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ANALYTICAL METHOD AND VALIDATION FOR THE DETERMINATION OF 1,2-DICHLOROPROPANE IN TELONE II AND TELONE EC FORMULATIONS

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Enforcement Method?

1,2-DICHLOROPROPANE

CHEMICAL

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Method Technique(s)

GC/FID

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SUMMARY

(In accordance with 40 CFR Part 152, this summary is available
for public release after registration)

STUDY TITLE

Analytical Method and Validation for the Determination of 1,2-Dichloropropane in Telone II
and Telone EC Formulations

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham

STUDY COMPLETED ON

June 28, 2005

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LABORATORY STUDY ID

DAS-AM-05-008

SUMMARY

This report describes the validation of an analytical method for determination of 1,2-dichloropropane in Telone II and Telone EC formulations. A gas chromatographic method was validated using a DB-1701 column with flame ionization detection (FID) and external standard quantitation.

The method is valid over a range of 32 ppm to 163 ppm 1,2-dichloropropane in Telone II and 32 ppm to 165 ppm 1,2-dichloropropane in EF-1478 (Telone EC). The average recovery for 1,2-dichloropropane in Telone II was 102%, with a relative standard deviation of 3.5%. The average recovery for 1,2-dichloropropane in Telone EC was 104%, with a relative standard deviation of 5.0%. Detector response was shown to be linear for 1,2-dichloropropane over a range of 30 ppm to 166 ppm, which is which is equivalent to 0.33 to 1.66 times the maximum concentration allowable by the current product specification for Telone II and EF-1478 (Telone EC).

Replicate analyses of Telone II and Telone EC formulations on two separate days gave a relative standard deviation of 2.07% at an average concentration of 81 ppm (wt/wt) 1,2-dichloropropane for Telone II and a relative standard deviation of 1.08% at an average concentration of 144 ppm for Telone EC. The analysis is complete in 66.3 minutes.

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STUDY DIRECTOR

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STUDY COMPLETED ON

June 28, 2005

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LABORATORY STUDY ID

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STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claims of confidentiality are being made for any information contained within this study on the basis of its falling within the scope of FIFRA Section 10(d)(1)(A), (B), or (C).*

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Company Agent: Bruce Houtman

Title: Regulatory Manager

Signature:

Bruce Houtman, cap

Date:

6/3/05

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STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Study Initiation Date: April 20, 2005 Experimental Start Date: April 20, 2005

Experimental End Date: June 1, 2005

All phases of this study were conducted according to the following Good Laboratory Practice Standard:

United States Environmental Protection Agency
Title 40 Code of Federal Regulations Part 160
FEDERAL REGISTER, August 17, 1989

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6-28-05

Study Completion Date

Dow AgroSciences Quality Assurance Unit
Good Laboratory Practice Statement Page

Compound: 1, 2-Dichloropropane

Study ID: DAS-AM-05-008

Title: Analytical Method and Validation for the Determination of 1,2-Dichloropropane in
Telone II and Telone EC Formulations

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19-Apr-2005	20-Apr-2005	Protocol Review
26-Apr-2005	27-Apr-2005	Preparation of Recovery Samples for Analysis
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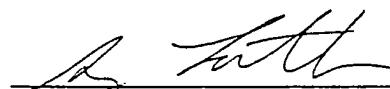
The Quality Assurance Unit has reviewed the final study report and has determined that the report reflects the raw data generated during the conduct of this study.

Lynn M Kennard
Lynn Kennard
Dow AgroSciences, Quality Assurance

13 - July - 2005
Date

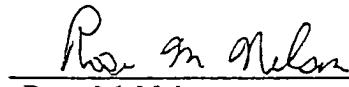
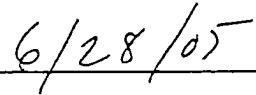
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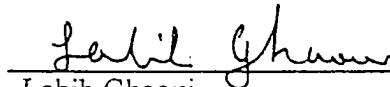
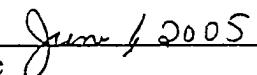
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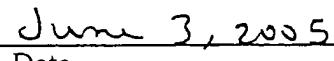


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I. ABSTRACT

This report describes the validation of an analytical method for determination of 1,2-dichloropropane in Telone II and Telone EC formulations. A gas chromatographic method was validated using a DB-1701 column with flame ionization detection (FID) and external standard quantitation.

The method is valid over a range of 32 ppm to 163 ppm 1,2-dichloropropane in Telone II and 32 ppm to 165 ppm 1,2-dichloropropane in EF-1478 (Telone EC). The average recovery for 1,2-dichloropropane in Telone II was 102%, with a relative standard deviation of 3.5%. The average recovery for 1,2-dichloropropane in Telone EC was 104%, with a relative standard deviation of 5.0%. Detector response was shown to be linear for 1,2-dichloropropane over a range of 30 ppm to 166 ppm, which is which is equivalent to 0.33 to 1.66 times the maximum concentration allowable by the current product specification for Telone II and EF-1478 (Telone EC).

Replicate analyses of Telone II and Telone EC formulations on two separate days gave a relative standard deviation of 2.07% at an average concentration of 81 ppm (wt/wt) 1,2-dichloropropane for Telone II and a relative standard deviation of 1.08% at an average concentration of 144 ppm for Telone EC. The analysis is complete in 66.3 minutes.

II. INTRODUCTION

A. Scope

This GC method is applicable to the determination of 1,2-dichloropropane (1,2-DCP) in Telone II and Telone EC formulations, which both have a maximum allowable specification limit of 100 ppm 1,2-DCP. The method was validated over the range of 32 ppm to 163 ppm 1,2-DCP in Telone II and 32 ppm to 165 ppm in Telone EC (EF-1478).

B. Principle

An aliquot of ethyl acetate is added to the sample to dilute the concentration of the mixture. The solution is analyzed by gas chromatography using a J & W Scientific DB-1701 column with FID detection. Quantitation is by external standard calculation using peak areas.

III. MATERIALS AND METHODS

A. Equipment

1. Analytical balance, capable of measuring to 0.1 mg, Mettler AE260, or equivalent.
2. Gas chromatograph (GC) equipped with a flame ionization detector and split/splitless injector.
3. Data acquisition and processing system: Hewlett Packard ChemServer, or equivalent.
4. J & W Scientific DB-1701, 60 m x 0.32 mm x 1 μ m
5. Autosampler vials and caps: 1.5 mL with screw caps
6. Miscellaneous laboratory glassware.

B. Reagents and Standards:

1. Test and Reference Substance: 1,2-dichloropropane Reference Standard, AGR277102, 99%, recertification date October 2, 2007.
2. Ethyl acetate: Mallinckrodt ChromAR HPLC Grade, or equivalent.
3. Test systems:
 - Telone II Formulation blank: TSN105022
 - Telone EC Formulation blank: E1912-48
 - Telone II formulation: TSN105121
 - Telone EC formulation: E1912-46

C. Safety

Each analyst should be acquainted with potential hazards of the reagents, products and solvents before beginning laboratory work. Sources of information include: material safety data sheets, literature and other related data. Disposal of reagents, reactants, and solvents must be in compliance with local, state and federal laws and regulations.

D. Analytical Procedures

1. Preparation of calibration solution:

Prepare stock calibration solutions by weighing ~ 50 mg of 1,2-DCP reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate. Transfer 1 mL of this stock solution into a 20 mL volumetric flask and fill to the mark with ethyl acetate to afford a working standard in the range of 40 ppm 1,2-DCP.

2. Calibration procedure:

Inject the calibration solution at least twice into a liquid chromatograph, using the conditions summarized in Section III.E, and calculate the response factor for 1,2-DCP using the equation given in Section III.F. The average of the response factors is used for calibration. A typical chromatogram of the calibration solution is shown in Figure 1.

3. Sample preparation and analysis:

Add 2 mL (~ 2.4 g) of formulation into an appropriate sized jar using a volumetric pipette and record the weight. Add by volumetric pipette 3 mL of ethyl acetate. Analyze using the conditions given in Section III.E.

A typical chromatogram of a prepared Telone II formulation sample solution is shown in Figure 2. A typical chromatogram of a prepared Telone EC formulation sample solution is shown in Figure 3.

4. Preparation of recovery samples:

A stock spiking solution was prepared by adding 25.6 mg 1,2-DCP to 25 mL Telone II to afford a 833 ppm (wt/wt) solution of 1,2-DCP in Telone II. An additional spiking solution was prepared by adding 25.8 mg 1,2-DCP to 25 mL Telone EC to afford a 840 ppm (wt/wt) solution.

Formulation samples containing 1,2-DCP were prepared for Telone II by weighing aliquots of the Telone II (TSN105022) into 20 mL scintillation vials (Table I). Aliquots of 1,2-DCP were then added to each sample via the stock solution of 1,2-DCP in Telone II, and contents were prepared for GC analysis.

Formulation samples containing 1,2-DCP were prepared for Telone EC by weighing aliquots of the Telone EC (E1912-48) into 20 mL scintillation vials (Table II). Aliquots of 1,2-DCP were then added to each sample via the stock solution of 1,2-DCP in Telone EC, and contents were prepared for GC analysis.

5. Preparation of linearity solutions:

The recovery samples were used to evaluate linearity of the method.

6. Preparation of precision samples:

The precision samples were prepared by accurately weighing 2 mL aliquots of Telone II and Telone EC formulations (fortified with 1,2-DCP to ensure formulations were in the appropriate range for analysis) into 20 mL scintillation vials, followed by 3 mL of ethyl acetate using a volumetric pipette. This procedure was followed five times on each of two days.

E. Instrumentation

1. Description:

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

2. Approximate time to prepare and analyze sample: 2 hours

F. Methods of Calculation

Calculation of response factors and weight percent values can be performed with a computing integrator/data system or with a spreadsheet.

1. Calculation of the response factor for 1,2-DCP in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(1,2\text{-dichloropropane})} \times 50 \text{ mL}} \times \frac{1}{20 \text{ mL}}$$

where: RF = Response factor for 1,2-DCP
mg reference std = Weight of 1,2-DCP reference standard in calibration solution, mg
P = Purity of reference standard, expressed as a fraction
Area (1,2-DICHLOROPROPANE) = Peak area for 1,2-DCP in calibration solution

2. Calculation of the weight % of 1,2-DCP in the sample:

$$\text{Weight \%} = \frac{\text{Area}(1,2\text{-dichloropropane}) \times \text{RF}}{\text{Sample wt}} \times \text{DF} \times 100$$

where: Weight % = Weight % of 1,2-DCP in the sample
Area_(1,2-DICHLOROPROPANE) = Peak area for 1,2-DCP in the sample solution
RF = Response factor calculated for 1,2-DCP
Sample wt = Weight of sample in mg
DF = Dilution factor (5 mL for a sample prepared as described)

3. Calculation of the parts per million (ppm) of 1,2-DCP in the sample:

$$\text{ppm} = \text{Weight \%} \times 10000$$

IV. RESULTS AND DISCUSSION

A. Linearity

The linearity for 1,2-DCP was evaluated using the GC conditions used for this study. A linear relationship between peak area and concentration ($r^2 = 0.9983$) was noted for 1,2-DCP from 31 ppm to 165 ppm. The linearity plot is shown in Figure 4.

B. Accuracy

The accuracy of the method was evaluated by analysis of a series of samples prepared as described in Section III.D.4. The preparation of the recovery samples is given in Tables I and II. Samples were analyzed using the calibration solution described in Section III.D.1. The blank formulations contained a small amount of 1,2-DCP prior to spiking so the blanks were analyzed without fortification and the amount present determined (24 ppm in Telone II and 23 ppm in Telone EC). The amount present prior to fortification was then added to the theoretical amount expected when calculating the recovery values. Recovery data were obtained over the range of 32 ppm to 163 ppm 1,2-DCP in Telone II and 32 ppm to 165 ppm 1,2-DCP in Telone EC. The average recovery for 1,2-DCP in Telone II was 102 % and in Telone EC was 104 %.

The recovery for 1,2-DCP in Telone II ranged from 94 % to 105 %, with an average recovery of 102%, and a relative standard deviation of 3.5 %. The recovery for 1,2-DCP in Telone EC ranged from 94 % to 108 %, with an average recovery of 104 %, and a relative standard deviation of 5.0 %. Recovery data are shown in Tables III and IV. The recovery values shown were calculated using an Excel spreadsheet. Due to rounding, minor differences may occur between percent recovery stated in Tables III and IV and numbers obtained if the values are calculated by hand.

C. Method Precision

The precision of the method was evaluated by analysis of Telone II and Telone EC formulations, with five samples prepared and analyzed on each of two days. A fresh standard solution was prepared each day and used for calibration. The precision data are shown in Table V.

The relative standard deviation was 2.1 % at an average concentration of 81 ppm (wt/wt) 1,2-DCP in Telone II and 1.08 % at an average concentration of 144 ppm (wt/wt) in Telone EC. The Horwitz RSD_r value was calculated to be 5.53 for Telone II and 5.07 for Telone EC; therefore, results are acceptable (Table V).

D. System Precision

System precision was determined by injecting a prepared solution of Telone II and Telone EC formulation five times. Data obtained are shown in Table VI. The relative standard deviation for the peak area 1,2-DCP between all five injections of Telone II was 1.14% and 2.65% for Telone EC.

E. Solution Stability

The solution stability was determined by analyzing sample solutions prepared for the day two precision study 8 days after the initial analysis. Fresh standard solutions were prepared. The t-test was used to compare the results. The t-test results indicated that the results obtained eight days after initial analysis were equivalent to the original results for Telone II and Telone EC.

F. Interferences

No interferences were detected for the ethyl acetate solvent, formulation inert ingredients, or 1,2-DCP. Chromatograms of a solvent blank, 1,2-DCP and formulation blank for Telone II and Telone EC, respectively, are shown in Figure 5.

G. Ruggedness

The method ruggedness was tested by reducing the flow rate from the nominal 2.2 mL/min to 1.5 mL/min. The retention times of the component of interest changed significantly, as shown in Figure 6. No interferences were observed for any of the components.

H. Limit of Quantitation (LOQ) and Limit of Detection (LOD)

Limit of quantitation was experimentally demonstrated by preparing five samples at approximately 40 ppm (wt/wt) following the same preparation of calibration solutions and samples as those in section III.D and analyzing using the conditions in section III.E. Data are shown in Table VI.

Limit of detection was determined by reviewing the recovery samples prepared at 8 ppm and determining that the 1,2-DCP had a greater than three times signal to noise. The 8 ppm samples also had 24 ppm already present prior to fortification so the limit of detection is the sum of the two, 32 ppm. See Figure 7 for representative chromatogram of the LOD samples.

V. CONCLUSIONS

This method is applicable to the determination of 1,2-DCP in Telone II and Telone EC formulations over the range of 32 ppm to 163 ppm 1,2-DCP by weight in Telone II and 32 ppm to 165 ppm Telone EC. The precision, recovery and linearity data have shown this method to be acceptable for the assay of 1,2-DCP in Telone II and Telone EC formulations. In accordance with good laboratory practices, it is suggested that the precision and linearity of the method be re-determined if another set of equipment is used. This report satisfies the data requirement for U.S. EPA OPPTS Guideline 830.1800, Enforcement Analytical Method.

The statistical methods used were means, standard deviations, relative standard deviations, regression analysis, Horowitz equation and the t-test. The databooks, raw data and the original copy of the final report for this study will be stored in the Dow AgroSciences LLC test facility archives at the 306 Building, 9330 Zionsville Road, Indianapolis, Indiana.

VI. TABLES

Table I. Preparation of Recovery Samples for Telone II Technical Formulation

Sample	Weight 1,2-DCP added (AGR277102), mg	Weight of Telone II (TSN105022) sample plus weight of spike, mg	Volume of spike solution added (uL)	1,2-DCP ppm*	1,2-DCP in blank (ppm)
Recovery 1	0.406	2899.6	400	140	24
Recovery 2	0.304	2780.6	300	109	24
Recovery 3	0.203	2654.5	200	76	24
Recovery 4	0.162	2617.8	160	62	24
Recovery 5	0.101	2542.5	100	40	24
Recovery 6	0.041	2468.1	40	16	24
Recovery 7	0.020	2439.4	20	8	24

Table II. Preparation of Recovery Samples for Telone EC Formulation

Sample	Weight 1,2-DCP added (AGR277102), mg	Weight of Telone EC (E1912-48) sample plus weight of spike, mg	Volume of spike solution added	1,2-DCP ppm*	1,2-DCP in blank (ppm)
Recovery 1B	0.406	2883.9	400	141	24
Recovery 2B	0.304	2765.4	300	110	24
Recovery 3B	0.203	2646.3	200	77	24
Recovery 4B	0.162	2593.7	160	63	24
Recovery 5B	0.101	2531.5	100	40	24
Recovery 6B	0.041	2452.4	40	17	24
Recovery 7B	0.020	2428.7	20	8	24

Weight of 1,2-DCP added to sample =

$$\frac{1,2 - \text{DCP wt} \times 1000 \times \text{vol spike solution used (mL)} \times \text{density aliquot (1.217 g/mL)}}{\text{vol of spike stock solution (mL)} \times \text{density of spike solution (1.217 g/mL)}}$$

where: 1,2-DCP wt = Wt. 1,2 - DCP in spike solution, mg x purity 1,2 - DCP

*The parts per million (ppm) 1,2-DCP in the recovery samples was calculated as follows:

$$\text{ppm} = \frac{\text{Wt. 1,2 - DCP added from spike solution, mg}}{\text{Wt. Telone II or EC technical, mg}} \times 100\% \times 10,000$$

Table III. Recovery Data for 1,2-DCP in Telone II Formulation

Sample	1,2-DCP ppm added*	1,2-DCP ppm found	Recovery %
Recovery 1	163	166	102
Recovery 2	133	136	102
Recovery 3	100	103	103
Recovery 4	85	88	103
Recovery 5	63	65	102
Recovery 6	40	42	105
Recovery 7	32	30	94
Average			102
Std. Dev.			3.56
R.S.D.			3.5

* Amount added plus amount present in blank

Table IV. Recovery Data for 1,2-DCP in Telone EC Formulation

Sample	1,2-DCP ppm added*	1,2-DCP ppm found	Recovery %
Recovery 1b	165	179	106
Recovery 2b	134	142	106
Recovery 3b	100	108	110
Recovery 4b	86	91	109
Recovery 5b	63	66	109
Recovery 6b	40	39	104
Recovery 7b	32	30	94
Average			105
Std. Dev.			5.30
R.S.D.			5.0

* Amount added plus amount present in blank

Table V. Method Precision Data for 1,2-DCP in Telone II and Telone EC Formulations

Sample	Telone II ppm (wt/wt)	Telone EC ppm (wt/wt)
Day 1 #1	80	142
Day 1 #2	80	145
Day 1 #3	79	143
Day 1 #4	80	145
Day 1 #5	78	141
** Day 2 #1	82	144
Day 2 #2	84	145
Day 2 #3	81	146
Day 2 #4	80	143
Day 2 #5	81	144
Average	81	144
Std. Dev.	1.67	1.55
*R.S.D.	2.07	1.08
*Horwitz RSD _R	8.26	7.57
*Horwitz RSD _r	5.53	5.07
RSD < Horwitz RSD _r ?	Yes; results are acceptable	Yes; results are acceptable

* Based on weight percent

** Only the first injection of two were used to remain consistent with Day 1 precision

Table VI. System Precision Data for 1,2-DCP

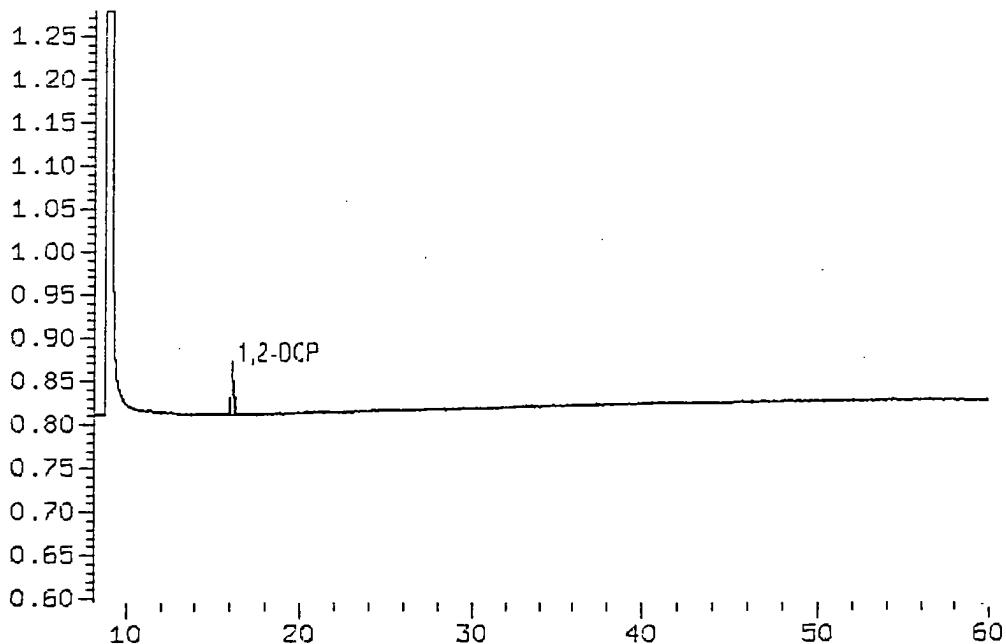
Sample	Peak Area
Telone EC	
Injection #1	62778
Injection #2	62673
Injection #3	62200
Injection #4	63505
Injection #5	64015
Average	63034
Std. Dev.	720.35
R.S.D. (%)	1.14
Telone II	
Injection #1	34790
Injection #2	33690
Injection #3	35704
Injection #4	34387
Injection #5	33422
Average	34399
Std. Dev.	910.12
R.S.D. (%)	2.65

Table VII. LOQ Data for 1,2-DCP

Sample	Peak Area
Telone II	
LOQ-1	17608
LOQ-2	17429
LOQ-3	17565
LOQ-4	16743
LOQ-5	17362
Average	0.00403
Std. Dev.	0.000081
R.S.D. (%)	2.00
Telone EC	
LOQ-1	16903
LOQ-2	17184
LOQ-3	16043
LOQ-4	18020
LOQ-5	16661
Average	0.00390
Std. Dev.	0.00017
R.S.D. (%)	4.27

VII. FIGURES

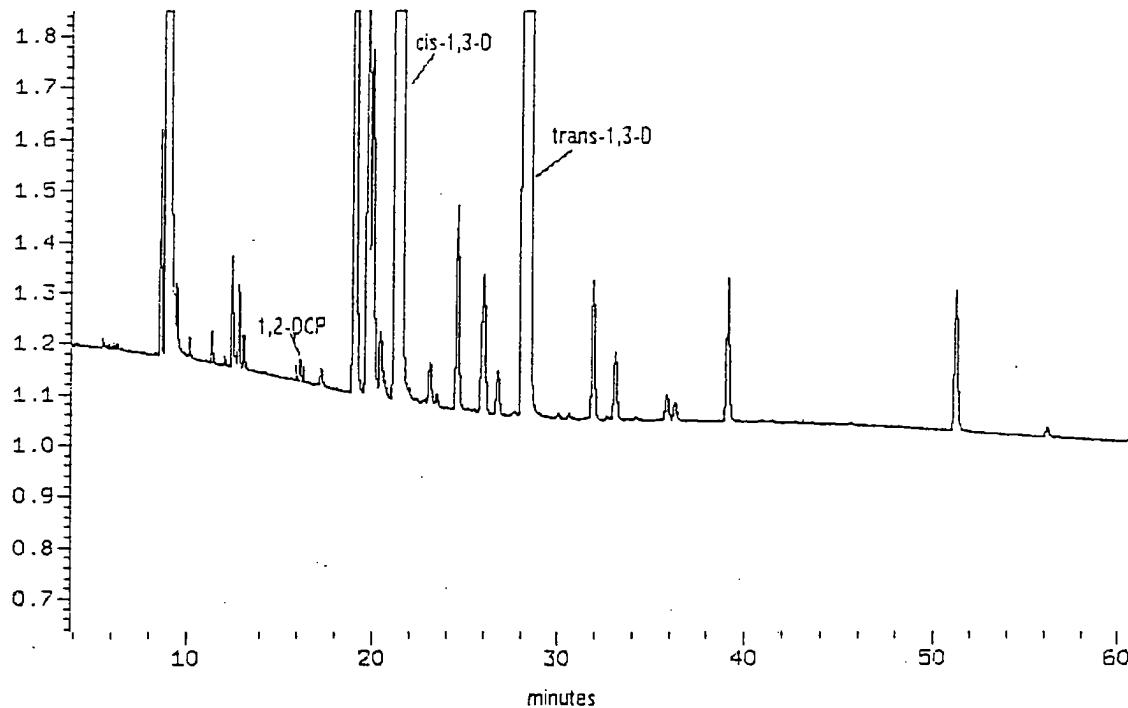
Figure 1. Chromatogram of a Calibration Solution



Datafile: 167gc021.i/DAS-AM-05-008.p/recovery.b/seq003_A.d

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

Figure 2. Chromatogram of a Sample Solution of Telone II Formulation



Datafile: 167gc021.i/DAS-AM-05-008.p/precisionDay2.b/seq006_A.d

Column:DB-1701 60 m x 0.32 mm x 1 μ m

Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes

Injection port: Split at 180°C with a ratio of 60:1

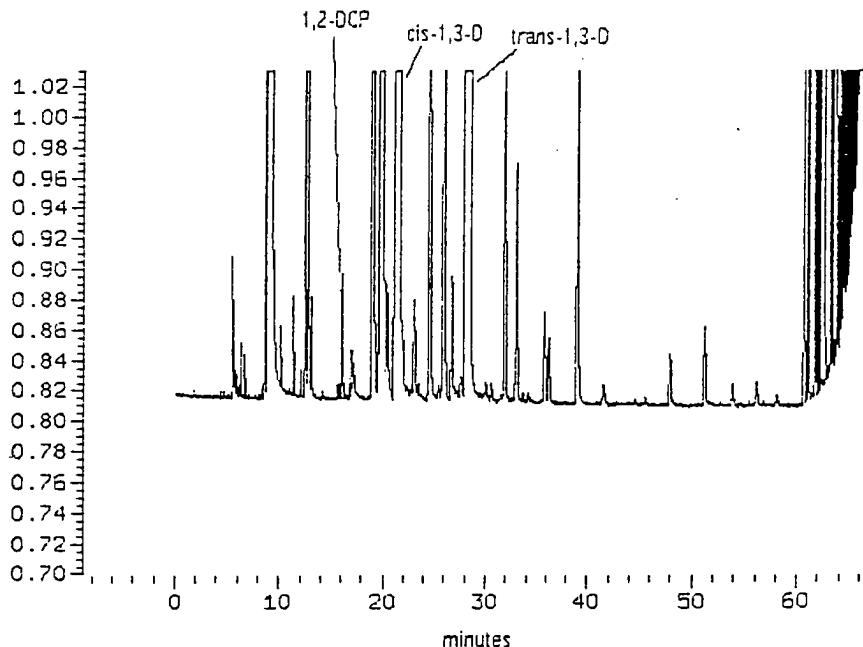
Detector: FID at 260°C

Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min

Injection volume: 2 μ L

Run Time: 66.3 minutes

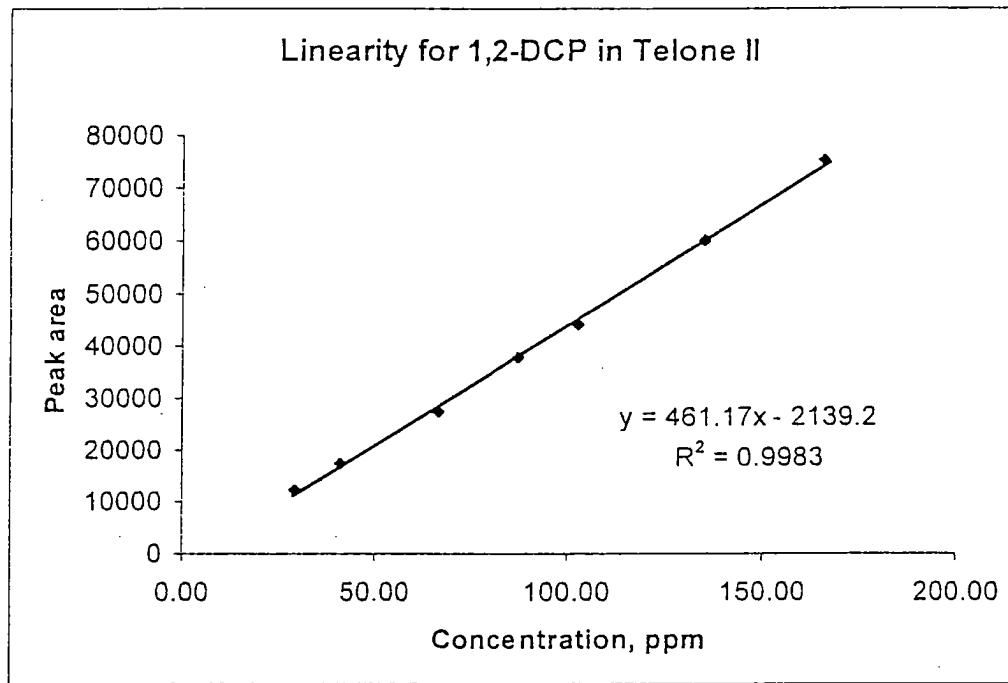
Figure 3. Chromatogram of a Sample Solution of Telone EC Formulation



Datafile: 167gc021.i/DAS-AM-05-008.p/precisionDay2.b/seq014_A.d

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

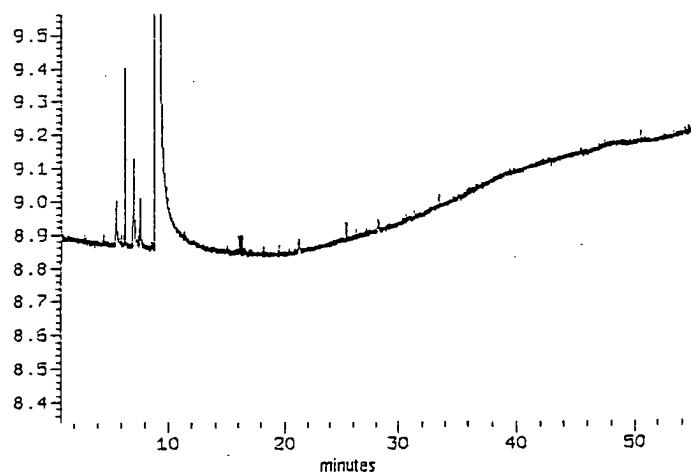
Figure 4. Linearity Plot for 1,2-DCP



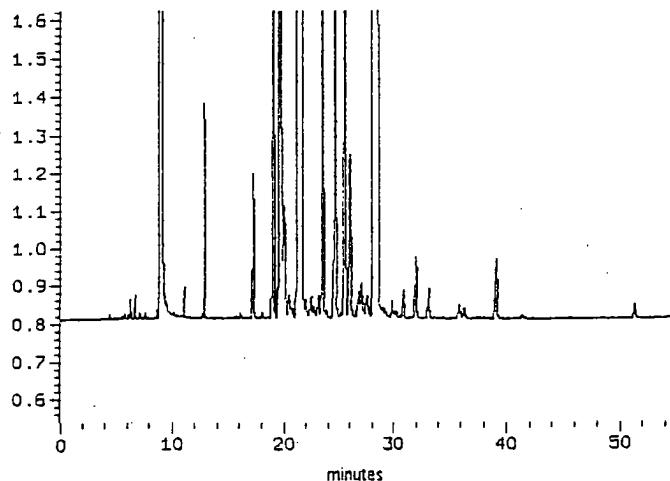
<u>Concentration,</u> <u>ppm</u>	<u>Peak area*</u>
166	75178
134	59956
103	44206
88	37762
65	27256
42	17371
30	12260

*Average of two injections.

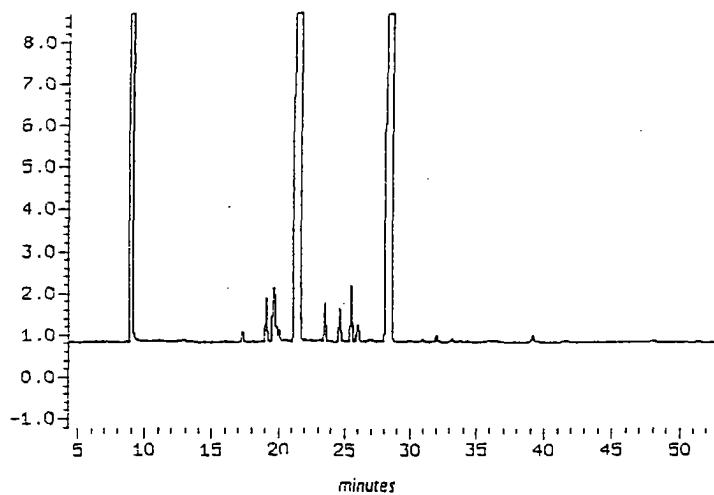
Figure 5. Chromatograms of Solvent Blank, Telone II Formulation Blank,
Telone EC Formulation Blank and 1,2-DCP



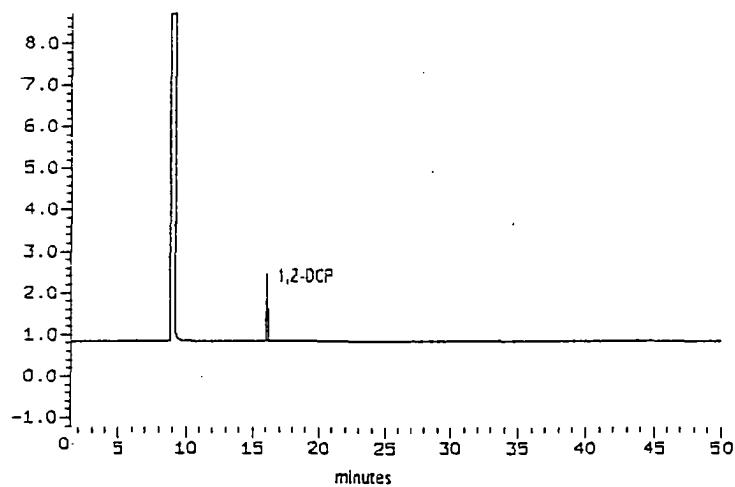
Ethyl acetate blank- seq018_A



Telone II Blank- seq015_A



Telone EC Blank- seq019_A

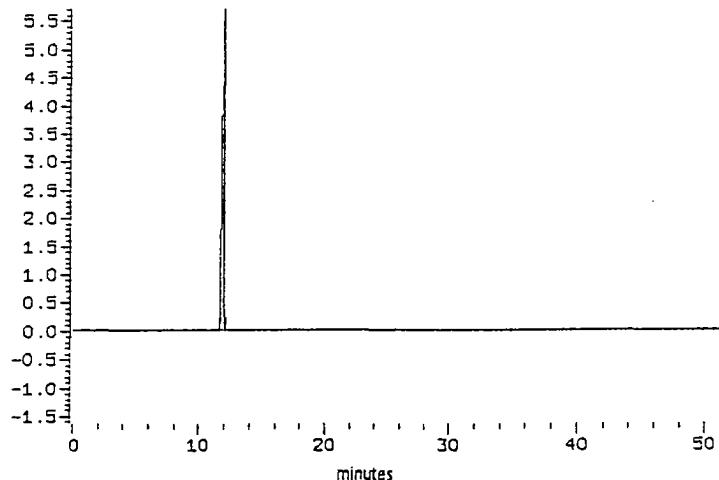


1,2-DCP- seq016_A

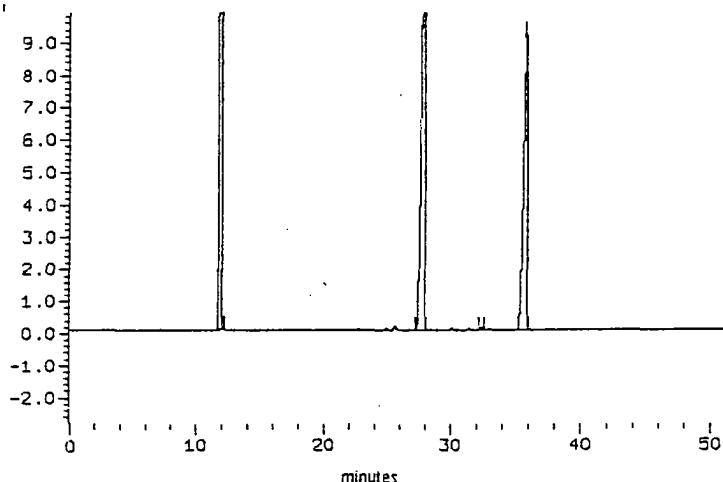
Datafiles: 167gc021.i/DAS-AM-05-008.p/interference.i/seq 18_A, 15_A, 19_A and 16_A

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

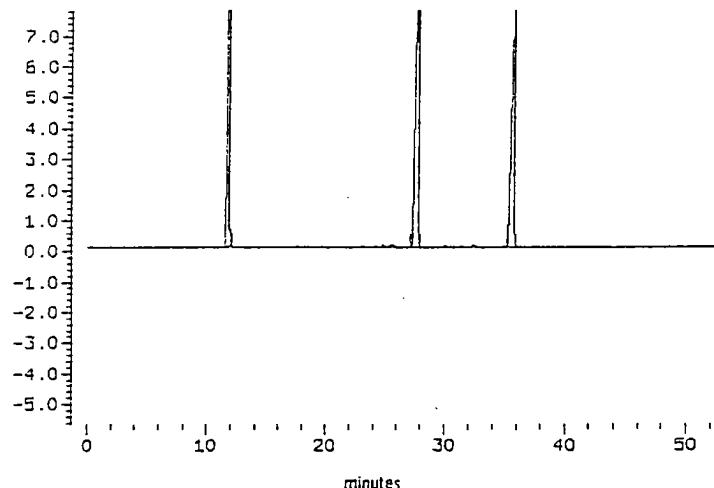
Figure 6. Effect of Flow Rate on Retention Times



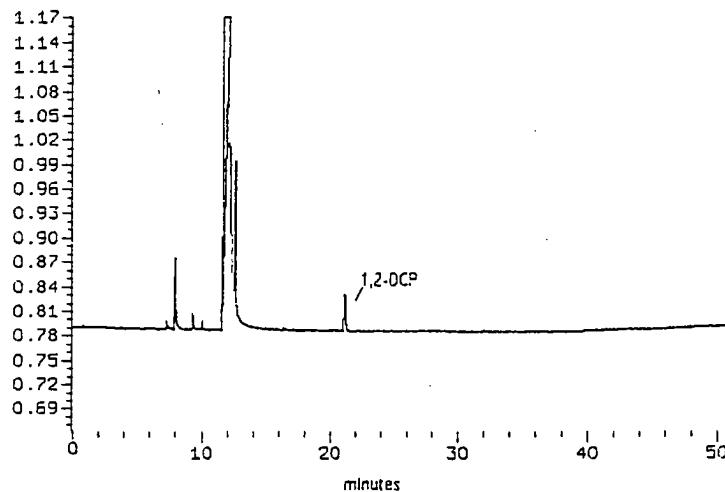
Solvent blank-seq001



Telone II blank-seq002



Telone EC blank-seq003

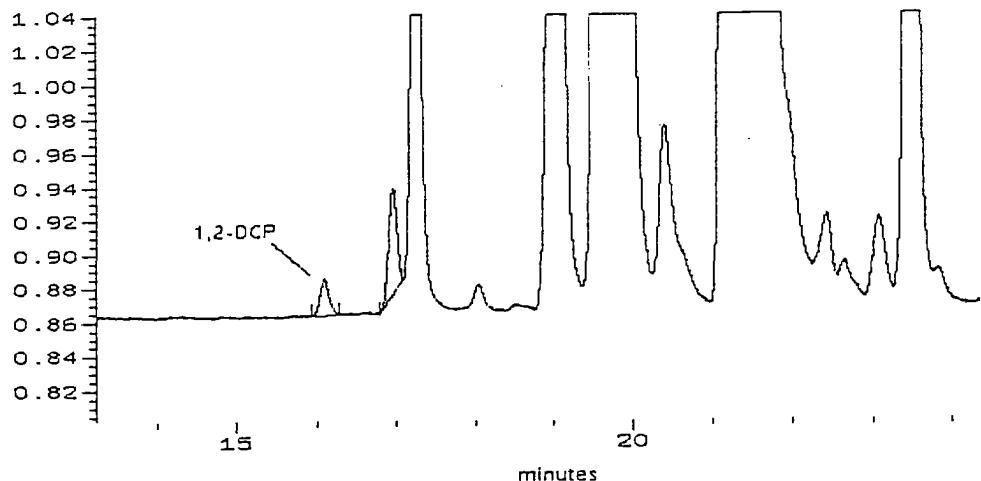


1,2-DCP-seq004d

Datafiles: 167gc021.i/DAS-AM-05-008.p/ruggedness.i/seq 001, 002, 003 and 004d

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 1.5 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

Figure 7. LOD Chromatogram



Datafiles: 167gc021.i/DAS-AM-05-008.p/ LOD.b/seq040.d

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier 2.2 mL/min of helium (constant flow)
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

VIII. APPENDIX

Analytical Method Summary

A. Preparation of calibration solution:

Prepare stock calibration solutions by weighing ~ 50 mg of 1,2-DCP reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate. Transfer 1 mL of this stock solution into a 20 mL volumetric flask and fill to the mark with ethyl acetate to afford a working standard in the range of 50 ppm 1,2-DCP.

B. Preparation of sample solution:

Add 2 mL (~ 2.4 g) of formulation into an appropriate sized jar via volumetric pipette and record the weight. Add 3 mL of ethyl acetate by volumetric pipette.

C. Instrumentation and Conditions:

1. Gas Chromatograph: Hewlett-Packard 6890 or equivalent

Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier ~ 2.2 mL/min of helium
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

Approximate Retention Time: 1,2-DCP 16 minutes

D. Calculations:

Calculation of response factors and weight percent values can be performed with a computing integrator/data system or with a spreadsheet.

1. Calculation of the response factor for 1,2-DCP in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(1,2\text{-dichloropropane})} \times 50 \text{ mL}} \times \frac{1}{20 \text{ mL}}$$

where:

RF = Response factor for 1,2-DCP

mg reference std = Weight of 1,2-DCP reference standard in calibration solution, mg

P = Purity of reference standard, expressed as a fraction

Area (1,2-DICHLOROPROPANE) = Peak area for 1,2-DCP in calibration solution

2. Calculation of the weight % of 1,2-DCP in the sample:

$$\text{Weight \%} = \frac{\text{Area}_{(1,2\text{-dichloropropane})} \times RF}{\text{Sample wt}} \times 5 \text{ mL} \times 100\%$$

where: Weight % = Weight % of 1,2-DCP in the sample

Area_(1,2-DICHLOROPROPANE) = Peak area for 1,2-DCP in the sample solution

RF = Response factor calculated for 1,2-DCP

Sample wt = Weight of sample in mg

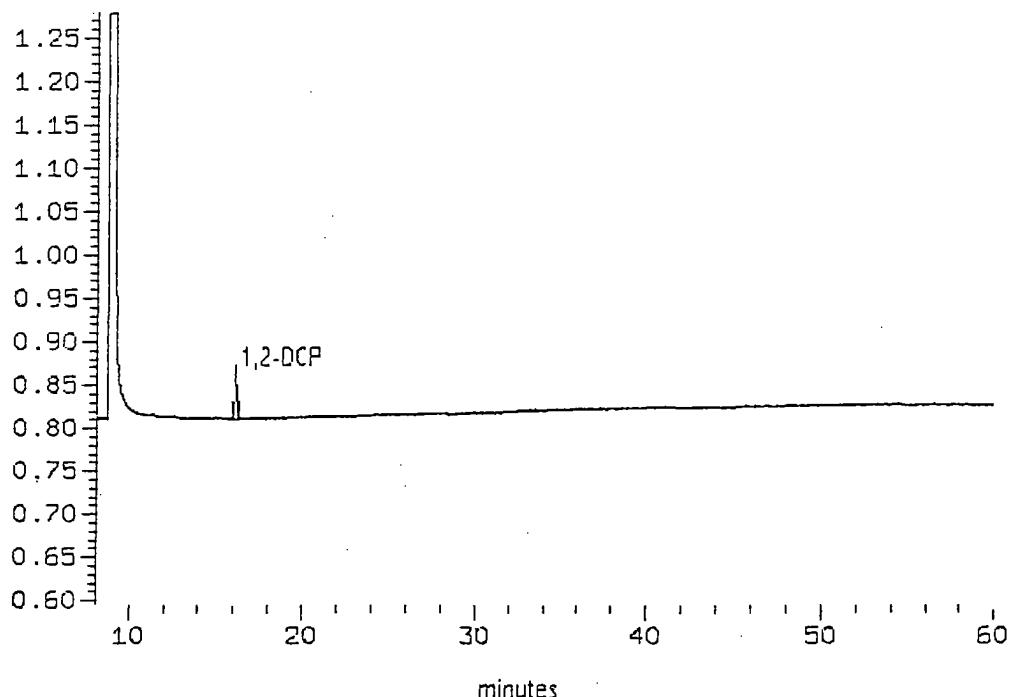
3. Calculation of the parts per million (ppm) of 1,2-DCP in the sample:

$$\text{ppm} = \text{Weight \%} \times 10000$$

Typical chromatograms of a calibration solution and sample solution are shown in the attached figures.

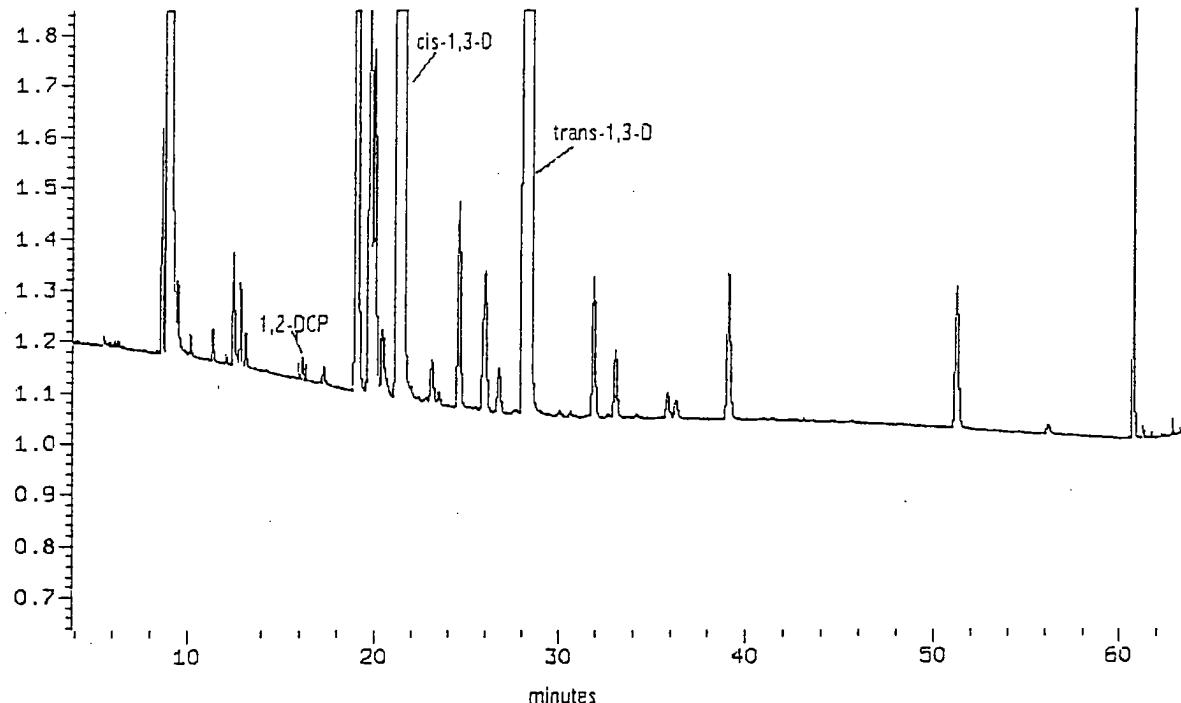
Additional details are provided in the body of the report.

Chromatogram of a Calibration Solution



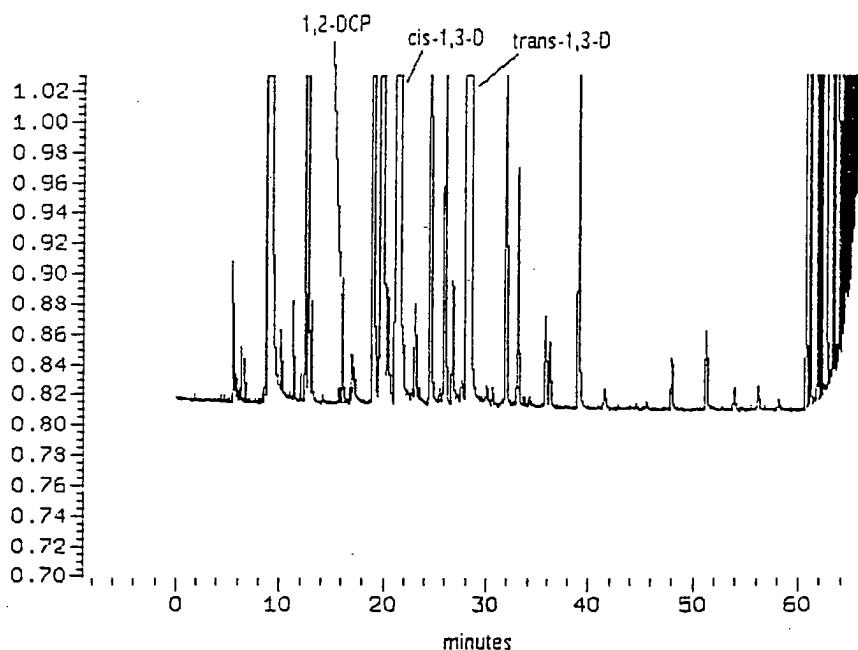
Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier ~ 2.2 mL/min of helium
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

Chromatogram of a Sample Solution of Telone II Formulation



Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier ~ 2.2 mL/min of helium
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes

Chromatogram of a Sample Solution of Telone EC Formulation



Column: DB-1701 60 m x 0.32 mm x 1 μ m
Oven Program: 50°C hold for 20 minutes,
0.5°C/minute to 70°C, hold for 0 minutes
30°C/minute to 260°C, hold for 0 minutes
Injection port: Split at 180°C with a ratio of 60:1
Detector: FID at 260°C
Flows: Carrier ~ 2.2 mL/min of helium
Hydrogen ~ 30 mL/min
Air ~ 360 mL/min
Aux (He) ~ 30 mL/min
Injection volume: 2 μ L
Run Time: 66.3 minutes