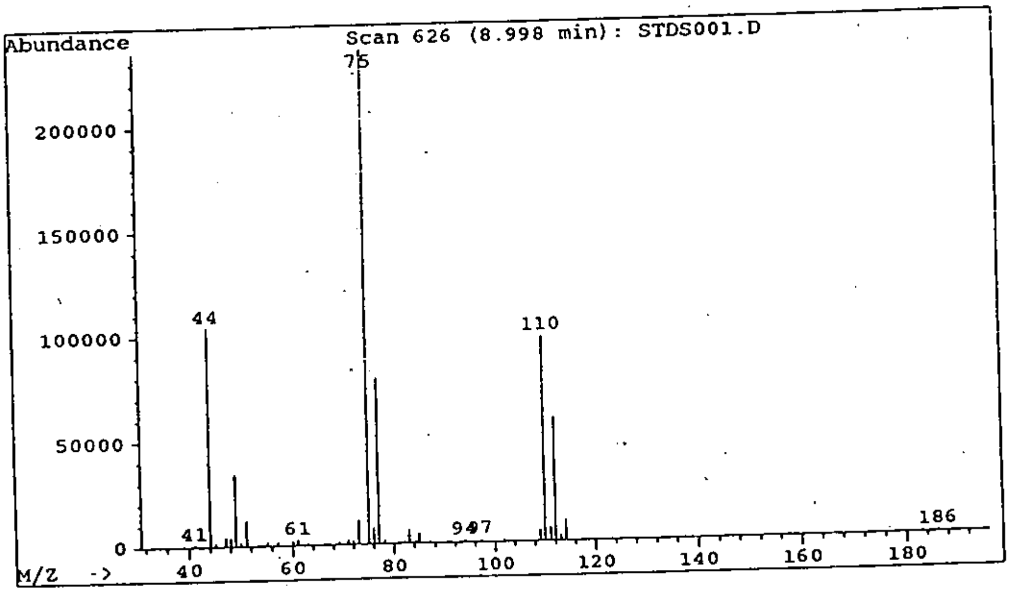
Figure 1. Mass Spectrum of 1,2-Dichloropropane

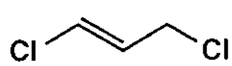




cis-1,3-D
Formula: $C_3H_4Cl_2$
Molecular Weight: 110

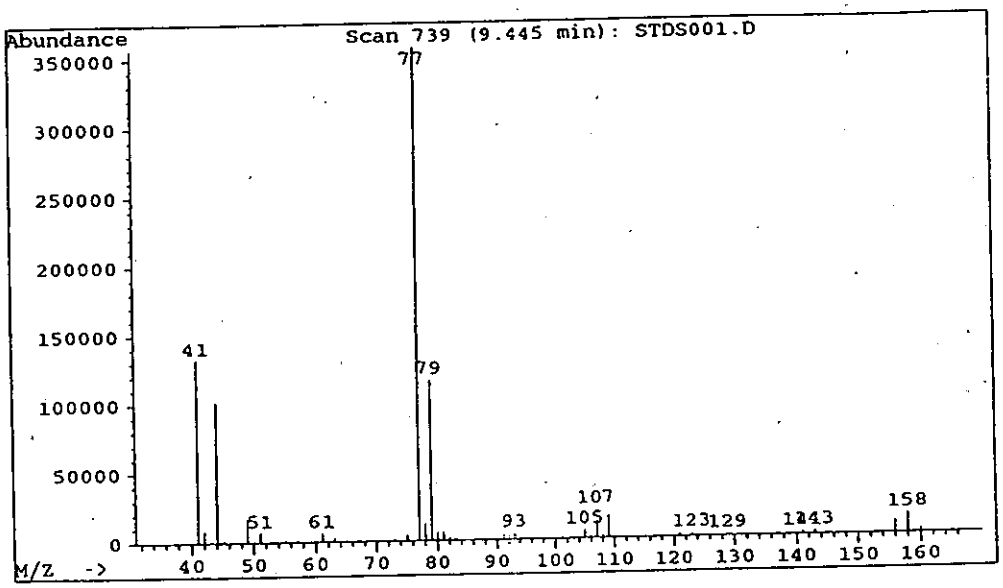
Figure 2. Mass Spectrum of *cis*-1,3-Dichloropropene

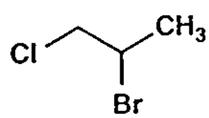




trans-1,3-D
Formula: $C_3H_4Cl_2$
Molecular Weight: 110

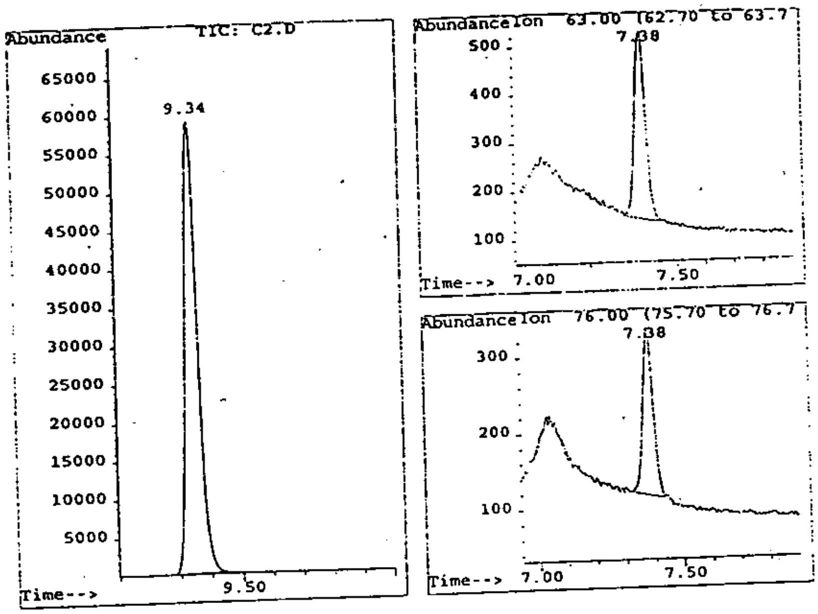
Figure 3. Mass Spectrum of *trans*-1,3-Dichloropropene





2-Bromo-1-chloropropane (IS)
Formula: C_3H_6BrCl
Molecular Weight: 156

Figure 4. Mass Spectrum of 2-Bromo-1-chloropropane

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Data File   : C2.D
ALS Bottle  : 1
Date        : 20 Oct 94  10:53 pm
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

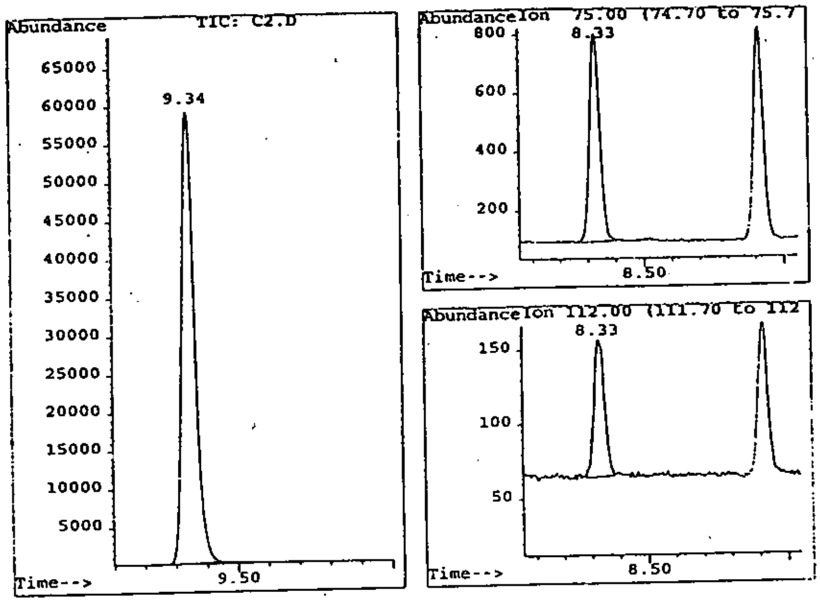
Sample Name: 100ng/L VOA standard
Sample Info: 10uL of 0.10ng/uL,10.0uL of10.0ng/uL ISTD
Operator   : Sean Dolder
```

S. Stark

INTERNAL STANDARD RETENTION TIME	:	9.34
2-BROMO-1-CHLOROPROPANE PEAK AREA	:	167631
1,2-D RETENTION TIME	:	7.38
PEAK AREA (M/Z 63)	:	1023
PEAK AREA (M/Z 76)	:	538
1,2-D CONFIRMATION RATIO OF (m/z 63 / m/z 76)	:	1.90149
1,2-D QUANTITATION RATIO OF (m/z 63 / Instd)	:	0.00610

Equivalent 1,2-D Concentration: 0.20 µg/kg
Average 1,2-D Standard Confirmation Ratio: 1.87

Figure 5. Typical Chromatogram of a 0.10 µg/L Standard Equivalent to 0.20 µg/kg 1,2-D in Soil

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Data File   : C2.D
ALS Bottle  : 1
Date        : 20 Oct 94   10:53 pm
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: 100ng/L VOA standard
Sample Info: 10uL of 0.10ng/uL,10.0uL of10.0ng/uL ISTD
Operator   : Sean Dolder

INTERNAL STANDARD RETENTION TIME          :     9.34
  2-BROMO-1-CHLOROPROPANE PEAK AREA       :   167631

cis-1,3-D RETENTION TIME                  :     8.33
  PEAK AREA (M/Z 75)                      :     1773
  PEAK AREA (M/Z 112)                     :      242

cis-1,3-D CONFIRMATION
  RATIO OF (m/z 75 / m/z 112):              7.32698

cis-1,3-D QUANTITATION
  RATIO OF (m/z 75 / Instd):                0.01058
```
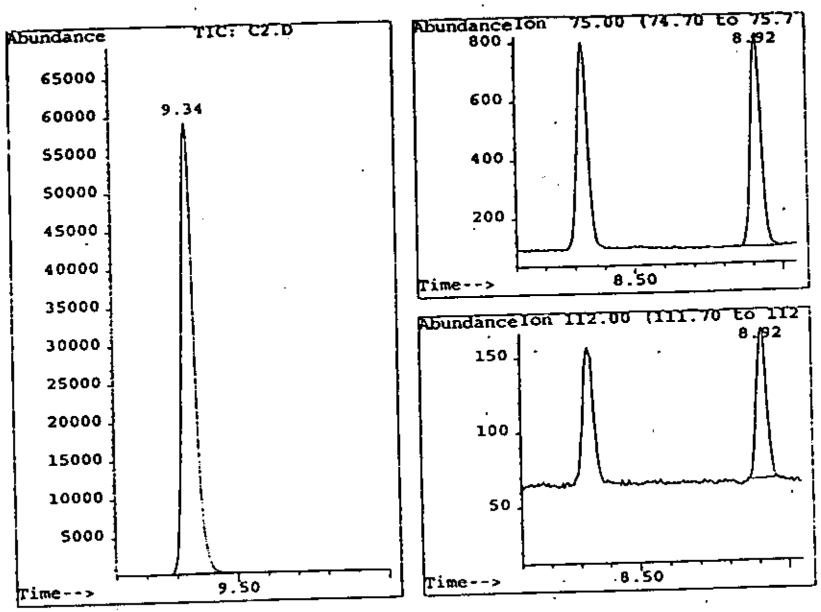

Equivalent *cis*-1,3-D Concentration: 0.20 µg/kg
Average Standard *cis*-1,3-D Confirmation Ratio: 7.38

Figure 6. Typical Chromatogram of a 0.10 µg/L Standard Equivalent to 0.20 µg/kg *cis*-1,3-D in Soil



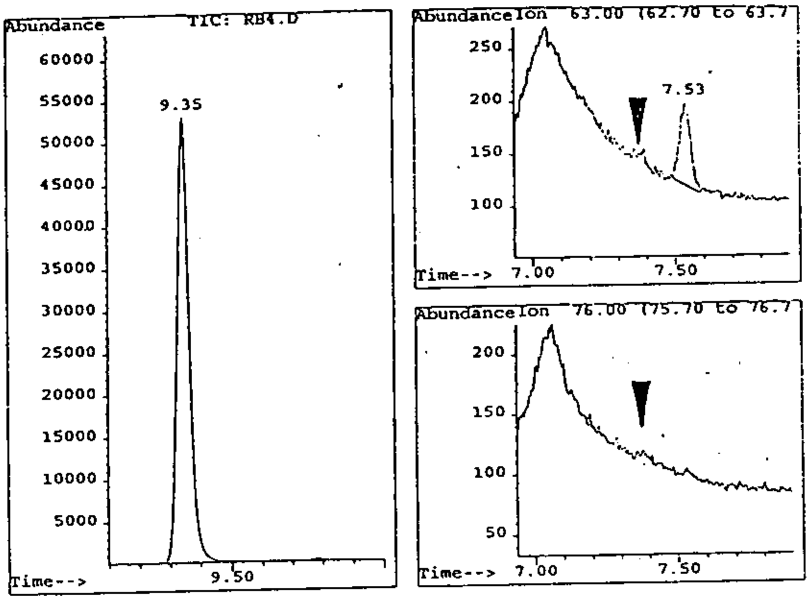

```
Data File   : C2.D
ALS Bottle  : 1
Date        : 20 Oct 94   10:53 pm
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: 100ng/L VOA standard
Sample Info: 10uL of 0.10ng/uL,10.0uL of10.0ng/uL ISTD
Operator   : Sean Dolder
```

SD 27 Oct 94

```
INTERNAL STANDARD RETENTION TIME         :        9.34
  2-BROMO-1-CHLOROPROPANE PEAK AREA      :      167631

trans-1,3-D RETENTION TIME               :        8.92
  PEAK AREA (M/Z 75)                     :        1842
  PEAK AREA (M/Z 112)                    :         245

trans-1,3-D CONFIRMATION
  RATIO OF (m/z 75 / m/z 112):               7.51837

trans-1,3-D QUANTITATION
  RATIO OF (m/z 75 / Instd):                 0.01099
```

Equivalent *trans*-1,3-D Concentration: 0.20 µg/kg
Average Standard *trans*-1,3-D Confirmation Ratio: 7.15

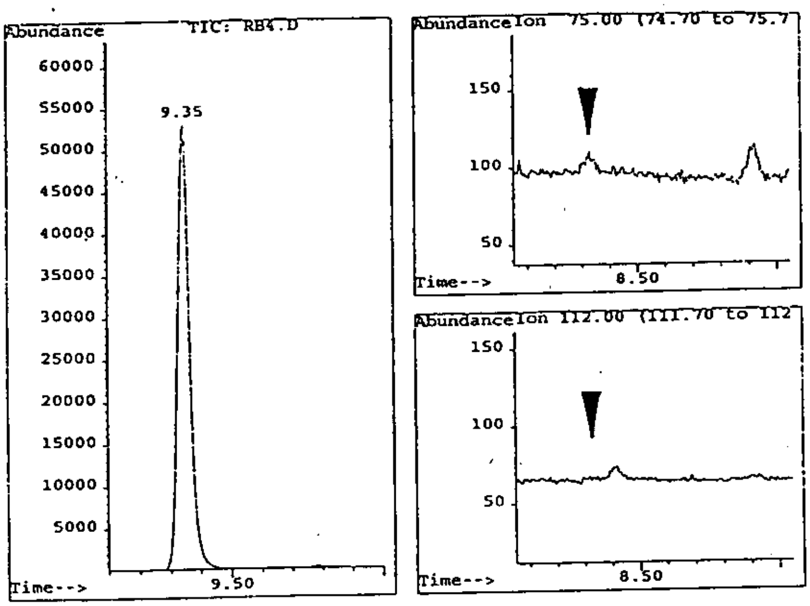
Figure 7. Typical Chromatogram of a 0.10 µg/L Standard Equivalent to 0.20 µg/kg *trans*-1,3-D in Soil




```
Data File   : RB4.D
ALS Bottle  : 1
Date        : 21 Oct 94    4:40 am
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: Soil E reagent blank, 5.03g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder
             SD 27OCT94

INTERNAL STANDARD RETENTION TIME        :     9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA :     146645

1,2-D RETENTION TIME                    :     7.53 waste.
  PEAK AREA (M/Z 63)                    :      206 The retention time of 1,2-D is 7.32.
  PEAK AREA (M/Z 76)                    : NOT FOUND            SD 27OCT94

NO 1,2-D FOUND
```

1,2-D Concentration: 0.0 µg/kg
Average Standard 1,2-D Confirmation Ratio: 1.87

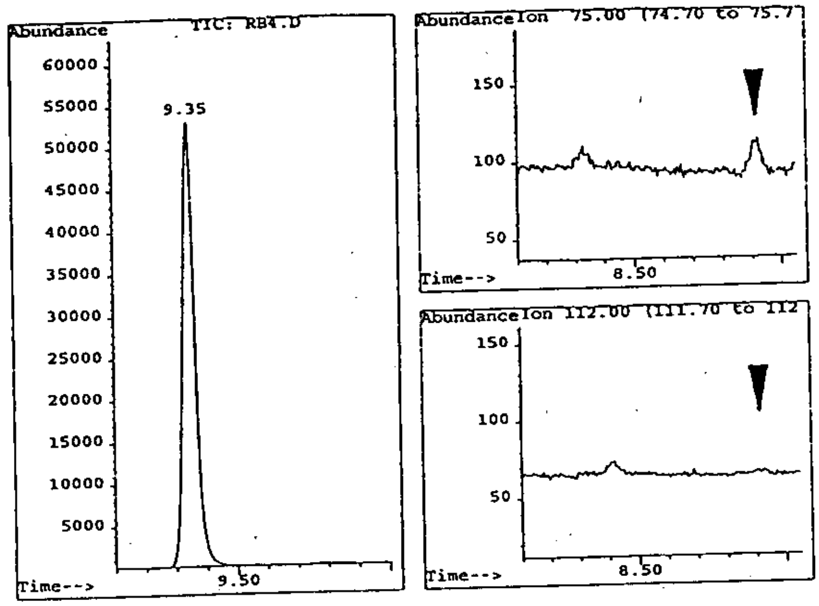
Figure 8. Typical Chromatogram of a Control Soil Sample for the Determination of 1,2-D




```
Data File   : RB4.D
ALS Bottle  : 1
Date        : 21 Oct 94    4:40 am
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: Soil E reagent blank, 5.03g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder

INTERNAL STANDARD RETENTION TIME      :      9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA :    146645

NO cis-1,3-D FOUND
```

cis-1,3-D Concentration: 0.0 µg/kg
Average Standard *cis*-1,3-D Confirmation Ratio: 7.38

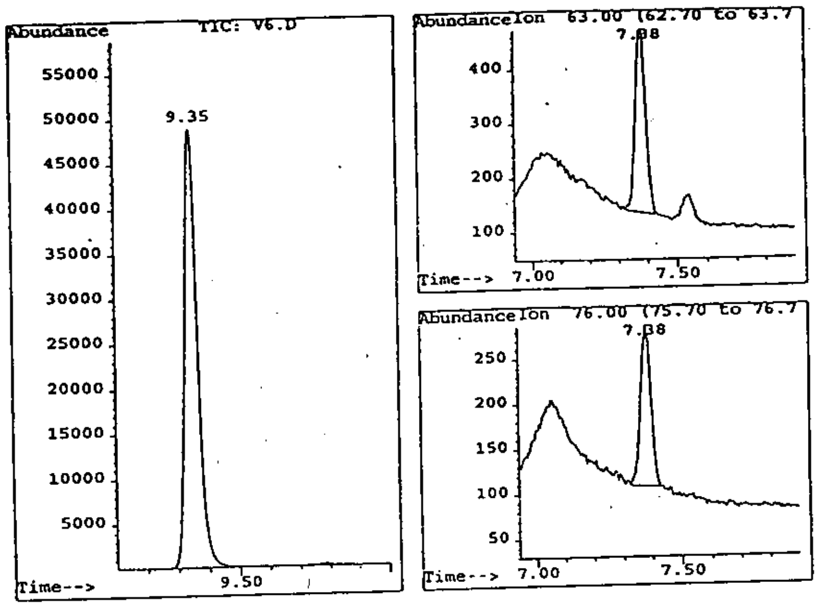
Figure 9. Typical Chromatogram of a Control Soil Sample for the Determination of *cis*-1,3-D




```
Data File   : RB4.D
ALS Bottle  : 1
Date        : 21 Oct 94    4:40 am
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: Soil E reagent blank, 5.03g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder
             SD 27 Oct 94

INTERNAL STANDARD RETENTION TIME        :      9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA :      146645

NO trans-1,3-D FOUND
```

trans-1,3-D Concentration: 0.0 µg/kg
Average Standard *trans*-1,3-D Confirmation Ratio: 7.15

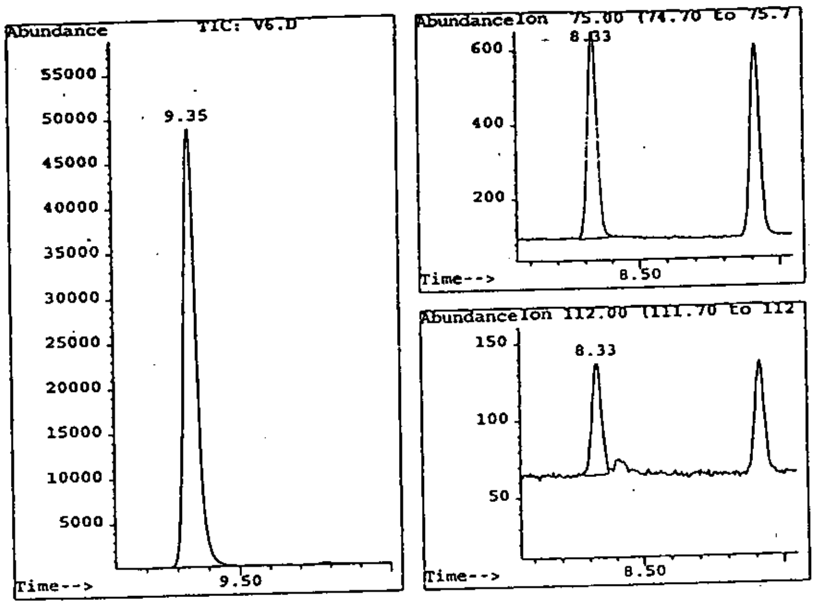
Figure 10. Typical Chromatogram of a Control Soil Sample for the Determination of *trans*-1,3-D




```
Data File  : V6.D
ALS Bottle : 1
Date       : 21 Oct 94    7:33 am
Data Path  : C:\HPCHEM\1\DATA\SD1020CV\
Instrument : GC/MSD S/N 2950A25792

Sample Name: 200ng/kg VOA std., soil E 5.02g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder

INTERNAL STANDARD RETENTION TIME          :      9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA       :    138921

1,2-D RETENTION TIME                      :      7.38
  PEAK AREA (M/Z 63)                      :       854
  PEAK AREA (M/Z 76)                      :       448

1,2-D CONFIRMATION
  RATIO OF (m/z 63 / m/z 76):                 1.90625

1,2-D QUANTITATION
  RATIO OF (m/z 63 / Instd):                  0.00615
```

1,2-D Concentration: 0.201 µg/kg
Average Standard 1,2-D Confirmation Ratio: 1.87
1,2-D Recovery: 101%

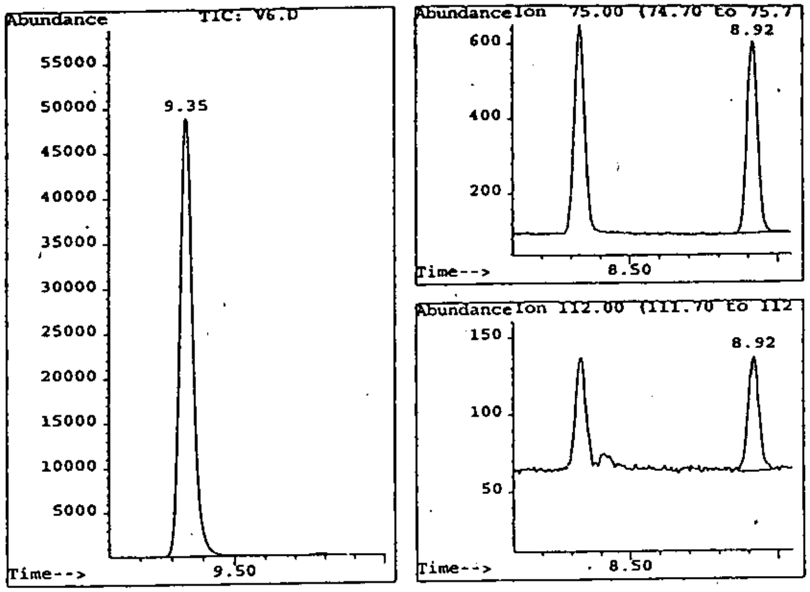
Figure 11. Typical Chromatogram of a Control Soil Sample Fortified with 0.20 µg/kg 1,2-D




```
Data File   : V6.D
ALS Bottle  : 1
Date        : 21 Oct 94    7:33 am
Data Path   : C:\HPCHEM\1\DATA\SD1020CV\
Instrument  : GC/MSD S/N 2950A25792

Sample Name: 200ng/kg VOA std., soil E 5.02g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder

INTERNAL STANDARD RETENTION TIME             :      9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA          :    138921

cis-1,3-D RETENTION TIME                     :      8.33
   PEAK AREA (M/Z 75)                        :      1378
   PEAK AREA (M/Z 112)                       :       182

cis-1,3-D CONFIRMATION
  RATIO OF (m/z 75 / m/z 112):                   7.57362

cis-1,3-D QUANTITATION
  RATIO OF (m/z 75 / Instd):                     0.00992
```

cis-1,3-D Concentration: 0.190 µg/kg
Average Standard *cis*-1,3-D Confirmation Ratio: 7.38
cis-1,3-D Recovery: 95%

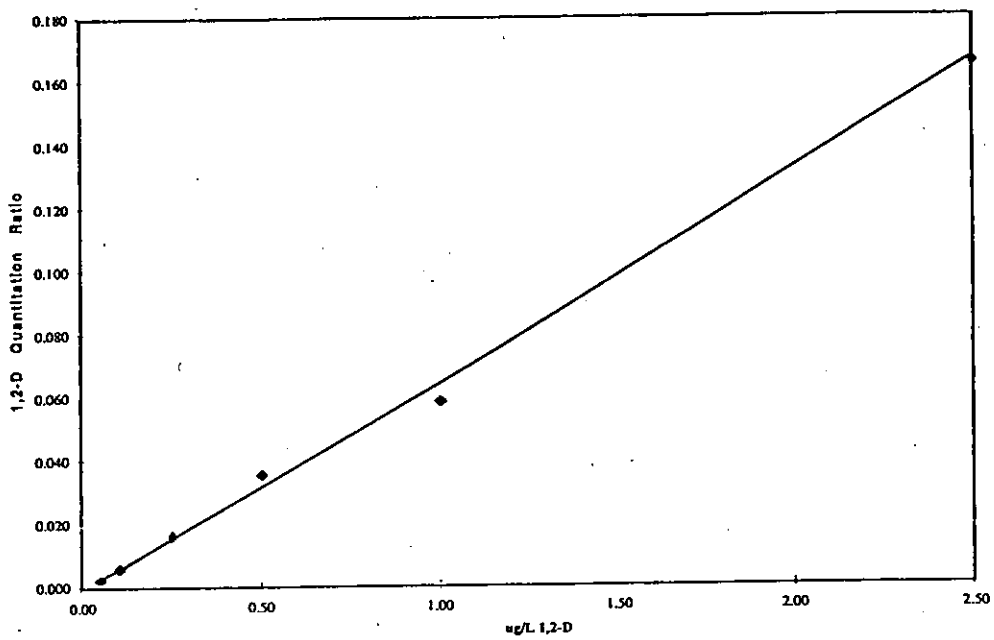
Figure 12. Typical Chromatogram of a Control Soil Sample Fortified with 0.20 µg/kg *cis*-1,3-D




```
Data File  : V6.D
ALS Bottle : 1
Date       : 21 Oct 94    7:33 am
Data Path  : C:\HPCHEM\1\DATA\SD1020CV\
Instrument : GC/MSD S/N 2950A25792

Sample Name: 200ng/kg VOA std., soil E 5.02g
Sample Info: 1.0uL of 10.0ng/uL ISTD
Operator   : Sean Dolder
             SD 27OCT94

INTERNAL STANDARD RETENTION TIME        :      9.35
  2-BROMO-1-CHLOROPROPANE PEAK AREA :        138921

trans-1,3-D RETENTION TIME              :      8.92
  PEAK AREA (M/Z 75)                    :      1269
  PEAK AREA (M/Z 112)                   :       193

trans-1,3-D CONFIRMATION
  RATIO OF (m/z 75 / m/z 112):               6.57358

trans-1,3-D QUANTITATION
  RATIO OF (m/z 75 / Instd):                 0.00913
```

trans-1,3-D Concentration: 0.167 µg/kg
Average Standard *trans*-1,3-D Confirmation Ratio: 7.15
trans-1,3-D Recovery: 84%

Figure 13. Typical Chromatogram of a Control Soil Sample Fortified with 0.20 µg/kg *trans*-1,3-D

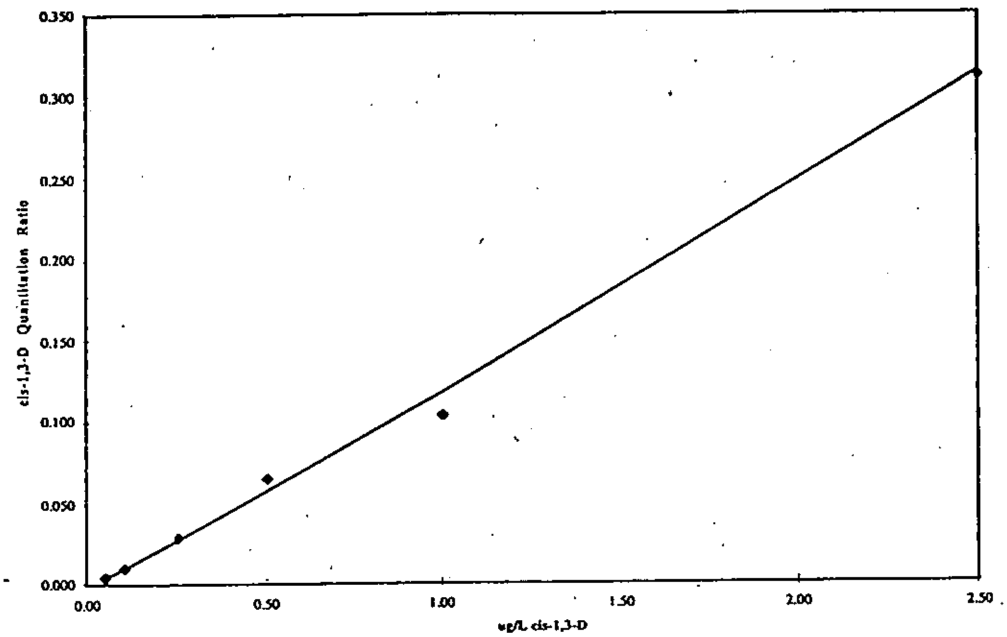


1,2-D Conc. µg/L	1,2-D Quantitation Ratio
0.05	0.00286
0.10	0.00610
0.25	0.01625
0.50	0.03568
1.0	0.05897
2.5	0.16530

Power Regression Equation: $X = (Y/0.06498)^{(1/1.02715)}$
$X = \mu g/L$, $Y =$ Quantitation Ratio

Coefficient of Determination (r^2): 0.9977

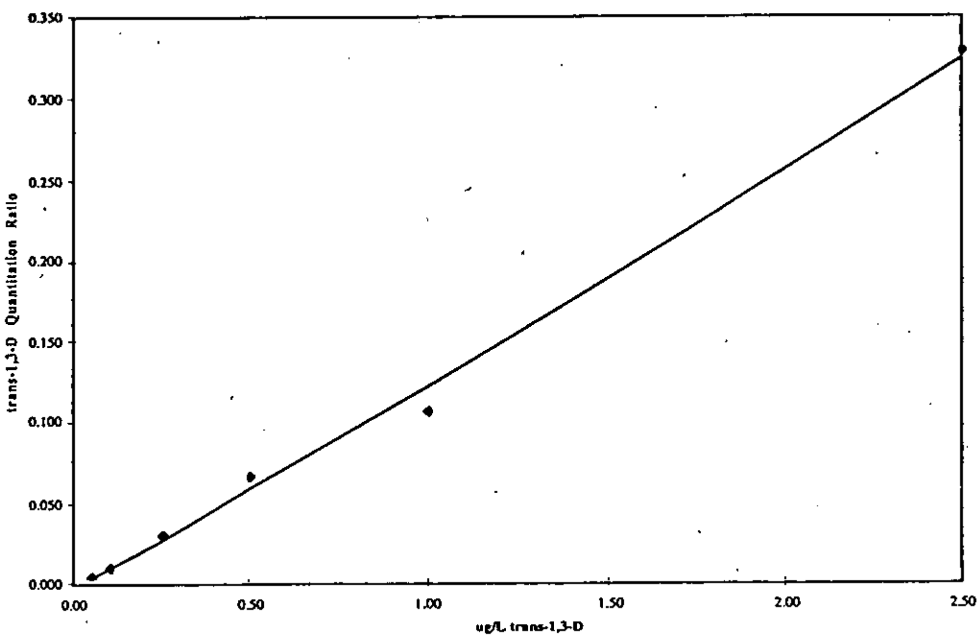
Figure 14. Typical Calibration Curve for the Low-Level (Low Range) Determination of 1,2-Dichloropropane in Soil Samples



cis-1,3-D Conc. µg/L	*cis*-1,3-D Quantitation Ratio
0.05	0.00464
0.10	0.01058
0.25	0.02973
0.50	0.06563
1.0	0.10383
2.5	0.31216

Power Regression Equation: $X = (Y/0.11930)^{(1/1.05761)}$
$X = \mu g/L$, Y = Quantitation Ratio
Coefficient of Determination (r^2): 0.9958

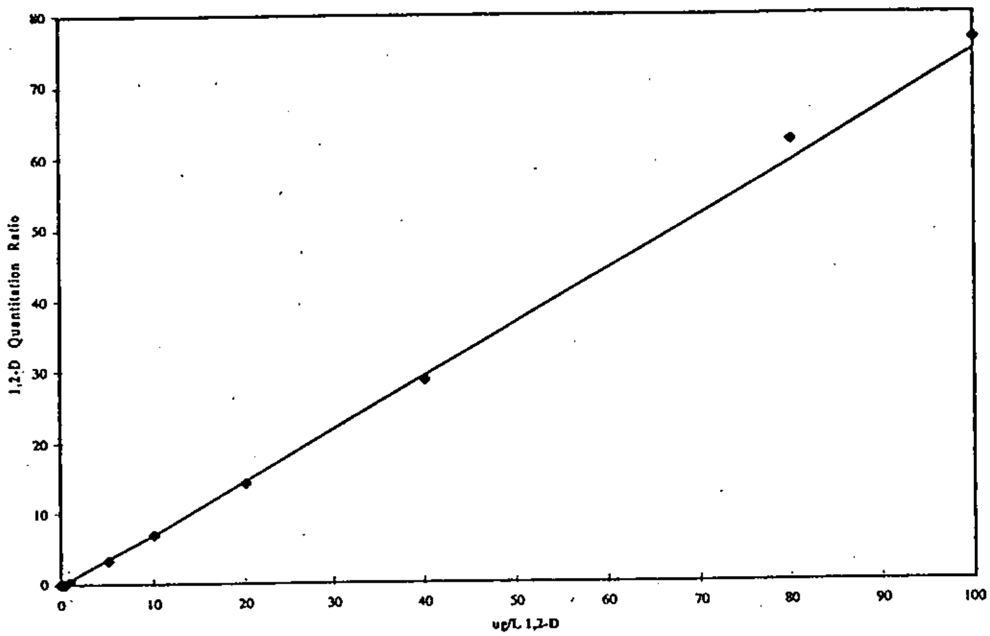
Figure 15. Typical Calibration Curve for the Low-Level (Low Range) Determination of *cis*-1,3-D in Soil Samples



trans-1,3-D Conc. µg/L	*trans*-1,3-D Quantitation Ratio
0.05	0.00498
0.10	0.01099
0.25	0.03121
0.50	0.06705
1.0	0.10676
2.5	0.32868

Power Regression Equation: $X = (Y/0.12383)^{(1/1.05218)}$
$X = \mu g/L$, Y = Quantitation Ratio
Coefficient of Determination (r^2): 0.9961

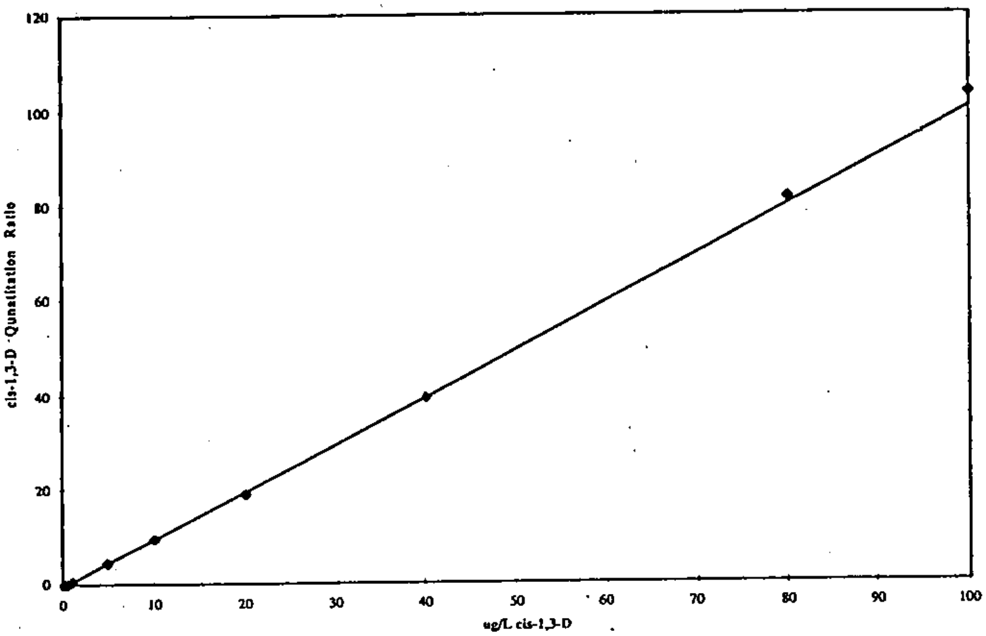
Figure 16. Typical Calibration Curve for the Low-Level (Low Range) Determination of *trans*-1,3-Dichloropropene in Soil Samples



1,2-D Conc. µg/L	1,2-D Quantitation Ratio
0.05	0.03362
0.1	0.06859
0.2	0.1316
0.4	0.2727
0.8	0.5548
1.0	0.7055
5.0	3.3979
10.0	7.1141
20.0	14.2156
40.0	28.9009
80.0	62.3643
100.0	76.4670

Power Regression Equation: $X = (Y/0.69382)^{(1/1.01609)}$
$X = \mu g/L$, $Y =$ Quantitation Ratio
Coefficient of Determination (r^2): 0.9999

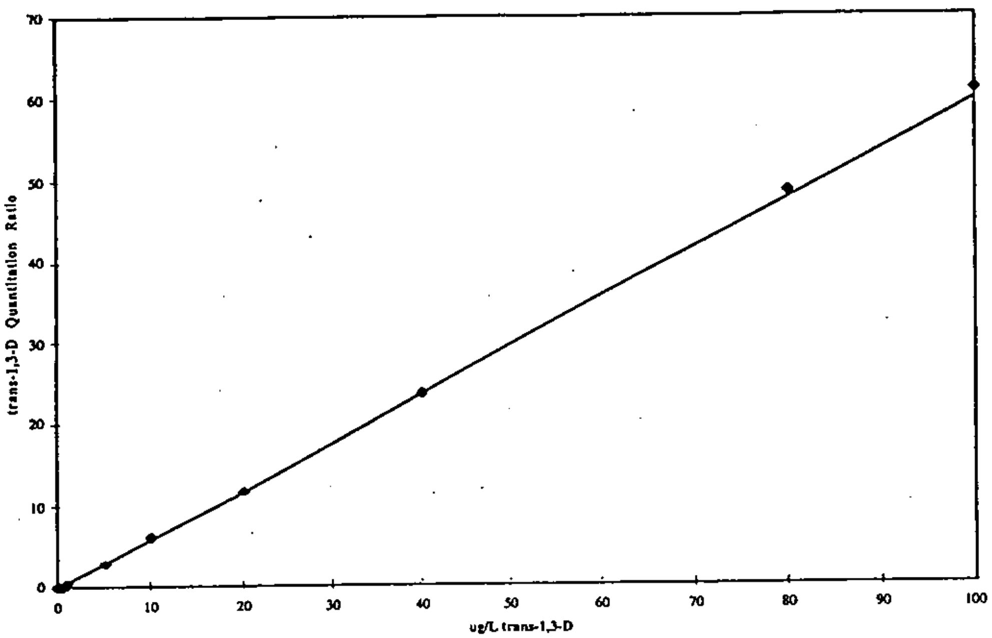
Figure 17. Typical Calibration Curve for the High-Level Determination of 1,2-Dichloropropane in Soil Samples



cis-1,3-D Conc. µg/L	*cis*-1,3-D Quantitation Ratio
0.05	0.04495
0.1	0.09241
0.2	0.1774
0.4	0.3564
0.8	0.7197
1.0	0.9201
5.0	4.6453
10.0	9.7034
20.0	19.0769
40.0	39.2184
80.0	81.4620
100.0	103.377

Power Regression Equation: $X = (Y/0.92536)^{(1/1.01747)}$
$X = \mu g/L$, Y = Quantitation Ratio
Coefficient of Determination (r^2): 0.9999

Figure 18. Typical Calibration Curve for the High-Level Determination of *cis*-1,3-D in Soil Samples



trans-1,3-D Conc. µg/L	*trans*-1,3-D *m/z* 75 / IS*m/z* 77 Peak Area Ratio
0.05	0.02961
0.1	0.05853
0.2	0.1119
0.4	0.2181
0.8	0.4367
1.0	0.5636
5.0	2.8842
10.0	6.0072
20.0	11.654
40.0	23.629
80.0	48.480
100.0	60.957

Power Regression Equation: $X = (Y/0.57496)^{(1/1.00812)}$
$X = \mu g/L$, Y = Quantitation Ratio
Coefficient of Determination (r^2): 0.9999

Figure 19. Typical Calibration Curve for the High-Level Determination of *trans*-1,3-Dichloropropene in Soil Samples

Appendix C: Characterization of Soils

A & L GREAT LAKES LABORATORIES, INC.
3505 Conestoga Drive • Fort Wayne, Indiana 46808-4413 • Phone 219-483-4759

GLP SOIL CHARACTERIZATION

Report Number: F94102-122
Date of Report: 05/20/94

Protocol Number:
ENV 93068

DOWELANCO
JACK R. MILLER
R & D BUILDING A-2/763
9410 ZIONSVILLE ROAD
INDIANAPOLIS IN 46268-1053

VERIFIED AS EXACT
COPY OF ORIGINAL
Initials AMK Date 6-13-94

Sample ID:	A&L Check	M463	M464	M465-A[illegible]	M465-E[illegible]
Lab Number:	50660	50661	50662	50663	50664
pH	5.5	7.6	7.8	7.7	8.5
CEC (meq/100g)	8.46	8.35	12.74	2.20	0.29
O.M. (%)	1.86	0.71	1.80	1.15	0.11
WHC (%) @ 1/10 Bar	23.34	35.24	34.80	22.78	17.57
WHC (%) @ 1/3 Bar	20.80	16.46	22.77	2.86	2.23
WHC (%) @ 1 Bar	15.41	8.83	16.42	2.62	2.11
WHC (%) @ 15 Bar	6.27	4.93	8.92	2.46	1.94
Sand (%)	38.0	34.0	30.0	96.0	96.0
Silt (%)	37.2	53.2	41.2	1.2	1.2
Clay (%)	24.8	12.8	28.8	2.8	2.8
Soil Classification	Loam	Silt Loam	Clay Loam	Sand	Sand
Bulk Density (g/cc)	1.52	1.22	1.30	1.50	1.60

Verified By: Gaylene D. Matthies Date: 5-20-94

A & L GREAT LAKES LABORATORIES, INC.
3505 Conestoga Drive • Fort Wayne, Indiana 46808-4413 • Phone 219-483-4759

GLP SOIL CHARACTERIZATION

Report Number: F94102-122
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DOWELANCO
JACK R. MILLER
R & D BUILDING A-2/763
9410 ZIONSVILLE ROAD
INDIANAPOLIS IN 46268-1053

VERIFIED AS EXACT COPY OF ORIGINAL
Initials: AMK Date: 6-13-94

Sample ID:	~~M465-Bs~~	~~M466~~	~~MET94016~~	A&L Check
Lab Number:	50665	50666	50667	50668
pH	6.6	7.4	7.8	
CEC (meq/100g)	6.01	5.15	6.80	
O.M. (%)	2.78	1.53	0.44	
WHC (%) @ 1/10 Bar	25.48	31.00	27.64	
WHC (%) @ 1/3 Bar	5.07	11.39	15.74	20.77
WHC (%) @ 1 Bar	3.87	7.23	9.92	16.03
WHC (%) @ 15 Bar	2.56	3.43	4.66	6.45
Sand (%)	94.0	60.0	56.0	
Silt (%)	1.2	31.2	29.2	
Clay (%)	4.8	8.8	14.8	
Soil Classification	Sand	Sandy Loam	Sandy Loam	
Bulk Density (g/cc)	1.39	1.35	1.50	

Verified By: Kaylene D. Matthias Date: 5-20-94