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REG	ANALYTICAL METHOD	NEW	15015029	34	07-NOV-2005

Report Title

ANALYTICAL METHOD AND VALIDATION FOR THE DETERMINATION OF HEXADIENE IMPURITY IN TELONE II TECHNICAL

Author(s) and ID's	Department
A. L. LATHAM (U364198)	
	Report / File Number(s)

Materials

H1A; ; TC; 1,3-D; TELONE II; INV

Keywords

2-CHLORO-1, 5-HEXADIENE; 1,3-D; LINEARITY; ACCURACY; PRECISION; RUGGEDNESS; LOQ; LOD; RECOVERY

Compound Number(s)	Batch/Lot Number(s)	Bayer Codes
	TSN104941; TSN105022	

Lab Notebooks	Language(s)	Performing Laboratory Name
	ENGLISH	DOW AGROSCIENCES LLC, INDIANAPOLIS, IN, USA

Data Requirement(s)

United States Guideline; 830.1800

Geo Area and Country (s) of Study		Protocol Number(s)	Study Number(s)	Published?	Date Study Completed
NA	US		DAS-AM-05-010	NO	07-NOV-2005
				Vertebrate?	GLP?
				N/A	YES

Method Number	Method Division	Method Status	Validated?
DAS-AM-05-010	FORMULATION	CURRENT	YES

Analyte	Method Type	SMC Code(s)	Enforcement Method?
2-CHLORO-1, 5-HEXADIENE	CHEMICAL		NO

Method Technique(s)

GC/FID

Reviewer(s)

R. M. NELSON (U424262);

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 Hexadiene Impurity in Telone II Technical

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham

AUTHOR

C. Alvis

STUDY COMPLETED ON

November 7, 2005

PERFORMING LABORATORY

Dow AgroSciences LLC
Supply R&D Laboratories
Analytical/Product Chemistry Center of Expertise
9330 Zionsville Road
Indianapolis, Indiana 46268

LABORATORY STUDY ID

DAS-AM-05-010

SUMMARY

This report describes the validation of an analytical method for determination of hexadiene impurities in Telone II. A gas chromatographic method was validated using a RTX-200 column with flame ionization detection (FID) and external standard quantitation.

The method is valid over a range of 0.096 to 1.05 wt. % hexadiene impurity in Telone II. The average recovery for the hexadiene impurity in Telone II was 99.4%, with a relative standard deviation of 16.1%. Detector response was shown to be linear for the hexadiene impurity over a range of 0.096 wt. % to 1.05 wt. %.

Replicate analyses of Telone II technical formulation on two separate days gave a relative standard deviation of 0.71% at an average concentration of 0.14 wt. % (wt/wt) hexadiene impurity. The analysis is complete in 66.3 minutes.

SUMMARY

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Analytical Method and Validation for the Determination of Hexadiene Impurity in Telone II Technical

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STATEMENT OF DATA CONFIDENTIALITY CLAIMS

Information claimed confidential on the basis of its falling within the scope of FIFRA Section 10(d)(1)(A), (B), or (C) has been removed to a confidential appendix, and is cited by cross-reference number in the body of the study

Company: Dow AgroSciences LLC

Company Agent: Bruce Houtman

Title: Regulatory Manager

Signature: Bruce Houtman /cal

Date: 9/26/05

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Study Initiation Date: April 20, 2005 Experimental Start Date: May 17, 2005

Experimental End Date: July 29, 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

Bruce Houtman /cae — 9/26/05

Bruce Houtman
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Dow AgroSciences LLC

Bruce Houtman /cae — 9/26/05

Bruce Houtman
Submitter
Dow AgroSciences LLC

— 11-7-05

Amy Latham
Study Director
Dow AgroSciences LLC

Study Completion Date

Dow AgroSciences Quality Assurance Unit
Good Laboratory Practice Statement Page

Compound: Hexadiene Impurity **Study ID:** DAS-AM-05-010

Title: Analytical Method and Validation for the Determination of Hexadiene Impurity in Telone II Technical

Study Initiation Date: 20-Apr-2005 **Study Completion Date:** 7-Nov-2005

GLP Quality Assurance Inspections

Date of GLP Inspection(s)	Date Reported to the Study Director and to Management	Phases of the Study which received a GLP Inspection by the Quality Assurance Unit
19-Apr-2005	20-Apr-2005	Protocol Review
20, 31-May-2005	1-Jun-2005	Recovery Analysis, Equipment Logs
26-Jul-2005	26-Jul-2005	Preparation of Limit of Quantitation (LOQ) Samples for Analysis
28-Jul-2005	29-Jul-2005	LOQ Determination
19, 20-Oct-2005	20-Oct-2005	Report and Raw Data, Test Substance Container and Sample Verification

QUALITY ASSURANCE STATEMENT:

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.

Julie Schwake — 7-Nov-2005

Julie Schwake
Dow AgroSciences, Quality Assurance

Date

SIGNATURES

Amy Latham Study Director Dow AgroSciences LLC	11-7-05 Date
Chris Alvis Investigator/Author Dow AgroSciences LLC	9/20/05 Date
Rose M. Nelson Reviewer Dow AgroSciences LLC	September 19, 2005 Date
Labib Ghaoui Manager Dow AgroSciences LLC	September 20, 2005 Date

Title: Analytical Method and Validation for the Determination of Hexadiene Impurity in Telone II Technical

Information found in the Confidential Attachment under Cross Reference Number 1.

CONFIDENTIAL ATTACHMENT

STUDY TITLE

Analytical Method and Validation for the Determination of Hexadiene Impurity in Telone II Technical

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR
Amy L. Latham
317-337-3582

AUTHOR
Chris Alvis

STUDY COMPLETED ON

November 7, 2005

PERFORMING LABORATORY
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LABORATORY STUDY ID

DAS-AM-05-010

CROSS REFERENCE NO. 1

This cross reference number noted on a place holder is used in place of the indicated page reference.

The deleted pages are attached immediately behind this page.

Page	Reason for Deletion	FIFRA Reference
6	Process Impurities Identified	10(d)(1)(A)

TABLE OF CONTENTS

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I. ABSTRACT

This report describes the validation of an analytical method for determination of hexadiene impurities in Telone II. A gas chromatographic method was validated using a RTX-200 column with flame ionization detection (FID) and external standard quantitation.

The method is valid over a range of 0.096 to 1.05 wt. % hexadiene impurity in Telone II. The average recovery for the hexadiene impurity in Telone II was 99.4%, with a relative standard deviation of 16.1%. Detector response was shown to be linear for the hexadiene impurity over a range of 0.096 wt. % to 1.05 wt. %.

Replicate analyses of Telone II technical formulation on two separate days gave a relative standard deviation of 0.71% at an average concentration of 0.14 wt. % (wt/wt) hexadiene impurity. The analysis is complete in 66.3 minutes.

II. INTRODUCTION

A. Scope

This GC method is applicable to the determination of 2-chloro-1, 5-hexadiene in Telone II formulation over the range of 0.096 wt. % to 1.05 wt. %. This report reflects what was actually done during the course of the study.

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 RTX-200 column with flame ionization 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, split/splitless inlet and autosampler, Hewlett Packard 6890, or equivalent
3. Data acquisition and processing system: Hewlett Packard ChemServer, or equivalent.
4. GC Column: RTX-200, 30 m x 0.32 mm x 1 µm
5. Autosampler vials and caps: 1.5 mL with screw caps
6. Autosampler vial inserts: 150 µL
7. Miscellaneous laboratory glassware.

B. Reagents and Standards:

1. Test and Reference Substance: 2-chloro-1, 5-hexadiene Reference Standard, TSN104941, 97.2%, recertification date February 20, 2007.
2. Ethyl acetate: EMD Omnisolv, or equivalent.
3. Test systems:
   Telone II Technical Formulation blank: E1912-54
   Telone II Technical Formulation blank: TSN105083
   Telone II Technical Formulation blank: TSN105085
   Telone II Technical Formulation: TSN105022

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 calibration solutions by weighing to the nearest 0.1mg ~ 50 mg of 2-chloro-1, 5-hexadiene reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate to afford a working standard containing approximately 1 mg/mL 2-chloro-1, 5-hexadiene.

2. Calibration procedure:

Inject the calibration solution at least twice into a gas chromatograph, using the conditions summarized in Section III.E, and calculate the response factor for 2-chloro-1, 5-hexadiene 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 technical formulation sample solution is shown in Figure 2.

4. Preparation of recovery samples:

A stock spiking solution was prepared by adding 13.4 mg 2-chloro-1, 5-hexadiene to 1 mL Telone II to afford a 10702 ppm (1.07 wt%) solution of 2-chloro-1,5-hexadiene in Telone II.

Samples containing 2-chloro-1, 5-hexadiene were prepared for Telone II by weighing aliquots of the Telone II (TSN105083) into 1.5 mL autosampler vials (Table I). Aliquots of 2-chloro-1, 5-hexadiene stock solution were then added to each sample of Telone II, diluted with ethyl acetate, and contents were analyzed by GC.

5. Preparation of linearity solutions:

   The five acceptable 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 technical formulations (fortified with 2-chloro-1, 5-hexadiene to ensure samples 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:
   Gas Chromatograph: Hewlett-Packard 6890 or equivalent

Column:	RTX-200 30 m x 0.32 mm x 1 µm
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 µL
Run Time:	66.3 minutes

2. Approximate time to prepare and analyze sample: 3 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. For this study spreadsheets were used.

1. Calculation of the response factor for 2-chloro-1, 5-hexadiene in the calibration solution:

$$RF = \frac{\text{mg reference std x P}}{\text{Area}_{(2\text{ - chloro - 1,5 - hexadiene})} \text{ x 50 mL}}$$

where: RF = Response factor for 2-chloro-1,5-hexadiene

mg reference std = Weight of 2-chloro-1,5-hexadiene reference standard in calibration solution, mg

P = Purity of reference standard, expressed as a fraction

$Area_{(2\text{-chloro-1,5-hexadiene})}$ = Peak area for 2-chloro-1,5-hexadiene in calibration solution

2. Calculation of the weight % of 2-chloro-1, 5-hexadiene in the sample:

$$\text{Weight \%} = \frac{Area_{(2\text{-chloro-1,5-hexadiene})} \times RF}{\text{Sample wt}} \times DF \times 100$$

where: Weight % = Weight % of 2-chloro-1,5-hexadiene in the sample

$Area_{(2\text{-chloro-1,5-hexadiene})}$ = Peak area for 2-chloro-1,5-hexadiene in the sample solution

RF = Response factor calculated for 2-chloro-1, 5-hexadiene

Sample Wt = Weight of sample in mg

DF = Dilution factor (5 mL for a sample prepared as described in Section III.D.3)

IV. RESULTS AND DISCUSSION

A. Linearity

The linearity for 2-chloro-1, 5-hexadiene was evaluated using the GC conditions used for this study. A linear relationship between peak area and concentration ($r^2 = 0.999$) was noted for 2-chloro-1, 5-hexadiene from 0.096 wt. % to 1.05 wt. %. The linearity plot is shown in Figure 3.

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 Table I. Samples were analyzed using the calibration solution described in Section III.D.1. The blank technical formulation contained a small amount of 2-chloro-1, 5-hexadiene prior to spiking so the blanks were analyzed without fortification and the amount present determined (0.0048 wt. % in Telone II). 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 0.096 wt. % to 1.05 wt. % 2-chloro-1,5 -hexadiene in Telone II.

The recovery for 2-chloro-1,5-hexadiene in Telone II ranged from 54 % to 119 %, with an average recovery of 99.4%, and a relative standard deviation of 16.1 %. An apparent bias

exists in the method with a decreased recovery (<100%) at low concentrations and an increased recovery (>100%) at higher concentrations. Recovery data are shown in Table II. The recovery values shown were calculated using an Excel spreadsheet. Due to rounding, minor differences may occur between percent recovery stated in Table II 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 technical formulation, 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 III.

The relative standard deviation was 0.71 % at an average concentration of 0.14 wt. % 2-chloro-1, 5-hexadiene in Telone II. The Horwitz RSD_r value was calculated to be 3.60 for Telone II; therefore, results are acceptable (Table III).

D. System Precision

System precision was determined by injecting a prepared solution of Telone II technical formulation five times. Data obtained are shown in Table IV. The relative standard deviation for the peak area for 2-chloro-1, 5-hexadiene for all five injections of Telone II was 1.5%.

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 not equivalent to the original results for Telone II.

F. Interferences

No interferences were detected for the ethyl acetate solvent, technical formulation inert ingredients, or 2-chloro-1, 5-hexadiene. Chromatograms of a solvent blank, technical formulation blank, and 2-chloro-1,5-hexadiene for Telone II, respectively, are shown in Figure 4.

G. Ruggedness

The method ruggedness was tested by reducing the flow rate from the nominal 2.2 mL/min to 1.2 mL/min. The retention times of the component of interest changed significantly, as shown in Figure 5. 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 0.13 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. The Horwitz RSD_r value was calculated to be 3.71 for Telone II with an average recovery of 87.5%. LOQ data are shown in Table V.

Limit of detection was determined by reviewing the formulation blank and determining that the 2-chloro-1, 5-hexadiene had a greater than three times signal to noise ratio. The LOD was determined to be 0.0048 wt. % 2-chloro-1,5-hexadiene.

V. CONCLUSIONS

This method is applicable to the determination of 2-chloro-1, 5-hexadiene in Telone II technical formulation over the range of 0.096 to 1.05 wt. %. The precision, recovery and linearity data have shown this method to be acceptable for the determination of 2-chloro-1, 5-hexadiene in Telone II. 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, protocol 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 of Telone II (TSN105083) sample plus weight of spike, mg	Weight of 2-chloro-1, 5-hexadiene added (mg)*	Volume of spike solution added (mL)	Wt.% 2-chloro-1, 5-hexadiene added *
Recovery 8	243.1	2.60	Stock 2	1.07
Recovery 9	610.6	1.30	0.100	0.213
Recovery 10	579.8	0.964	0.074	0.166
Recovery 11	548.1	0.651	0.050	0.119
Recovery 12	543.4	0.521	0.040	0.096
Recovery 13	527.3	0.391	0.030	0.074
Recovery 14	516.7	0.260	0.020	0.050

* Not including amount present in blank

Weight of 2-chloro-1, 5-hexadiene added (mg) =

$$\frac{\text{Weight of 2 - chloro present in spike solution (mg) x Purity (97.2\%)}}{\text{Total volume (mL)}} \text{ x volume of spike added}$$

Wt. % of 2-chloro-1, 5-hexadiene added =

$$\frac{\text{Weight of 2 - chloro - 1, 5 - hexadiene added (mg)}}{\text{Weight of Telone II (TSN105083) sample plus weight of spike, mg}} \text{ x 100\%}$$

Table II. Recovery Data for 2-chloro-1, 5-hexadiene in Telone II Technical Formulation

Sample	2-chloro-1, 5-hexadiene Wt. % added*	2-chloro-1, 5-hexadiene Wt. % found	Recovery %
Recovery 8	1.07	1.28	119
Recovery 9	0.218	0.233	107
Recovery 10	0.171	0.174	102
Recovery 11	0.124	0.115	93
Recovery 12	0.101	0.077	77
Recovery 13	0.0790	0.052	66
Recovery 14	0.0551	0.030	55
		Average	99.4**
		Std. Dev.	16.1
		R.S.D	16.2

* Amount added plus amount present in blank (0.0047%)
Recovery % = Wt % 2-chloro-1, 5-hexadiene found / Wt. % 2-chloro-1, 5-hexadiene added x 100
Std. Dev. = Standard Deviation
R.S.D. = Relative Standard Deviation= Standard Deviation / Average x 100
**Average recovery does not include recovery 13 and 14

Table III. Method Precision Data for 2-chloro-1,5-hexadiene in Telone II Technical Formulation

Sample	Telone II Wt. %
TSN105022-prec 1-1	0.1397
TSN105022-prec 1-2	0.1406
TSN105022-prec 1-3	0.1425
TSN105022-prec 1-4	0.1410
TSN105022-prec 1-5	0.1409
TSN105022-prec 2-1	0.1394
TSN105022-prec 2-2	0.1417
TSN105022-prec 2-3	0.1399
TSN105022-prec 2-4	0.1418
TSN105022-prec 2-5	0.1413
Average	0.14
Std. Dev.	0.0010
*R.S.D.	0.71
*Horwitz RSD_R	5.37
*Horwitz RSD_r	3.60
RSD < Horwitz RSD_r?	Yes; results are acceptable

* Based on weight percent
Std. Dev. = Standard Deviation
R.S.D = Relative Standard Deviation = Standard Deviation/Average x 100
Average of duplicate injections

Table IV. System Precision Data for 2-chloro-1,5-hexadiene

Sample	Peak Area
Telone II	
Injection #1	1652389
Injection #2	1689122
Injection #3	1644674
Injection #4	1635123
Injection #5	1624994
Average	1649260
Std. Dev.	24538
R.S.D.	1.5

Std. Dev. = Standard Deviation
R.S.D = Relative Standard Deviation = Standard Deviation / Average x 100

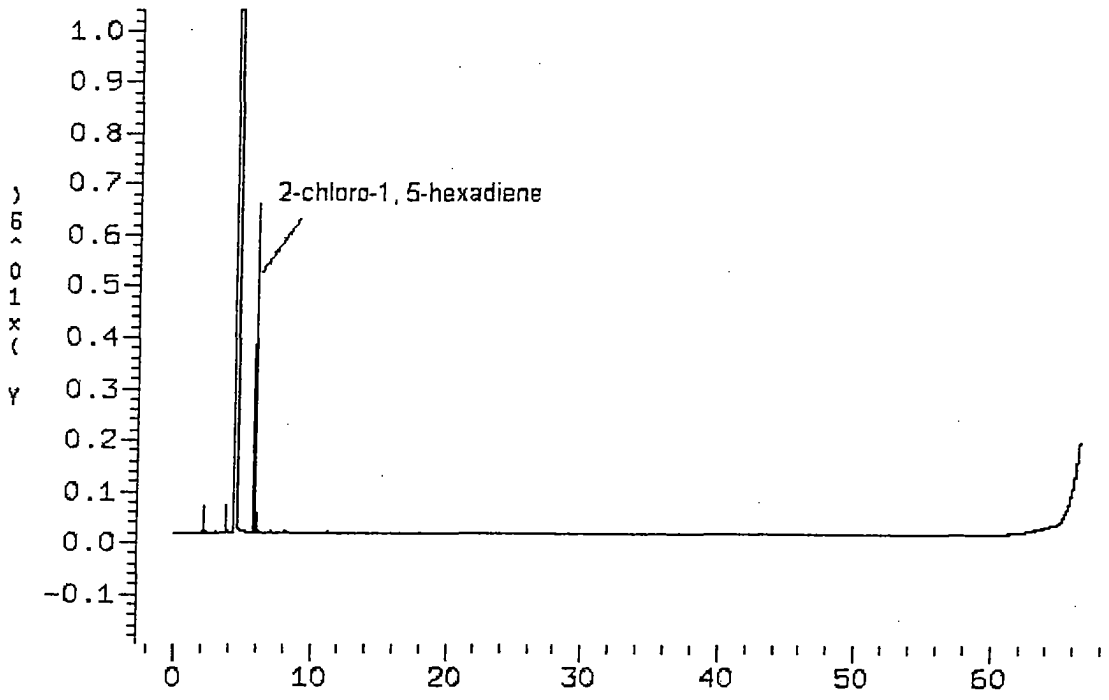
Table V. LOQ Data for 2-chloro-1,5-hexadiene

Sample	Wt. % 2-chloro-1,5-hexadiene Found	Wt. % 2-chloro-1,5-hexadiene added	% Recovery
Telone II			
LOQ-6	0.111	0.128	86.7
LOQ-7	0.120	0.134	89.6
LOQ-8	0.113	0.131	86.3
LOQ-9	0.114	0.131	87.0
LOQ-10	0.115	0.131	87.8
Average	0.115		87.5
Std. Dev.	0.003		
*R.S.D.	2.61		
*Horwitz RSD_R	5.54		
*Horwitz RSD_r	3.71		
RSD < Horwitz RSD_r?	Yes; results are acceptable		

Std. Dev. = Standard Deviation
R.S.D. = Relative Standard Deviation = Standard Deviation/Average x 100

VII. FIGURES

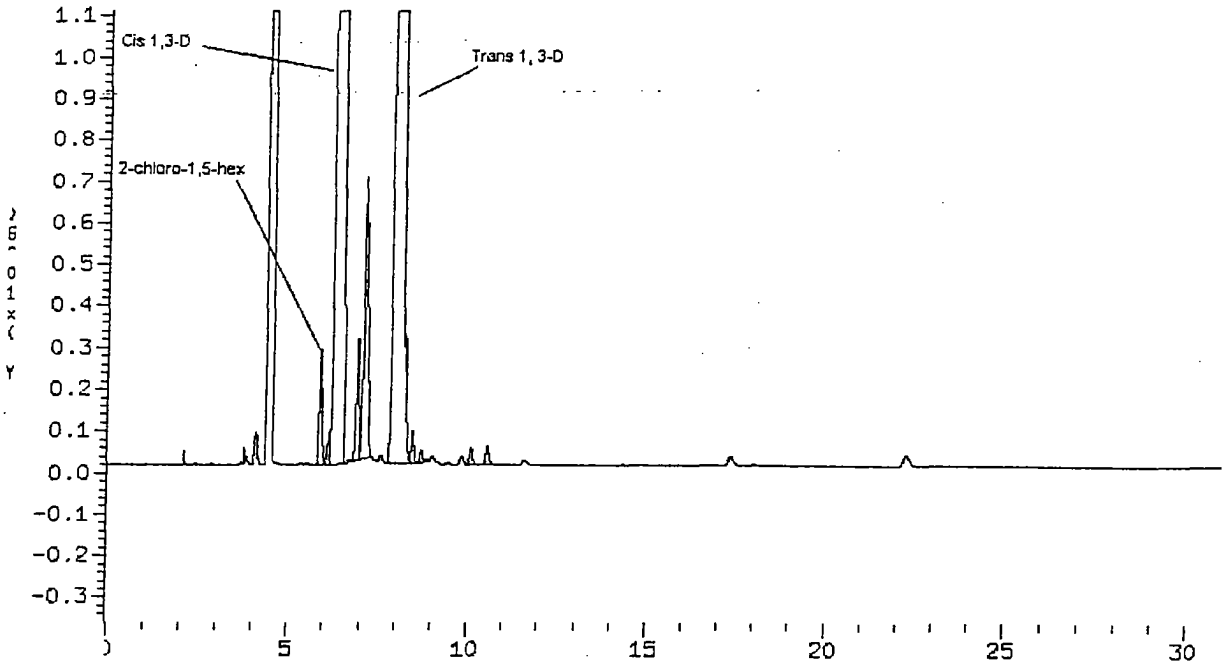
Figure 1. Chromatogram of a Calibration Solution



Datafile: /var/chem./167gc062.i/DAS-AM-05-010.p/E2004p30.b/seq006.d

Column:	RTX-200 30 m x 0.32 mm x 1µm
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 µL
Run Time:	66.3 minutes

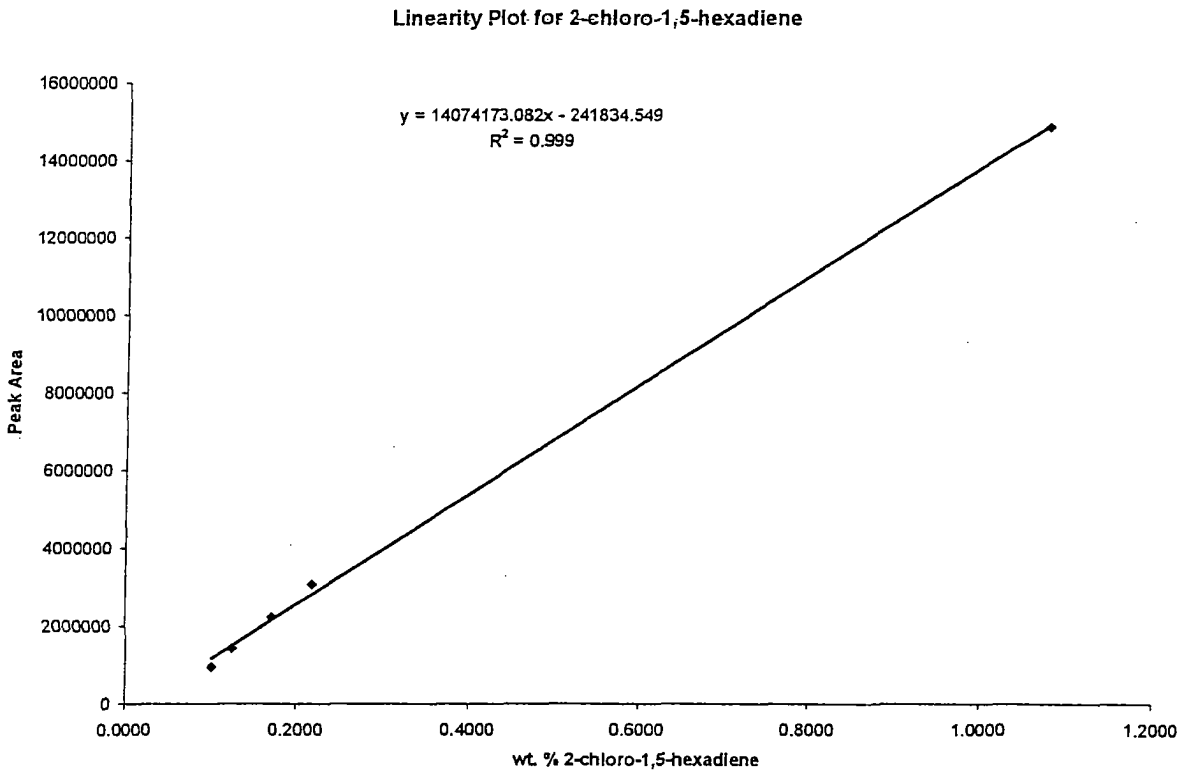
Figure 2. Chromatogram of a Sample Solution of Telone II Technical Formulation



Datafile: /var/chem./167gc62.i/DAS-AM-05-010.p/E2004p30.b/seq010.d

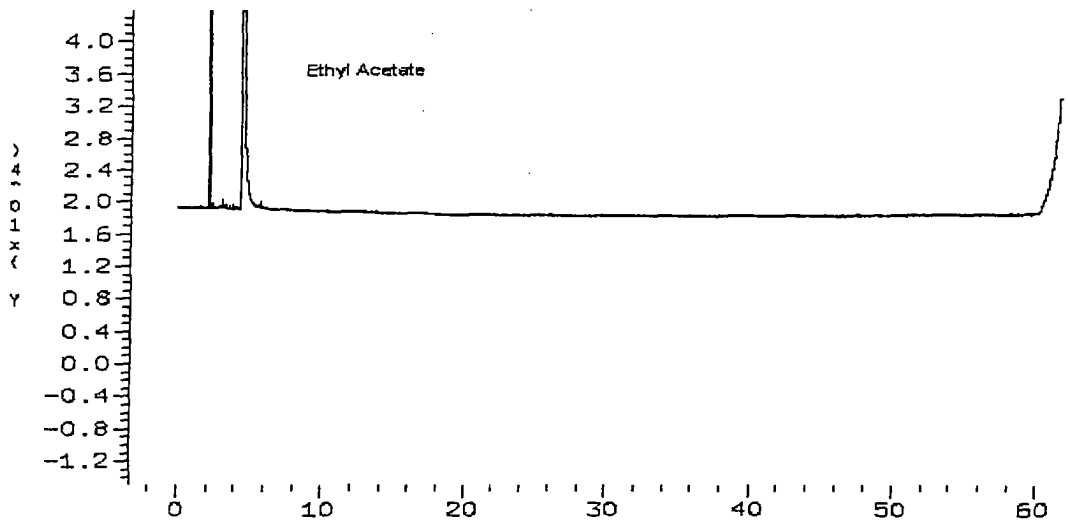
Column:	RTX-200 30 m x 0.32 mm x 1µm
Oven Program:	50°C hold for 20 minutes, 0.5°C/minute to 70°C, hold for 0 minutes 30°C/minute to 260°C, hold for 0 minutes
Injection port:	Split at 180°C with a ratio of 60:1
Detector:	FID at 260°C
Flows:	Carrier 2.2 mL/min of helium (constant flow) Hydrogen ~ 30 mL/min Air ~ 360 mL/min Aux (He) ~ 30 mL/min
Injection volume:	2 µL
Run Time:	66.3 minutes

Figure 3. Linearity Plot for 2-chloro-1, 5-hexadiene



Weight %, 2-chloro-1,5-hexadiene	Peak area
1.07	14850833
0.218	3078907
0.171	2234885
0.124	1429288
0.101	952711

Figure 4. Chromatograms of Solvent Blank, Telone II Technical Formulation Blank, and 2-chloro-1,5-hexadiene



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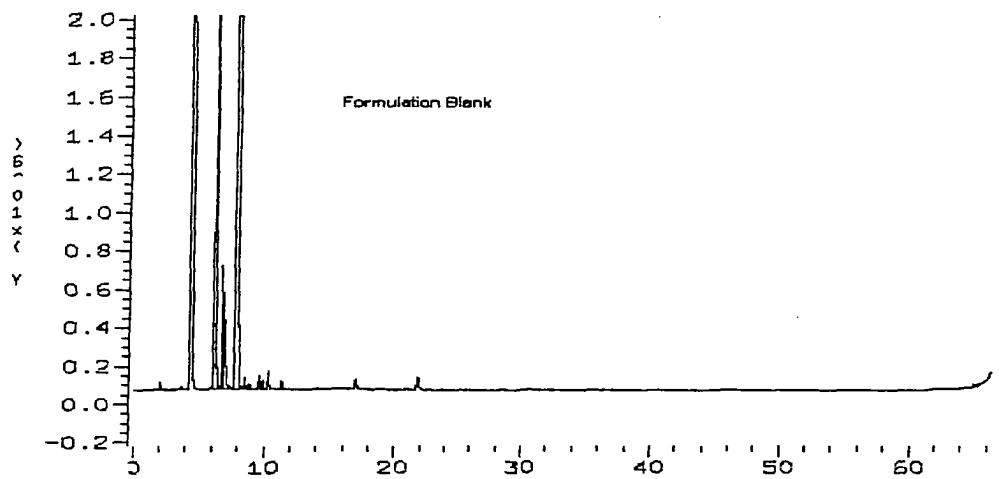
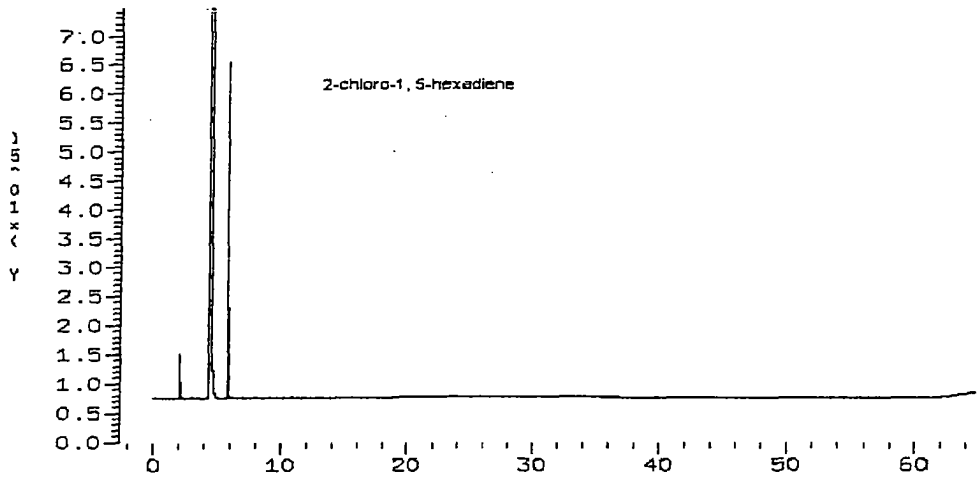


var/chem./167gc055.i/FOR-05-034_das-am-05-009./SolvayLotsAndRecovery.b/seq004.d

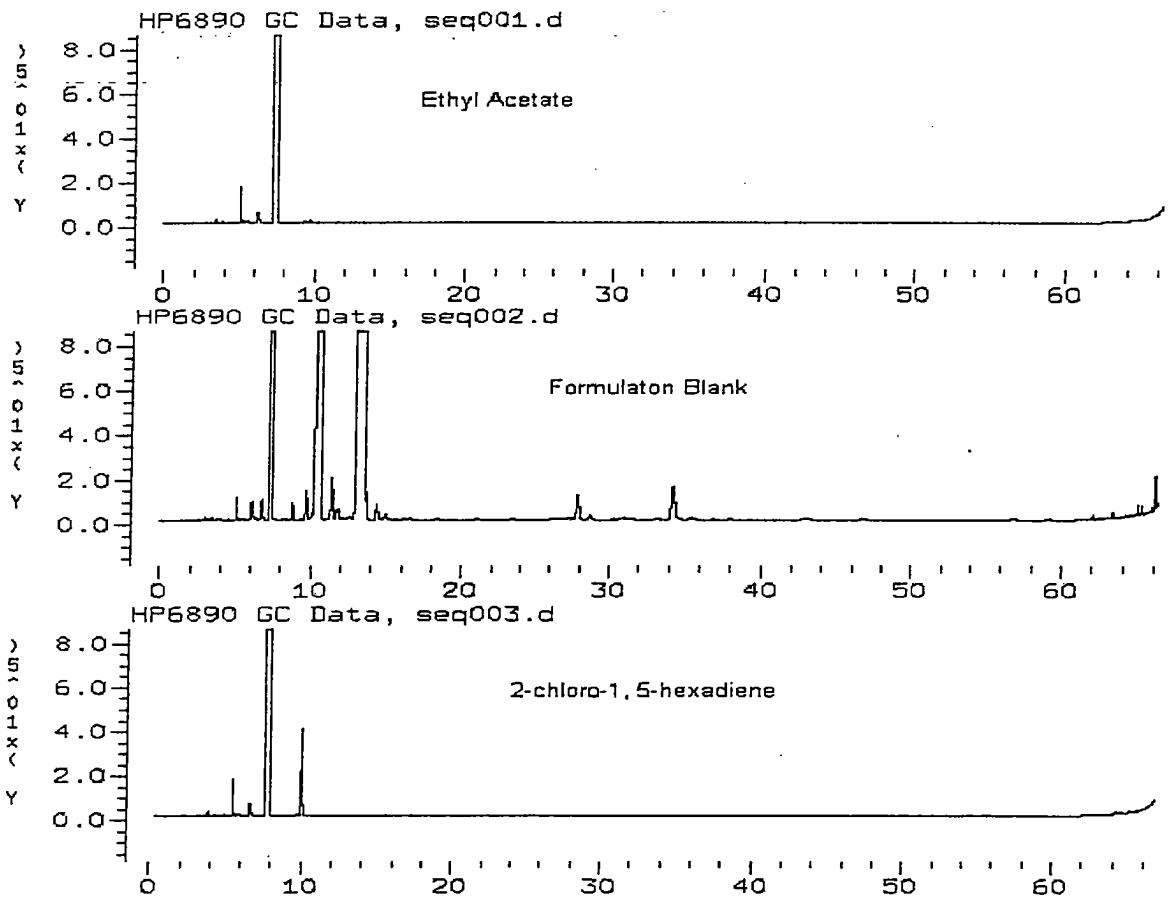
Figure 4, continued, Chromatograms of Solvent Blank, Telone II Technical Formulation Blank,
and 2-chloro-1,5-hexadiene



var/chem./167gc055.i/FOR-05-034_das-am-05-009./SolvayLotsAndRecovery.b/seq002.d

Column:	RTX-200 30 m x 0.32 mm x 1µm
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 µL
Run Time:	66.3 minutes

Figure 5. Effect of Flow Rate on Retention Times



Datafiles: /var/chem/167gc062.i/DAS-AM-05-010.p/E2004p35-2.b/seq 001.d, seq002.d, seq003.d

Column:	RTX-200 30 m x 0.32 mm x 1µm
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 1.2 mL/min of helium (constant flow) Hydrogen ~ 30 mL/min Air ~ 360 mL/min Aux (He) ~ 30 mL/min
Injection volume:	2 µL
Run Time:	66.3 minutes

VIII. APPENDIX

Analytical Method Summary

A. Preparation of calibration solution:

Prepare calibration solutions by weighing ~ 50 mg of 2-chloro-1 ,5-hexadiene reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate to afford a working standard in the range of 1mg/mL 2-chloro-1,5-hexadiene.

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:	RTX-200 30m x 0.32 mm x 1µm
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 µL
Run Time:	66.3 minutes

Approximate Retention Time: 2-chloro-1, 5-hexadiene 5.7 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 2-chloro-1,5-hexadiene in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(2\text{-chloro-1,5-hexadiene})} \times 50\ \text{mL}}$$

where:
RF = Response factor for 2-chloro-1,5-hexadiene
mg reference std = Weight of 2-chloro-1, 5-hexadiene reference standard in calibration solution, mg
P = Purity of reference standard, expressed as a fraction
$\text{Area}_{(2\text{-chloro-1,5-hexadiene})}$ = Peak area for 2-chloro-1,5-hexadiene in calibration solution

2. Calculation of the weight % of 2-chloro-1,5-hexadiene in the sample:

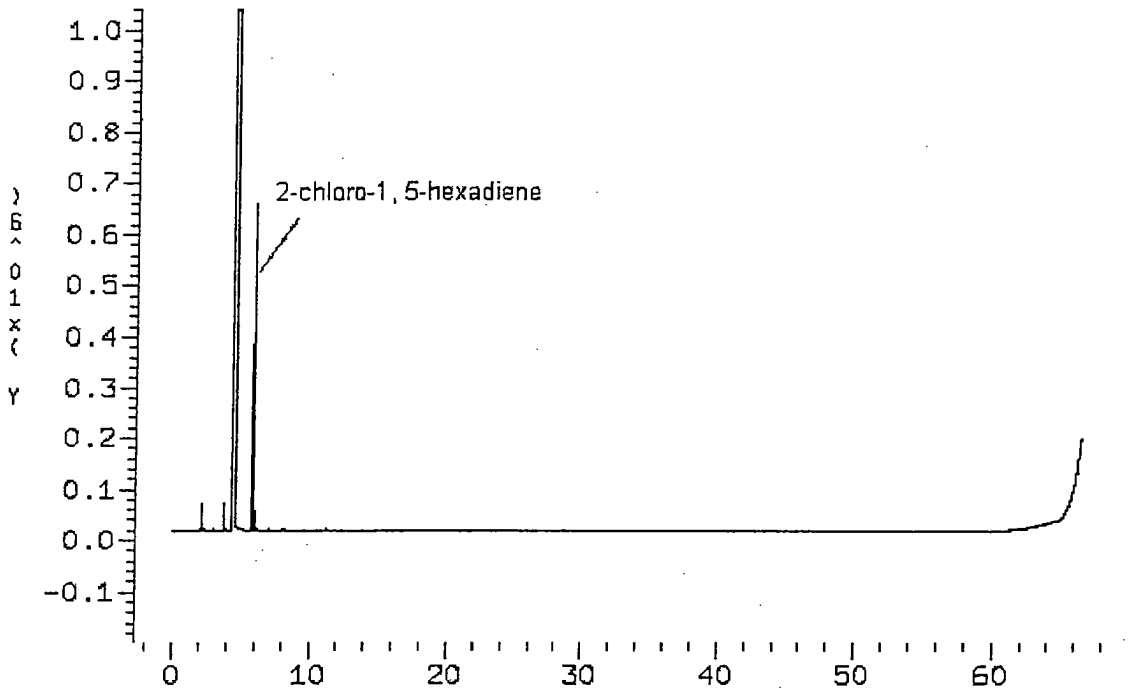
$$\text{Weight \%} = \frac{\text{Area}_{(2\text{-chloro-1,5-hexadiene})} \times RF}{\text{Sample wt}} \times 5\ \text{mL} \times 100\%$$

where:
Weight % = Weight % of 2-chloro-1,5-hexadiene in the sample
$\text{Area}_{(2\text{-chloro-1,5-hexadiene})}$ = Peak area for 2-chloro-1,5-hexadiene in the sample solution
RF = Response factor calculated for 2-chloro-1,5-hexadiene
Sample wt = Weight of sample in mg

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



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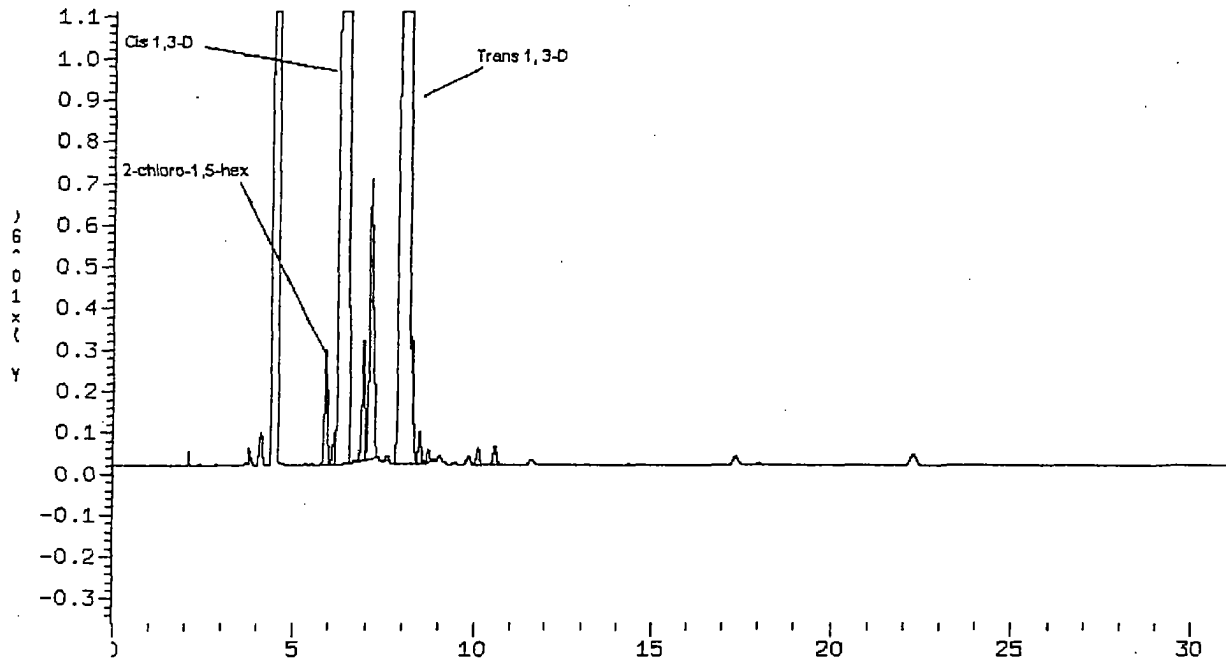
Injection port:	Split at 180°C with a ratio of 60:1
Detector:	FID at 260°C
Flows:	Carrier 2.2 mL/min of helium Hydrogen ~ 30 mL/min Air ~ 360 mL/min Aux (He) ~ 30 mL/min
Injection volume:	2 µL
Run Time:	66.3 minutes

Chromatogram of a Sample Solution of Telone II Technical Formulation



Column:	RTX-200 30m x 0.32 mm x 1µm
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 µL
Run Time:	66.3 minutes