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Report Title

ANALYTICAL METHOD AND VALIDATION FOR THE DETERMINATION OF PROCESS IMPURITIES IN TELONE II TECHNICAL

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

Materials

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

Keywords

3-CHLORO-1, 5-HEXADIENE; LINEARITY; RELATIVE STANDARD DEVIATION; ACCURACY; PRECISION; LOQ; LOD; RUGGEDNESS; RECOVERY; 1,3-DICHLOROPROPENE; 1,3-D; HEXADIENE IMPURITY

Compound Number(s)	Batch/Lot Number(s)	Bayer Codes
	TSN104959; TSN105019	

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-05-009	NO	04-NOV-2005
				Vertebrate?	GLP?
				N/A	YES

Method Number	Method Division	Method Status	Validated?
DAS-AM-05-009	FORMULATION	CURRENT	YES

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

Method Technique(s)

GC/TCD

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 Process Impurities in Telone II Technical

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham

STUDY COMPLETED ON

November 4, 2005

PERFORMING LABORATORY

Dow AgroSciences LLC
Supply R&D Laboratories
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Indianapolis, Indiana 46268

LABORATORY STUDY ID

DAS-AM-05-009

SUMMARY

This report describes the validation of an analytical method for determination of a hexadiene impurity in Telone II technical formulation. A gas chromatographic method was validated using a DB-1701 column with thermal conductivity detection (TCD) and external standard quantitation.

STUDY TITLE

Analytical Method and Validation for the Determination of Process Impurities in Telone II Technical

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham
317-337-3582

STUDY COMPLETED ON

November 4, 2005

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LABORATORY STUDY ID

DAS-AM-05-009

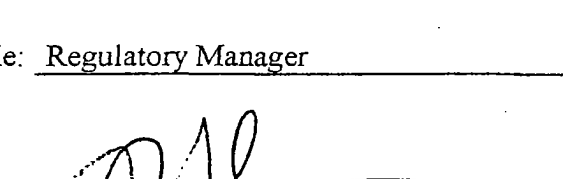
STATEMENT OF DATA CONFIDENTIALITY CLAIMS

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: 

Date: 10/6/05

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Study Initiation Date: May 25, 2005

Experimental Start Date: June 16, 2005

Experimental End Date: August 15, 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
Sponsor
Dow AgroSciences LLC

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Date

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Submitter
Dow AgroSciences LLC

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Date

Amy Latham
Study Director
Dow AgroSciences LLC

11-4-05
Study Completion Date

Dow AgroSciences Quality Assurance Unit
Good Laboratory Practice Statement Page

Compound: Telone II Technical **Study ID:** DAS-AM-05-009

Title: Analytical Method and Validation for the Determination of Process Impurities in Telone II Technical

Study Initiation Date: 25-May-2005 **Study Completion Date:** 4-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
9-May-2005	1-Jun-2005	Protocol Review
31-May & 1-Jun-2005	2-Jun-2005	Calibration Solution Preparation, Recovery Sample Preparation & Analysis, Equipment Logs
21, 24, 25-Oct-2005	26-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 — 4-Nov-2005

Julie Schwake
Dow AgroSciences, Quality Assurance

Date

SIGNATURE PAGE

11-4-05

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Dow AgroSciences LLC

Date

September 29, 2005

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Reviewer
Dow AgroSciences LLC

Date

10/7/05

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Date

October 7, 2005

Labib Ghaoui
Analytical Leader
Dow AgroSciences LLC

Date

Title: Analytical Method and Validation for the Determination of Process Impurities 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 Process Impurities in Telone II Technical

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

Amy L. Latham
317-337-3582

STUDY COMPLETED ON

November 4, 2005

PERFORMING LABORATORY

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LABORATORY STUDY ID

DAS-AM-05-009

Cross Reference Number 1 This cross reference number noted on a place holder page is used in place of the indicated page reference.

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

TABLE OF CONTENTS, continued

I. ABSTRACT

This report describes the validation of an analytical method for determination of 3-chloro-1, 5-hexadiene in Telone II technical formulation. A gas chromatographic method was validated using a DB-1701 column with thermal conductivity detection (TCD) and external standard quantitation.

The method is valid over a range of 0.045 to 0.934 wt. % 3-chloro-1, 5-hexadiene in Telone II. The average recovery for 3-chloro-1, 5-hexadiene in Telone II was 94.6%, with a relative standard deviation of 10.9%. Detector response was shown to be linear for 3-chloro-1, 5-hexadiene over a range of 0.045 to 0.934 wt. %.

Replicate analyses of Telone II technical formulation on two separate days gave a relative standard deviation of 2.89% at an average concentration of 0.068 wt. % for Telone II. The analysis is complete in 30 minutes.

II. INTRODUCTION

A. Scope

This GC method is applicable to the determination of 3-chloro-1, 5-hexadiene in Telone formulations. The method was validated over the range of 0.045 wt. % to 0.934 wt. % 3-chloro-1, 5-hexadiene in Telone II.

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 TCD 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 thermal conductivity detector and split/splitless Injector, Hewlett Packard 6890 or equivalent
3. Data acquisition and processing system: Hewlett Packard ChemServer, or equivalent.
4. GC Column J & W Scientific DB-1701, 60 m x 0.32 mm x 1 µm
5. Autosampler vials and caps: 1.5 mL with screw caps
6. Miscellaneous laboratory glassware.

B. Reagents and Standards:

1. Test and Reference Substance: 3-chloro-1, 5-hexadiene Reference Standard, TSN104959, 95%, recertification date February 10, 2007.
2. Ethyl acetate: Mallinckrodt ChromAR HPLC Grade, or equivalent.
3. Test systems:
   Telone II technical formulation blank: E1912-54
   Telone II technical formulation: TSN105019

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 3-chloro-1, 5-hexadiene reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate.

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 3-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 g) of technical 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 38.1 mg 3-chloro-1, 5-hexadiene to 3 mL Telone II blank.

   Recovery samples containing 3-chloro-1, 5-hexadiene were prepared for Telone II by weighing 400 uL aliquots of the Telone II (TSN105019) into glass autosampler vials (Table I). Volumetric aliquots of 3-chloro-1, 5-hexadiene were then added to each sample via the stock solution of 3-chloro-1, 5-hexadiene in Telone II, and contents were prepared for GC analysis by adding 600 uL of ethyl acetate.

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 (fortified with 3-chloro-1, 5-hexadiene to ensure technical formulations were in the appropriate range for analysis) into 2 ounce jars 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:

Instrument:	Hewlett Packard 6890 or equivalent
Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min Carrier gas = helium
Injection volume:	2 µL
Run Time:	30.9 minutes
Retention Time:	3-chloro-1,5-hexadiene ~15.3 minutes

2. Approximate time to prepare and analyze sample: 1 hour

F. Methods of Calculation

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

1. Calculation of the response factor for 3-chloro-1, 5-hexadiene in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(3\text{-chloro-1,5-hexadiene})} \times 50\ \text{mL}}$$

where:
- RF = Response factor for 3-chloro-1, 5-hexadiene
- mg reference std = Weight of 3-chloro-1, 5-hexadiene reference standard in calibration solution, mg
- P = Purity of reference standard, expressed as a fraction
- $\text{Area}_{(3\text{-chloro-1, 5-hexadiene})}$ = Peak area for 3-chloro-1, 5-hexadiene in calibration solution

2. Calculation of the weight % of 3-chloro-1, 5-hexadiene in the sample:

$$\text{Weight \%} = \frac{\text{Area}_{(3\text{-chloro-1,5-hexadiene})} \times RF}{\text{Sample wt}} \times DF \times 100$$

where: Weight % = Weight % of 3-chloro-1, 5-hexadiene in the sample
$Area_{(3\text{-chloro-1, 5-hexadiene})}$ = Peak area for 3-chloro-1,5-hexadiene in the sample solution
RF = Response factor calculated for 3-chloro-1,5-hexadiene
Sample wt = Weight of sample in mg
DF = Dilution factor (5 mL for a sample prepared as described)

IV. RESULTS AND DISCUSSION

A. Linearity

The linearity for 3-chloro-1, 5-hexadiene was evaluated using the GC conditions used for this study. A linear relationship between peak area and concentration ($r^2 = 0.9997$) was noted for 3-chloro-1, 5-hexadiene from 0.045 wt. % to 0.934 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. Recovery data were obtained over the range of 0.045 wt. % to 0.934 wt. % 3-chloro-1, 5-hexadiene in Telone II.

The recovery for 3-chloro-1, 5-hexadiene in Telone II ranged from 72 % to 104 %, with an average recovery of 94.6%, and a relative standard deviation of 10.9 %. 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 Tables II and numbers obtained if the values are calculated by hand.

C. Method Precision

The precision of the method was evaluated by analysis of a 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 2.89 % at an average concentration of 0.068 wt. % 3-chloro-1, 5-hexadiene in Telone II. The Horwitz RSD_r value was calculated to be 4.03 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 of 3-chloro-1, 5-hexadiene for all five injections of Telone II was 2.99%.

E. Solution Stability

The solution stability was determined by analyzing sample solutions prepared for the day one precision study 3 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 three days after initial analysis were not statistically equivalent to the original results for Telone II. It is recommended that samples and standards be prepared fresh and used on for analysis the same day they are prepared.

F. Interferences

No interferences were detected for the ethyl acetate solvent, technical formulation inert ingredients, or 3-chloro-1, 5-hexadiene. Chromatograms of a solvent blank, 3-chloro-1,5-hexadiene standard and technical formulation blank for Telone II are shown in Figure 4.

G. Ruggedness

The method ruggedness was tested by increasing the flow rate from the nominal 1.9 mL/min to 2.4 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 using the precision samples.(five samples at 0.065 % (wt/wt)). In addition to precision being proven at the LOQ level, acceptable recovery was demonstrated at 0.071% and 0.048% during the recovery phase of the study which covers the range of the LOQ level.
Limit of detection was determined by reviewing the recovery samples prepared at 0.045 % (wt/wt) and determining that the 3-chloro-1,5-hexadiene had a greater than three times signal to noise ratio, as shown in Figure 6.

V. CONCLUSIONS

This method is applicable to the determination of 3-chloro-1, 5-hexadiene in Telone II technical formulations over the range of 0.045 wt. % to 0.934 wt. % 3-chloro-1, 5-hexadiene in Telone II. The precision, recovery and linearity data have shown this method to be acceptable for the determination of 3-chloro-1, 5-hexadiene in Telone II technical 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, and accurately reflects what was done during the study.

The statistical methods used were means, standard deviations, relative standard deviations, regression analysis, Horwitz 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 3-chloro-1,5-hexadiene added (TSN104959), mg	Weight of Telone II (E1912-54) sample plus weight of spike, mg	Volume of spike solution added (uL)	3-chloro-1, 5-hexadiene Wt. %
Recovery 1	4.8	516.5	N/A	0.934
Recovery 2	1.2	636.3	100	0.190
Recovery 3	0.9	607.8	74	0.147
Recovery 4	0.6	581.1	50	0.104
Recovery 5	0.5	563.7	40	0.086
Recovery 6	0.4	556.7	30	0.065
Recovery 7	0.2	539.7	20	0.045

Weight of 3-chloro-1, 5-hexadiene added to sample =

$$\frac{\text{Wt. of spike solution used (mg) x Purity (95\%) x vol of spike added (mL)}}{\text{Total volume of solution (mL)}}$$

3-chloro-1, 5-hexadiene wt. % =

$$\frac{\text{Weight 3 - chloro - 1, 5 - hexadiene added (TSN104959), mg}}{\text{Weight of Telone II (E1912 - 54) sample plus weight of spike, mg}} \times 100$$

Table II. Recovery Data for 3-chloro-1, 5-hexadiene in Telone II Technical Formulation

Sample	3-chloro-1, 5-hexadiene wt % added	3-chloro-1, 5-hexadiene wt % found	Recovery %
Recovery 1	0.934	0.968	104
Recovery 2	0.190	0.175	92
Recovery 3	0.147	0.139	95
Recovery 4	0.104	0.074	72
Recovery 5	0.086	0.083	97
Recovery 6	0.065	0.066	101
Recovery 7	0.045	0.046	102
		Average	94.6
		Std. Dev.	10.9
		R.S.D.	11.5

Table III. Method Precision Data for 3-chloro-1, 5-hexadiene in Telone II Technical Formulation

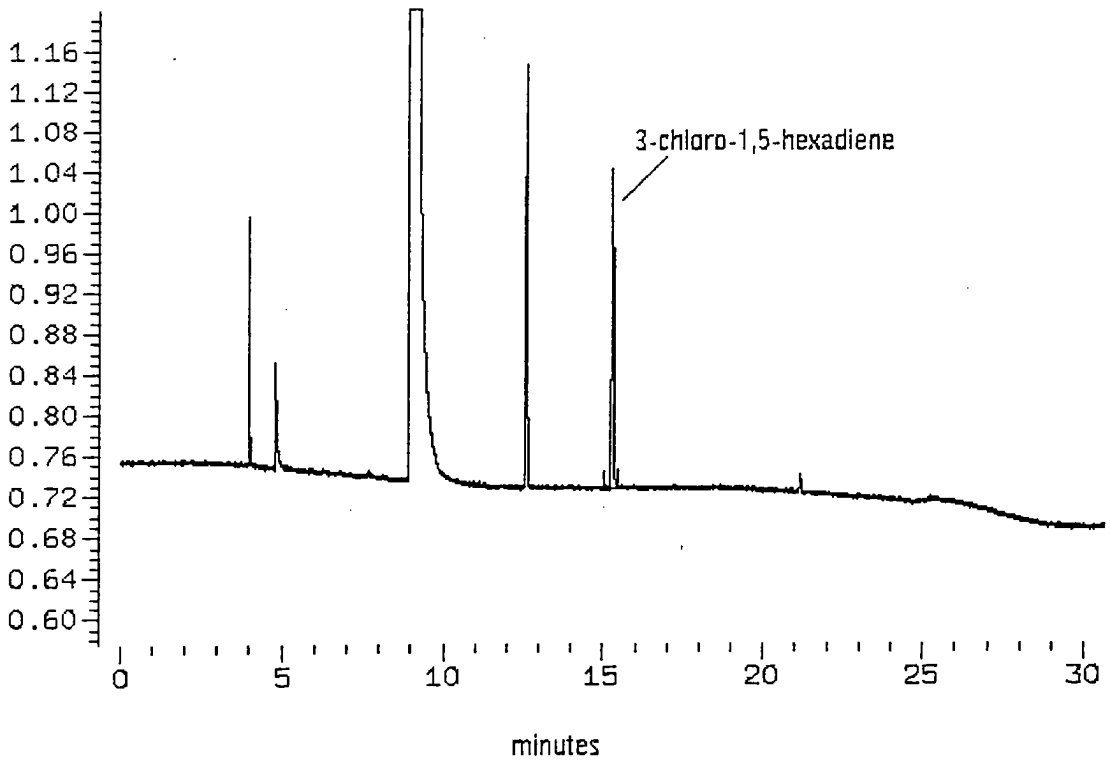
Sample	Wt. % 3-chloro-1, 5-hexadiene
tsn105019-Prec 1-1	0.0661
tsn105019-Prec 1-2	0.0653
tsn105019-Prec 1-3	0.0661
tsn105019-Prec 1-4	0.0680
tsn105019-Prec 1-5	0.0681
tsn105019-Prec 2-1	0.0697
tsn105019-Prec 2-2	0.0694
tsn105019-Prec 2-3	0.0698
tsn105019-Prec 2-4	0.0676
tsn105019-Prec 2-5	0.0659
Average	0.0676
Std. Dev.	0.001950
R.S.D.	2.89
Horwitz RSD_R	6.01
Horwitz RSD_r	4.03
RSD < Horwitz RSD_r?	Acceptable

Table IV. System Precision Data for 3-chloro-1, 5-hexadiene

Sample	Peak Area
Telone II	
Injection #1	39459
Injection #2	42257
Injection #3	41054
Injection #4	41666
Injection #5	42591
Average	41405
Std. Dev.	1236.0
R.S.D. (%)	2.99

VII. FIGURES

Figure 1. Chromatogram of a Calibration Solution *

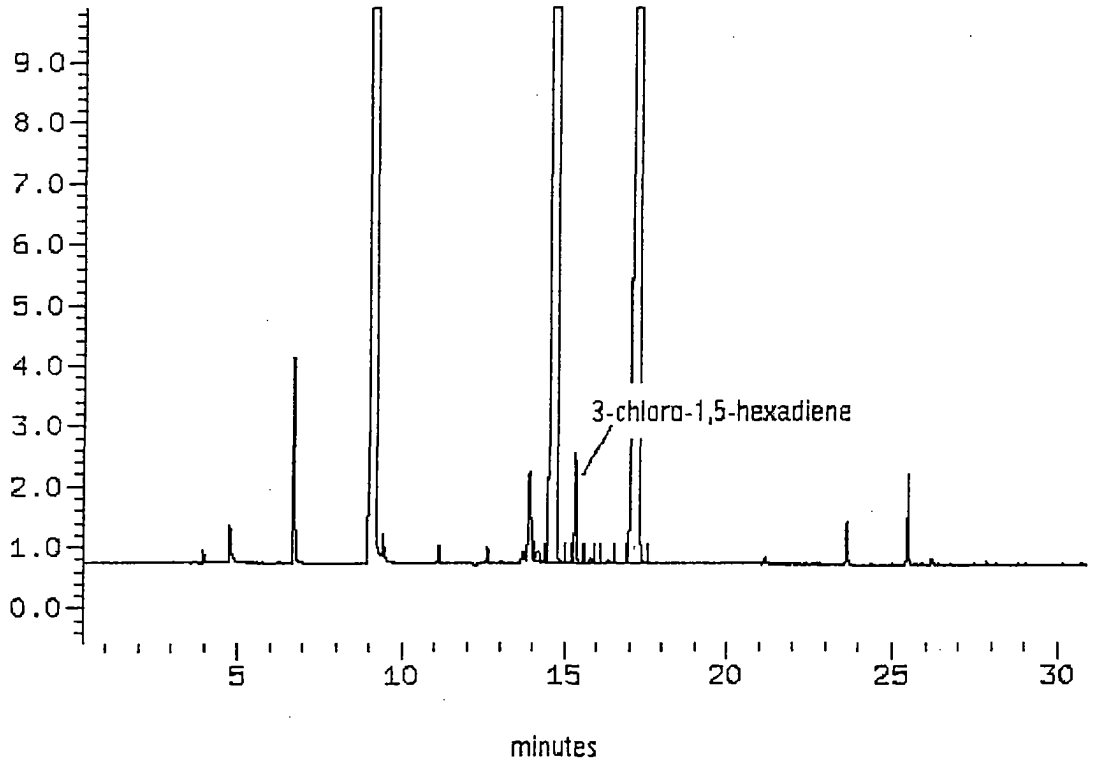


Datafile: /var/chem/167gc055.i/das-am-05-009.p/recovery_interfer_precDay1.b/seq037_A.d

Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30.9 minutes

* Note that peak at approximately 12.5 minutes corresponds to another standard that is not being evaluated in this study.

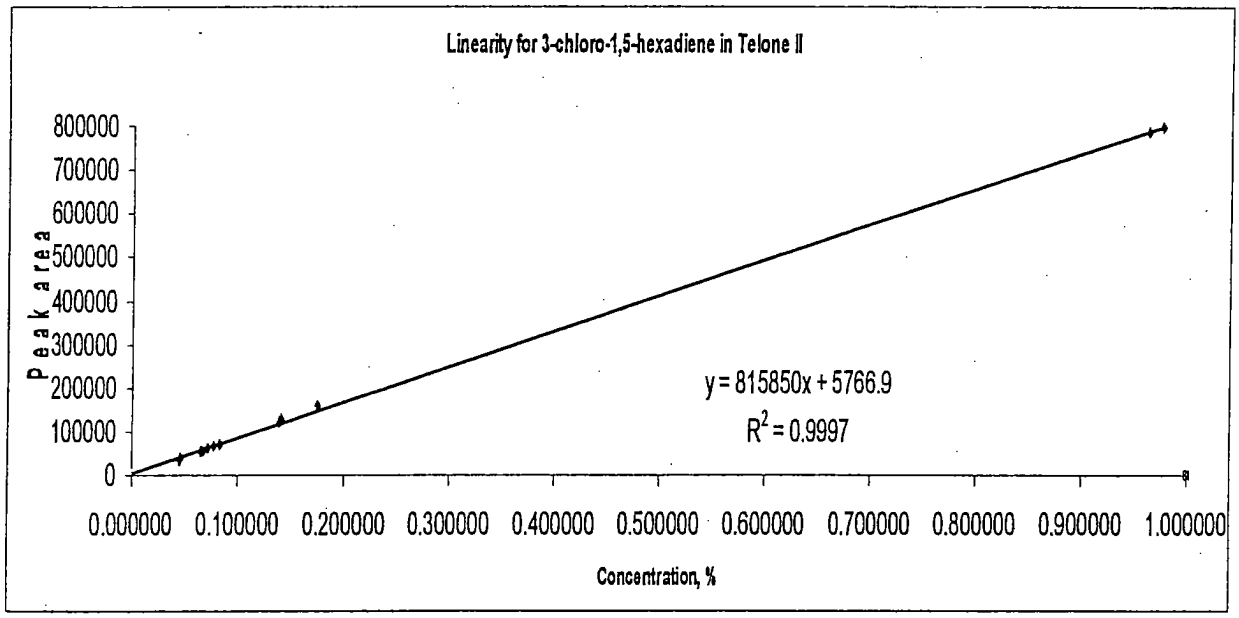
Figure 2. Chromatogram of a Sample Solution of Telone II Technical Formulation



Datafile: /var/chem/167gc055.i/das-am-05-009.p/recovery_interfer_precDay1.b/seq008_A.d

Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30 minutes

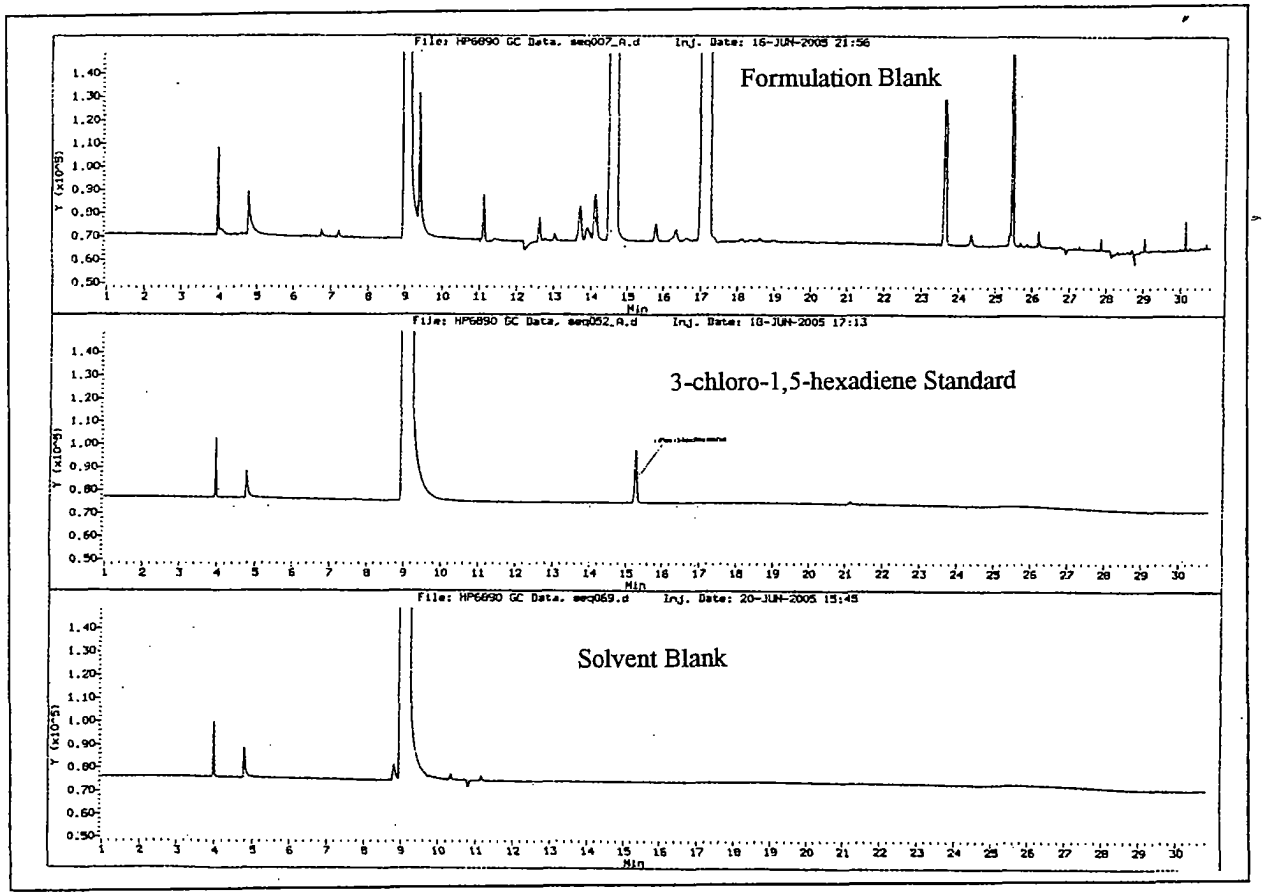
Figure 3. Linearity Plot for 3-chloro-1, 5-hexadiene



Concentration, Wt. %	Peak area*
0.934	794556
0.190	160588**
0.147	125242
0.104	65437
0.086	71337
0.065	56601
0.045	38257

* Average of two injections.
** One injection

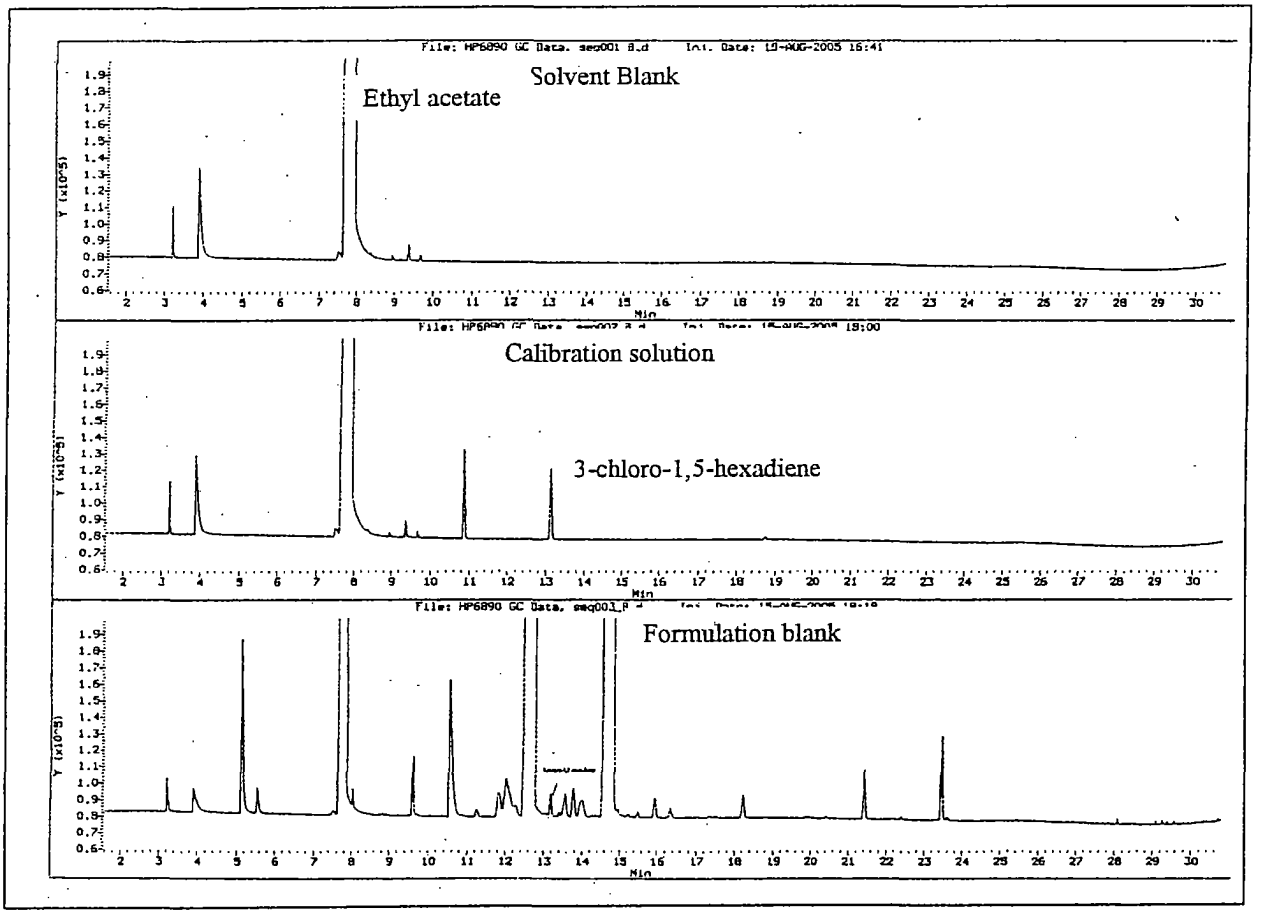
Figure 4. Chromatograms of Telone II Technical Formulation Blank, 3-chloro-1,5-hexadiene Standard, and Solvent Blank



Datafile: /var/chem/167gc055.i/das-am-05-009.p/recovery_interfer_precDay1.b/seq007_A, 052_A and 069.d

Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Carrier Flow: 72 mL/min of helium (constant flow) Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30.9 minutes

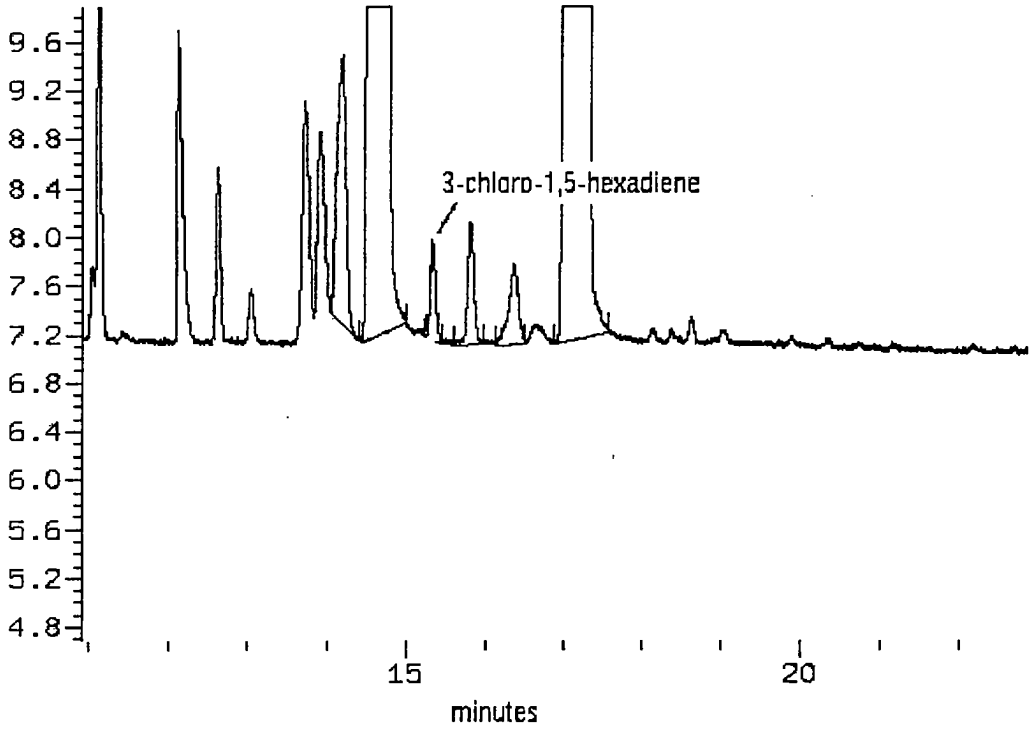
Figure 5. Effect of Flow Rate on Retention Times



Datafile: /var/chem/167gc055.i/das-am-05-009.p/ruggedness.b/seq001_B, 002_B and 003_B.d

Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 2.4 mL/min
Injection volume:	2 µL
Run Time:	30.9 minutes

Figure 6. LOD Chromatogram



Datafile: 167gc055.i/das-am-05-009.p/recovery_interfer_precDay1.b/seq014_A.d

Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30 minutes

VIII. APPENDIX

Analytical Method Summary

A. Preparation of calibration solution:

Prepare stock calibration solutions by weighing ~ 50 mg of 3-chloro-1, 5-hexadiene reference standard into a 50 mL volumetric flask and fill to the mark with ethyl acetate.

B. Preparation of sample solution:

Add 2 mL (~ 2 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 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min Carrier gas: Helium
Injection volume:	2 µL
Run Time:	30.9 minutes

Approximate Retention Time: 3-chloro-1, 5-hexadiene 15.3 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 3-chloro-1, 5-hexadiene in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(3\text{-chloro-1, 5-hexadiene})} \times 50\ \text{mL}}$$

where:
RF = Response factor for 3-chloro-1, 5-hexadiene
mg reference std = Weight of 3-chloro-1, 5-hexadiene reference standard in calibration solution, mg
P = Purity of reference standard, expressed as a fraction
$\text{Area}_{(3\text{-chloro-1, 5-hexadiene})}$ = Peak area for 3-chloro-1, 5-hexadine in calibration solution

2. Calculation of the weight % of 3-chloro-1, 5-hexadiene in the sample:

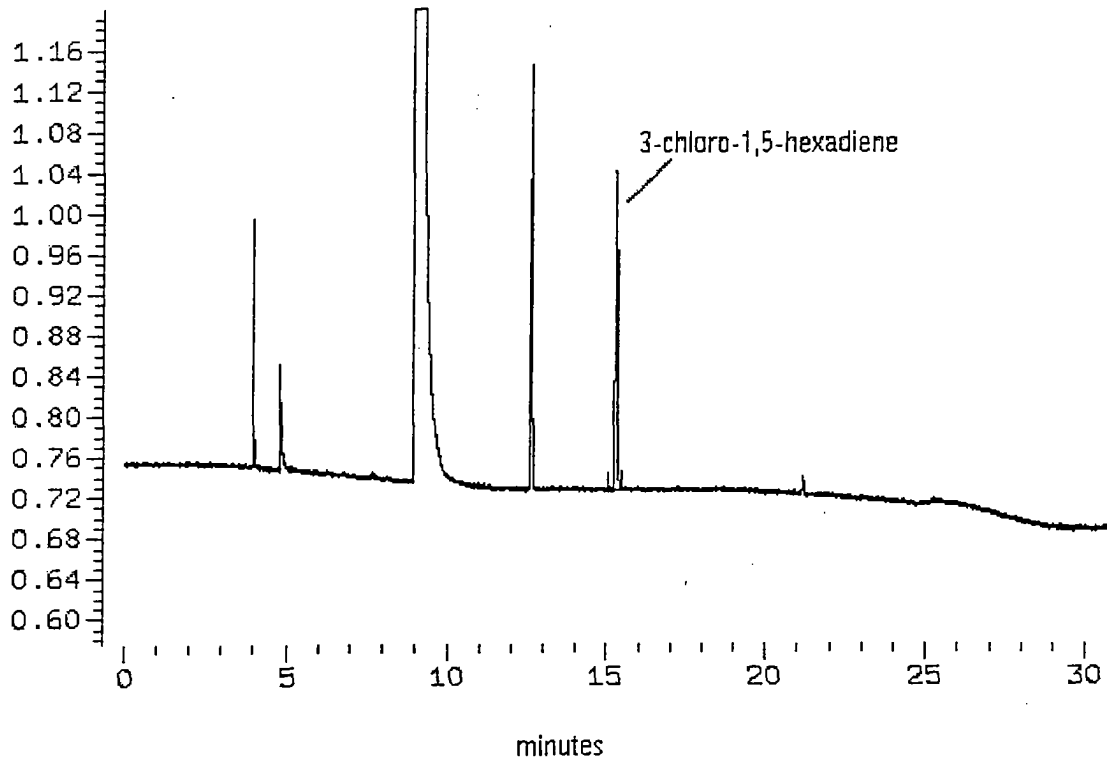
$$\text{Weight \%} = \frac{\text{Area}_{(3\text{-chloro-1, 5-hexadiene})} \times RF}{\text{Sample wt}} \times 5\ \text{mL} \times 100\%$$

where:
Weight % = Weight % of 3-chloro-1, 5-hexadiene in the sample
$\text{Area}_{(3\text{-chloro-1, 5-hexadiene})}$ = Peak area for 3-chloro-1, 5-hexadiene in the sample solution
RF = Response factor calculated for 3-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.

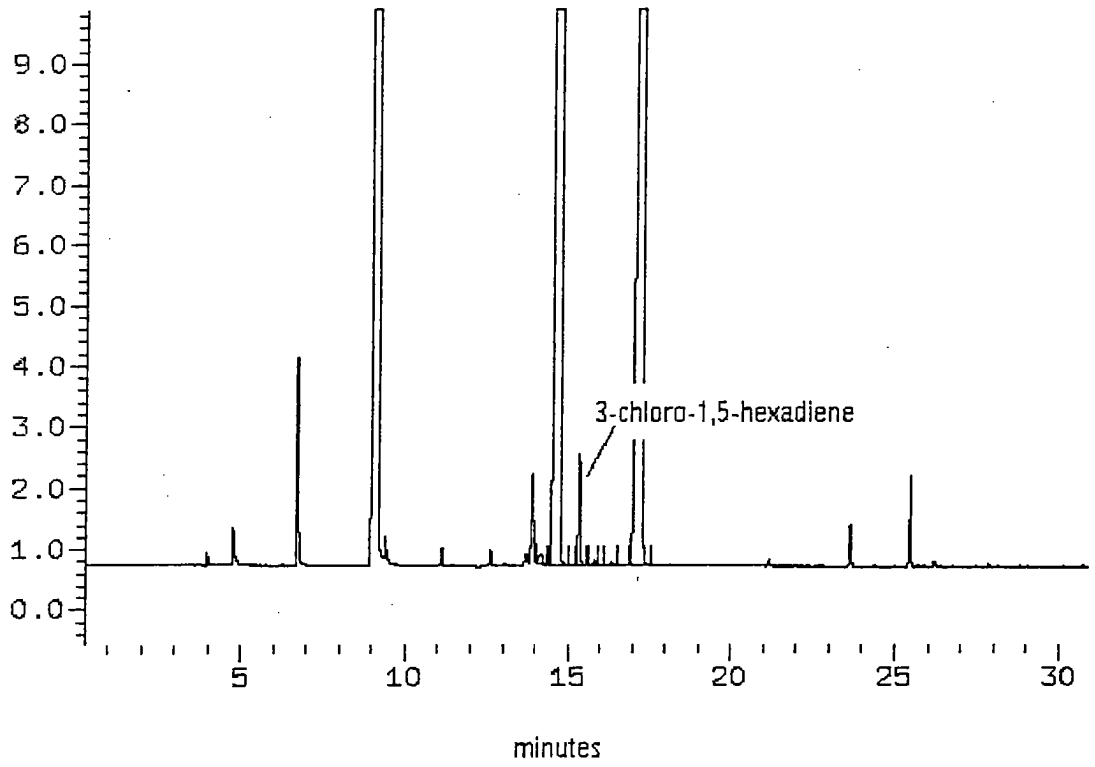
Chromatogram of a Calibration Solution *



Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30.9 minutes

* Note that peak at approximately 12.5 minutes corresponds to another standard that is not being evaluated in this study.

Chromatogram of a Sample Solution of Telone II Technical Formulation



Column:	DB-1701 60 m x 0.32 mm x 1µm
Oven Program:	40°C hold for 2 minutes, 5°C/minute to 80°C, hold for 7.5 minutes 5°C/minute to 110°C, hold for 1 minutes 25°C/minute to 270°C, hold for 0 minutes
Injection port:	Split at 150°C with a ratio of 38:1
Detector:	TCD at 280°C
Flows:	Reference Flow: 15 mL/min Make-up Flow: 10 mL/min Purge Flow: 1.2 mL/min Column Flow: 1.9 mL/min
Injection volume:	2 µL
Run Time:	30.9 minutes