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ANALYTICAL METHOD

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TELONE II Soil Fumigant

1. Scope

This method is applicable to the determination of cis-1,3-Dichloropropene-1 and trans-1,3-Dichloropropene-1 and six low level components in TELONE* II soil fumigant. The method is validated over the concentration ranges listed below:

cis-1,3-Dichloropropene-1	0.8 - 93.2%
trans-1,3-Dichloropropene-1	2.4 - 94.2
2-Chloro-2-methylpentane	0.02 - 0.92
2-Chloro-2,3-dimethylbutane	0.02 - 0.91
1,3-Dichloropropane	0.03 - 1.50
1,2,2-Trichloropropane	0.03 - 1.45
cis-1,3,3-Trichloropropene-1	0.13 - 5.52
trans-1,3,3-Trichloropropene-1	0.02 - 0.96

2. Safety

- 2.1 TELONE II soil fumigant is a toxic, strongly odiferous and flammable liquid. It is hazardous if inhaled and toxic if absorbed through the skin. Contact with the eyes and prolonged or repeated contact with skin will cause irritation. Prevent contact with the skin, eyes and clothing. Leather gloves should not be worn when handling samples or standards. Do not breathe the vapors. Work only in a well-ventilated hood away from any source of ignition. Do not store in the presence of strong bases, aluminum, magnesium or their alloys.
- 2.2 Acetone is a flammable solvent. Work in a well-ventilated area away from any source of ignition.
- 2.3 Liquid nitrogen can cause severe frost burns. Leather gloves should be worn while handling or transporting this material. In addition, explosions can result from condensation of air oxygen exposed to liquid nitrogen temperatures, either by way of the reactivity of oxygen or

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by rapid evaporation of oxygen in a closed container. Ensure that cold traps are clean and that they are purged with helium while immersed in liquid nitrogen.

- 2.4 Leather gloves should be worn to prevent thermal burns when handling injection ports and column connections in the gas chromatograph.
- 2.5 Each analyst should be acquainted with potential hazards of the reagents, products, and solvents before commencing laboratory work. SOURCES OF INFORMATION INCLUDE: MATERIAL SAFETY DATA SHEETS, LITERATURE, AND OTHER RELATED DATA. Safety information on non-Dow products should be requested from the supplier. Disposal of reagents, reactants, and solvents must be in compliance with local, state, and federal laws and regulations.

3. Principle

Capillary gas chromatography is used to separate the components from the neat sample, with detection by thermal conductivity detector. Quantitation is by peak area measurement with a computing integrator or chromatography data system using external standard calibration. Analysis precision is controlled by use of an autosampler.

4. Interferences

No direct interferences have been observed in the use of this method other than those indicated in Section 9. If the results are suspect based on the analytical history of the product, the data should be confirmed by an alternate method.

5. Apparatus

- 5.1 Capillary gas chromatograph: Hewlett-Packard 5890A, or equivalent, equipped with thermal conductivity detector and make-up gas and configured for split sample injection, available from Hewlett-Packard, 39550 Orchard Hill Drive, Novi, MI 48050.
- 5.2 Autosampler: Hewlett-Packard 7673A, or equivalent, available from Hewlett-Packard.
- 5.3 Autosampler syringe; 5 μ L: Hamilton 23 gauge, available from Hewlett-Packard.

- 5.4 Autosampler vials: crimp-top, 0.1 μ L, 11 mm x 32 mm; 2 mL, 11 mm x 32 mm, with septa lined with Teflon resin, available from Hewlett-Packard.
- 5.5 Capillary column: 60 m x 0.25 mm fused silica coated with DB-1 (crosslinked methylsilicone) liquid phase, 1- μ m film thickness, or equivalent, available from J & W Scientific Inc., 91 Blue Ravine Road, Folsom, CA 95630.
- 5.6 Computing integrator or chromatography data system: Model 6000, or equivalent, available from Nelson Analytical, Inc., 10061 Bubb Road, Cupertino, CA 95014 (Note 16.1).
- 5.7 Packed column gas chromatograph: Hewlett-Packard 5710A, or equivalent, equipped with thermal conductivity detector, available from Hewlett-Packard.
- 5.8 Preparatory gas chromatographic column: 7' x 1/4" stainless steel, packed with 10% 1,2,3-Tris(2-cyanoethoxy)-propane (TCEP) on Chromosorb P, AW-DMCS, 100/120 mesh. The pre-coated packing is available by special order, with or without preconditioning, from Alltech Associates, 1511 Bingle Road, Houston, TX 77055.
- Alternatively, prepare packing as follows: Dissolve 10 g of TCEP with 500 mL of acetone and pour this mixture onto 90 g of Chromosorb P, AW-DMCS, 100/120 mesh in a large beaker. Mix the contents thoroughly with a glass stirring rod and evaporate the acetone using a rotary evaporator. Condition the packed column overnight at 130°C before connecting to the detector.
- 5.9 U-tube: glass, 3 mm ID, fitted for collection of fractions from the detector outlet of the packed column gas chromatograph.
- 5.10 Syringes: glass, 250 μ L, 100 μ L and 50 μ L, available from Hamilton Company, Reno, NV 89510. The size of the needles should not exceed 22 gauge.
- 5.11 Micro-vials: 1 mL, fitted with septa lined with Teflon, available from Alltech Associates.
- 5.12 Digital density meter: capable of determining density to \pm 0.001 g/mL, Mettler/Paar DMA35G, or equivalent, available from the Fisher Scientific Company, 2400 James Savage Road, Midland, MI 48640.

- 5.13 Oxygen removal system: General Electric Go-Getter, or equivalent, available from the Anspec Company, 122 Enterprise Drive, P.O. Box 2044, Ann Arbor, MI 48107.

6. Reagents

- 6.1 Acetone: HPLC grade, available from the Fisher Scientific Company.
- 6.2 TELONE II soil fumigant: available from the Sample Coordinator, The Dow Chemical Company, Agricultural Products Department, P.O. Box 1706, Midland, MI 48674.
- 6.3 cis-1,3-Dichloropropene-1 Analytical Standard: available from the Sample Coordinator, The Dow Chemical Company.
- 6.4 trans-1,3-Dichloropropene-1 Analytical Standard: available from the Sample Coordinator, The Dow Chemical Company.
- 6.5 2-Chloro-2-methylpentane: available from the Sample Coordinator, The Dow Chemical Company.
- 6.6 2-Chloro-2,3-dimethylbutane: available from the Sample Coordinator, The Dow Chemical Company.
- 6.7 1,3-Dichloropropane: available from the Sample Coordinator, The Dow Chemical Company.
- 6.8 1,2,2-Trichloropropane: available from the Sample Coordinator, The Dow Chemical Company.
- 6.9 cis-1,3,3-Trichloropropene-1: available from the Sample Coordinator, The Dow Chemical Company.
- 6.10 trans-1,3,3-Trichloropropene-1: available from the Sample Coordinator, The Dow Chemical Company.
- 6.11 Helium, high purity (99.999%), cylinder, available from Liquid Carbonics, 135 South LaSalle, Chicago IL 60603.

Reagents 6.5 through 6.10 should be checked for purity under the conditions listed in Section 7.2 and, if interfering compounds are present, adjustment must be made in preparing the standards. Percentage values used in the calculations should be adjusted based on analysis of the individual compounds if they are greater than 90% pure (on an area-percent basis). It is recommended that reagents less than 90% pure not be used.

7. Chromatographic Conditions

7.1 Preparatory gas chromatographic conditions

Instrument: HP 5710A GC/TCD
Column: 7' x 1/4" ss, 10% TCEP on Chromosorb P, AW-DMCS, 100/120 mesh

Temperatures:

Injector: unheated, on column

Detector: 150°C

Oven: 80°C, isothermal

Detector:

Filament: 145 mA

Sensitivity: 4

Flows:

Carrier: 100 mL/min, helium

Recorder: 10 mV

GC Attenuation: 1

7.2 Assay and Impurities gas chromatographic conditions

Instrument:

HP 5890A GC/TCD, HP 7673A

Autosampler, Nelson 6000 Analytical Data System

Column:

60 m x 0.25 mm, DB-1, 1- μ m film

thickness, fused silica capillary

Temperatures:

Injector: 150°C

Detector: 155°C

Oven: 50°C, 3 min hold

50°C - 3°C/min - 110°C

110°C, 5 min hold

Detector:

TCD

Sensitivity: High

Flows:

Carrier: 1.9 mL/min, helium (measured at
oven temperature of 50°C)Split: 72.2 mL/min, helium (measured at
oven temperature of 50°C)

Make-up: 3.2 mL/min, helium

Reference: 16.1 mL/min, helium

Septum purge: 1.2 mL/min

Sample Size: 0.5 μ L

GC Range: 1

Ambient temperature: constant \pm 2°F (Note 16.2)

An efficient oxygen scavenging system such as the General Electric Go-Getter should be used on the carrier gas.

See Figure 1 for a typical chromatogram.

8. Standard Preparations

PURE ISOMER MIXTURE

(cis-1,3-dichloropropene-1 and
trans-1,3-dichloropropene-1)

- 8.1 Install the preparatory gas chromatographic column in the packed-column gas chromatograph. Install a U-tube immersed in liquid nitrogen at the detector outlet of the gas chromatograph. Ensure that helium is flowing through the U-tube prior to immersing in liquid nitrogen to preclude condensation of moisture in the trap (WARNING: Do not insert the U-tube into liquid nitrogen without removing caps, if any, from the U-tube. Failure to observe this precaution may lead to an explosion due to boiling of condensed gases upon removal of the U-tube from the liquid nitrogen).
- 8.2 Inject a 50- μ L aliquot of TELONE II soil fumigant under the preparatory gas chromatographic conditions (7.1), collecting the pure isomer mixture as it elutes from the detector. The cis- and trans-1,3-dichloropropene-1 elute at about 10 and 15 min. After 5 injections, transfer the trap contents to a 1 mL micro-vial fitted with a Teflon-lined septum cap, allowing the trap to come to ambient temperature before exposing the mixture to air. Repeat this procedure until at least 600 mg of pure isomer mixture has been collected. Store the collected pure isomer mixture in a freezer set at -10°C and discard or reanalyze after two weeks (Note 16.3).
- 8.3 Analyze 0.5 μ L of the pure isomer mixture under the chromatographic conditions listed in Section 7.2.
- 8.4 The area ratio (cis/trans) should be $50 \pm 1/45 \pm 1$. If the area ratio is outside of acceptable limits, it can be adjusted by addition of pure cis- or trans-1,3-dichloropropene-1, which may be prepared by injecting 50- μ L aliquots of the appropriate cis- or trans-1,3-dichloropropene-1 Analytical Standards and proceeding as described for the preparation of the pure isomer mixture. The individual pure isomers should be stored as described in 16.3.

- 8.5 No peaks should appear in the chromatogram within the retention time identification windows for the six low level components determined by this method which would result in reported concentrations of greater than 0.05%.

COMPONENT STOCK SOLUTION

- 8.6 Using a 250- μ L syringe, accurately weigh to the nearest 0.1 mg approximately 240 mg of cis-1,3,3,-trichloropropene-1 (6.9) into a 2-mL autosampler vial which has been previously fitted with a septum cap lined with Teflon resin and tared, ensuring that all of the material is in the bottom of the vial. Using a 50- μ L syringe, add and accurately weigh to the nearest 0.1 mg approximately 40 mg of the reagents in 6.5, 6.6 and 6.10, and approximately 60 mg of reagents in 6.7 and 6.8. Mix the contents by inverting the vial several times and then by sonication for 1 minute. Do not allow the vial to become warm! Store the stock solution in a freezer set at -10°C except while it is being used, and discard after two weeks (Note 16.3). Calculate the actual weight in mg of each component in the component stock solution, correcting for reagent purity as described in Section 6.

COMPONENT DILUTED SOLUTION

- 8.7 Using a 250- μ L syringe, accurately weigh to the nearest 0.1 mg, approximately 130 mg of the pure isomer mixture (8.2) into a 1-mL micro-vial which has been fitted with a septum cap lined with Teflon resin and tared. Using a 50- μ L syringe, add and accurately weigh to the nearest 0.1 mg approximately 18 mg of the component stock solution (8.6). Mix the contents as described above (8.6). Store the diluted solution in a freezer set at -10°C except while it is being used, and discard after two weeks (Note 16.3).

PRIMARY ASSAY STANDARDS

- 8.8 Using a 50- μ L syringe, accurately weigh to the nearest 0.1 mg approximately 50 mg of cis-1,3-dichloropropene-1 Analytical Standard and 45 mg of trans-1,3-dichloropropene-1 Analytical Standard into each of two 0.1 mL autosampler vials which have been previously capped with septum caps lined with Teflon resin and tared. Mix the solutions well by inverting each vial several times and then by sonication for 1 minute. Do not allow the vials to become warm! Store the primary assay standards in a freezer set at -10°C except while they are being used, and discard after two weeks (Note 16.3).

- 8.9 Calculate the percent weight of the isomers in each primary assay standard as follows:

$$\% \text{ cis-1,3-Dichloropropene-1} = \frac{[(A \times B) + (C \times D)]}{B + D}$$

where: A = % cis-1,3-Dichloropropene-1 in the cis-1,3-Dichloropropene-1 Analytical Standard
 B = weight in mg of cis-1,3-Dichloropropene-1 Analytical Standard
 C = % cis-1,3-dichloropropene-1 in the trans-1,3-Dichloropropene-1 Analytical Standard
 D = weight in mg of trans-1,3-Dichloropropene-1 Analytical Standard

$$\% \text{ trans-1,3-Dichloropropene-1} = \frac{[(E \times B) + (F \times D)]}{B + D}$$

where: E = % trans-1,3-Dichloropropene-1 in the cis-1,3-Dichloropropene-1 Analytical Standard
 B = weight in mg of cis-1,3-Dichloropropene-1 Analytical Standard
 F = % trans-1,3-Dichloropropene-1 in the trans-1,3-Dichloropropene-1 Analytical Standard
 D = weight in mg of trans-1,3-Dichloropropene-1 Analytical Standard

- 8.10 Using the digital density meter, measure the density, mg/ μ L (g/mL), of the primary assay standard (Note 16.2).

PRIMARY COMPONENT STANDARD

- 8.11 Using a 50- μ L syringe, accurately weigh to the nearest 0.1 mg approximately 40 mg of the pure isomer mixture (8.2) and 10 mg of component diluted solution (8.7) into a 0.1 mL autosampler vial which has been previously capped with a septum cap lined with Teflon resin and tared. Mix the solution well by inverting the vial several times and then by sonication for 1 minute. Do not allow the vial to become warm! Store the primary component standard in a freezer set at -10°C except while it is being used, and discard after two weeks (Note 16.3). Calculate the percent weight to the nearest 0.001% of each low level component in the primary component standard, correcting for any low level components present in the pure isomer mixture as follows:

$$\% \text{ Component} = \frac{G \times I \times K}{H \times J} + \frac{(L \times M)}{100} \times \frac{100}{N}$$

where: G = weight of component in component stock
solution, corrected for purity (8.6)
H = total weight of component stock solution
I = weight of component stock solution in
component diluted solution (8.7)
J = total weight of component diluted solution
K = weight of component diluted solution in primary
component standard
L = % weight of component in pure isomer mixture
M = weight of pure isomer mixture in primary
component standard
N = total weight of primary component standard

SECONDARY STANDARD

Select a sample of TELONE II soil fumigant which has been shown to conform to all product specifications for use as a secondary standard. It is recommended that at least 500 mL of the sample be available to minimize the need for frequent preparation of primary standards. Certify the sample as a secondary standard as follows:

- 8.12 Obtain response factors for the cis- and trans-1,3-Dichloropropene-1 isomers from either primary assay standard as described in Section 10.
- 8.13 Analyze the second primary assay standard as a sample as described in Section 11, based on the calibration factors obtained above. The values obtained for percent of cis- and trans-1,3-Dichloropropene-1 must agree with those calculated for the second primary assay standard in 8.9 within the precision of the method ($\pm 1.0\%$). If satisfactory results are not obtained, additional primary assay standard(s) must be prepared until results are obtained which are within the required precision.
- 8.14 Obtain response factors for the low level components from the primary component standard as described in Section 10.
- 8.15 Using the digital density meter, measure the density, mg/ μ L (g/mL), of the sample of TELONE II soil fumigant (Note 16.4).
- 8.16 Analyze the sample of TELONE II soil fumigant five times versus the primary assay standard and five times versus the primary component standard as described in Section 11 to accurately determine the levels of cis-1,3-dichloropropene-1, trans-1,3-dichloropropene-1, and the low level components in the sample. Calculate the level of each component in the secondary standard (TELONE II soil fumigant) as described in Section 12.

- 8.17 The results obtained for each component from the five analyses of TELONE II soil fumigant must agree within the precision of the method (Section 13) in order for the sample to be certified as a secondary standard.
- 8.18 Store the secondary standard bulk sample in a freezer set at -10°C except while it is being used. Discard individual vials after they have been used. The bulk secondary standard is stable for approximately 1 year at -10°C but should be reanalyzed against primary assay and component standards at least once yearly (Note 16.3).

9. System Suitability Test

A system suitability test should be performed for a new chromatographic system or a new column, at least monthly thereafter, and whenever system performance is suspect. The test can be combined with system calibration to minimize time requirements.

- 9.1 Record a stable baseline with the chromatograph operating under the initial conditions described in 7.2.
- 9.2 Without injecting a sample, run a temperature program as described in Section 7.2 and record the background. A satisfactory background will show no peaks within the retention identification windows for the components determined by this method which would result in reported concentrations of greater than 0.02%.
- 9.3 Inject five 0.5- μL aliquots of the secondary standard and analyze each according to the chromatographic conditions stated in Section 7.2. A recorder chart speed of at least 5 cm/min should be used for the portion of the chromatogram including 2-chloro-2,3-dimethylbutane.
- 9.4 After analysis is complete, verify that the 2-chloro-2,3-dimethylbutane is sufficiently resolved from the peak which elutes about 0.05 min later, that the integrator or data system has detected a valley between them, and that the correct peak has been identified as 2-chloro-2,3-dimethylbutane for all five chromatograms.
- 9.5 From the same five chromatograms, verify that the height (above baseline) of the minimum between the trans-1,3-dichloropropene-1 and 2-chloro-2-methylpentane peaks does not exceed 10% of the height above baseline of the 2-chloro-2-methylpentane peak.

- 9.6 For the five chromatograms obtained, verify that the peak for 6-chlorohexene-1 is sufficiently resolved from the tail of the cis-1,3-dichloropropene-1 peak and that the integrator or data system has consistently detected the valley between them for all five chromatograms.
- 9.7 For the five chromatograms obtained, verify that satisfactory precision is obtained for all components (Section 13).
- 9.8 If the chromatographic system does not pass the suitability test, minor adjustments may be made in gas flow rates and/or column temperatures. If satisfactory performance is not obtained, another column should be used.

10. Calibration

- 10.1 Using a disposable pipette, dispense approximately 1 mL of the secondary standard into a 2-mL autosampler vial and cap with a septum cap lined with Teflon resin (Note 16.3).
- 10.2 Inject a 0.5- μ L aliquot of the secondary standard of TELONE II soil fumigant (8.17) and analyze according to the chromatographic conditions stated in Section 7.2. See Figure 1 for a typical chromatogram of a sample of TELONE II soil fumigant (Note 16.5).
- 10.3 If manual calculations are used, calculate the response factor, RF, for each component as follows (Note 16.2):

$$RF = \frac{O \times P \times Q}{R}$$

where: O = area of component peak in chromatogram of standard

P = density of standard, mg/ μ L (Note 16.4)

Q = volume of standard injected (0.5 μ L)

R = percent weight of component in the standard

- 10.4 Initial calibration should be performed by duplicate analysis of the secondary standard, with response factors for the cis- and trans-1,3-dichloropropene-1 isomers agreeing within 0.8% relative and with response factors for the low level components agreeing within 5% relative. Subsequent calibration can be made with a single analysis of the standard as long as response factors do not deviate more than these limits.

11. Procedure

- 11.1 Using the digital density meter, measure the density, mg/ μ L (g/mL), of the sample of TELONE II soil fumigant (Note 16.4).
- 11.2 Using a disposable pipette, dispense approximately 1 mL of the sample into a 2-mL autosampler and cap with a septum cap lined with Teflon resin (Note 16.3).

11.3 Inject a 0.5- μ L aliquot of the sample and analyze according to the chromatographic conditions stated in Section 7.2. See Figure 1 for a typical chromatogram of a sample of TELONE II soil fumigant (Note 16.5).

12. Calculations

If manual calculations are used, calculate the percent weight of each component as follows (Note 16.2):

$$\% \text{ Component} = \frac{S \times T \times U}{V}$$

where: S = area of component peak in chromatogram of sample

T = density of sample, mg/ μ L (Note 16.4)

U = volume of sample injected (0.5 μ L)

V = response factor of component (10.3)

13. Precision

Data obtained for the method by ten replicate analyses of a sample of TELONE II soil fumigant and by multiple analyses of six samples of TELONE II soil fumigant indicate the relative standard deviations shown below. For each component, values represent the case of greater variability (Note 16.6). The values may be expected to vary from the average by not more than the relative precision at the 95% confidence level (2.26 x relative standard deviation based on the ten replicate analyses or 2.13 x relative standard deviation based on the analyses of the six samples). These precision values were obtained using the chromatographic system described in Section 5 with automated sample injection. The precision performance must be experimentally redetermined for other instruments.

<u>Component</u>	<u>Concentration</u> ¹	<u>Relative Standard Deviation (RSD)</u>	<u>Relative Precision</u>
cis-1,3-Dichloropropene-1	49.51 - 50.50%	0.48%	1.0%
trans-1,3-Dichloro- propene-1	44.94 - 45.88 0.49	1.0	
2-Chloro-2-methylpentane	0.126- 0.190	6.6	14.0
2-Chloro-2,3-dimethyl- butane	0.086 1.7	3.8	
1,3-Dichloropentane	0.151 1.0	2.3	
1,2,2-Trichloropropane	0.078 - 0.171	5.0	10.6
cis-1,3,3-Trichloro- propene-1	0.930 0.6	1.4	
trans-1,3,3-Trichloro- propene-1	0.160 - 0.259 1.3	2.8	

¹Concentration at which precision was determined. Ranges indicate data based on analyses of six samples.

14. Recovery

Analysis of a series of twenty-four synthetic mixtures for the cis- and trans-1,3-dichloropropene-1 and of ten synthetic mixtures for the low level components gave the following recoveries and precisions:

<u>Component</u>	<u>Concentration Range (wt%)</u>	<u>Recovery Range (%)</u>	<u>Average Recovery</u>	<u>RSD</u>
cis-1,3-Dichloro- propene-1	19.45 - 52.59 70.48 - 93.20 0.80	99.3-100.4 97.3-102.0 108	99.9% 99.9 ----	0.40 2.4 ----
trans-1,3-Dichloro- propene-1	42.74 - 94.20 2.40 - 24.98	99.0-100.8 98.1-102.1	99.9 100.3	0.57 2.0
2-Chloro-2-methyl- pentane	0.02-0.92	98-105	101	2.0
2-Chloro-2,3-dimethyl- butane	0.05-0.91 0.02	99-106 124	101 ----	1.9 ----
1,3-Dichloropropane	0.03-1.50	92-106	98	4.3
1,2,2-Trichloro- propane	0.07-1.45 0.03	100-105 121	102 ----	1.7 ----
cis-1,3,3-Trichloro- propene-1	0.28-5.52 0.13	100-103 110	101 ----	1.0 ----
trans-1,3,3-Trichloro- propene-1	0.10-0.96 0.02-0.05	101-105 112-116	103 114	1.2 2.5

15. Linearity

Detector response was found to be linear for the components determined by this method throughout the concentration ranges shown in Section 1 with the exception of 2-chloro-2,3-dimethyl-butane below 0.05%, 1,2,2-trichloropropane below 0.07% and cis-1,3,3-trichloropentene-1 below 0.28%. The method is applicable at these levels since absolute error will be minimal.

16. Notes

- 16.1 The computing integrator must have the capability of identifying peaks in a time window of ± 5 sec.
- 16.2 The accuracy of an external standard method is dependent on injection of a consistent amount of sample. The variation in the amount of sample injected is a function of the volume injected and the density of the sample. The volume of sample injected is constant, within the precision of injection. The density will be constant only if sample temperature does not vary greatly. Based on comparison with ethylene dichloride and 1,1,2-trichloro-ethylene, the density of 1,3-dichloropropene-1 solutions would be expected to vary approximately $-0.2\%/^{\circ}\text{F}$. Therefore, variation greater than $\pm 2^{\circ}\text{F}$ in laboratory ambient temperatures should be avoided.
- 16.3 The dichloropropenes are strong solvents which will swell caps made of SARAN* or polyethylene resins, as well as react with aluminum foil-lined caps. The primary standards and the TELONE II soil fumigant secondary standard and samples are stable for several hours in vials with caps lined with Teflon resin at room temperature, however for long-term storage a freezer at -10°C is required to prevent volatilization, decomposition or trans-isomerization of the components.
- 16.4 The densities of six samples of TELONE II soil fumigant, obtained using a Paar Digital Density Meter DMA10 (Anton Paar K. G., Graz, Austria), were found to vary from 1.2168 to 1.2172 g/mL (mg/ μL).
- 16.5 Components identified by letters in Figure 1 follow. None are present at the 0.1% level:

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<u>Letter</u>	<u>Compound</u>
A	Air
B	Acetone (syringe rinse solvent)
C	1,2-Dichloropropane
D	6-Chlorohexene-1
E	C ₆ H ₁₃ Cl (two isomers)
F	C ₄ H ₆ Cl ₂
G	C ₆ H ₁₃ Cl
H	C ₆ H ₉ Cl and C ₆ H ₁₁ Cl
I	C ₆ H ₁₁ Cl and C ₆ H ₉ Cl
J	C ₆ H ₁₁ Cl
K	C ₆ H ₁₁ Cl
L	Chlorobenzene
M	C ₃ H ₃ Cl ₃
N	C ₃ H ₅ Cl ₃

16.6 The precision data showing the greatest variability were chosen to account for sample to sample variability and variation in laboratory conditions.

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Figure 1. Assay and Low Level Components in TELONE II Soil Fumigant by Capillary Gas Chromatography: Chromatogram of Precision Sample.

