

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 Impurities in Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

AMENDED REPORT

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800;
European Commission Regulation (EU) No 544/2011 implementing Regulation (EC) No
1107/2009; and
SANCO/3030/99 rev.4

STUDY DIRECTOR/AUTHOR

Brandi Thomas

STUDY COMPLETED ON

05-June-2014

Amended Report Date: 30-January-2015

PERFORMING LABORATORY

Dow AgroSciences LLC
9330 Zionsville Road
Indianapolis, Indiana 46268

LABORATORY STUDY ID

DAS-AM-G-13-48

SUMMARY

NA-AM-98-081.00, a gas chromatographic (GC) method, was validated for the determination of additional impurities in Telone II technical grade material. The accuracy, precision, and linearity of the method have been shown to be acceptable.

STUDY TITLE

Analytical Method and Validation for the Determination of Impurities in Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

AMENDED REPORT

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800;
European Commission Regulation (EU) No 544/2011 implementing Regulation (EC) No 1107/2009; and
SANCO/3030/99 rev.4

STUDY DIRECTOR/AUTHOR

Brandi Thomas
317-337-4704
bdthomas@dow.com

STUDY COMPLETED ON
05-June-2014

Amended Report Date: 30-January-2015

PERFORMING LABORATORY

Dow AgroSciences LLC
9330 Zionsville Road
Indianapolis, Indiana 46268

LABORATORY STUDY ID

DAS-AM-G-13-48

STATEMENT OF DATA CONFIDENTIALITY CLAIMS

Information claimed confidential has been removed to a confidential attachment.

Company: Dow AgroSciences LLC

Company Agent: Jim Baxter

Title: Regulatory Manager

Signature: J.P. Baxter

Date: 15 May 2014

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

STUDY TITLE: Analytical Method and Validation for the Determination of Impurities in
Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

Study Initiation Date: 22-October-2013

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

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

J.P.Baxter
Jim Baxter
Sponsor
Dow AgroSciences LLC

28 January 2015
Date

J.P. Baxter
Jim Baxter
Submitter
Dow AgroSciences LLC

28 January 2015
Date

Brandi Thomas
Brandi Thomas
Study Director
Dow AgroSciences LLC

30-January-2015
* Amended Report Date

***This report has been amended. The original report was completed and signed on 05 June 2014. The study director's signature and date reflect the date of the report amendment.**

**Dow AgroSciences Quality Assurance Unit
Good Laboratory Practice Statement Page**

Study ID: DAS-AM-G-13-48

Title: Analytical Method and Validation for the Determination of Impurities in Telone II
Technical Grade Material- Method NA-AM-98-081.00 Extension

Study Initiation Date: 22-Oct-2013

Study Completion Date: 5-Jun-2014

***Amended Report Date:** 30-Jan-2015

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
18-Oct-2013	22-Oct-2013	Protocol Review
20-Jan-2014	24-Jan-2014	Sample Analysis (Precision)
24-Jan-2014	24-Jan-2014	Sample Analysis (Precision Repeat)
22, 23, 24, 27-May-2014	3-Jun-2014	Report and Raw Data Review; Test Substance Container verification
29-Jan-2015	30-Jan-2015	Incorporation of LOQ Data Review

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.


Tracey Froggatt
Dow AgroSciences, Quality Assurance

Date

30-Jan-2015

* The original report was completed and signed on 5-Jun-2014. The new signature and date reflect the date the report was amended.

STUDY TITLE: Analytical Method and Validation for the Determination of Impurities in Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

Summary of amendment changes:

- Changes were made on summary page 1R1 of 2, title page 1R1 of 2, pages 3R1, page 4R1, and confidential attachment page 1R1 of 52 in order to meet SOP requirements.
- This page was added to describe the changes
- Confidential Attachment, Page 5R1: Summary table was updated with linearity including the LOQ samples.
- Confidential Attachment, Page 10R1: Linearity sample prep was updated to include LOQ samples.
- Confidential Attachment, Page 15R1: Linearity results was updated to include LOQ samples.
- Confidential Attachment, Page 40R1 through 45R1 (Figures 3-8): Updated Linearity figures.

Reason for Amendment:

LOQ data was added to the linear range.

Impact on Study:

This has a positive impact on the study as the LOQ is included in the linear range. No additional sample prep, data collection, or processing in EZChrom was required. The existing data was simply presented differently in order to include the LOQ in the linear range.

Signatures:

Brandi Thomas
Brandi Thomas, Study Director

28-January-2015
Date

Annette Brown
Annette Brown, Manager

29-January-2015
Date

TJaggard.
Quality Assurance

30-Jun-2015
Date

SIGNATURES

Brandi Thomas

Brandi Thomas
Study Director
Dow AgroSciences LLC

16-May-2014

Date

Todd Kajdan

Todd Kajdan
Reviewer
Dow AgroSciences LLC

19-May-2014

Date

Jon Sauer

Jon Sauer
Manager
Dow AgroSciences LLC

19-May-2014

Date

STUDY PERSONNEL

Study Title: Analytical Method and Validation for the Determination of Impurities in Telone II
Technical Grade Material – Method NA-AM-98-081.00 Extension

Study Director / Analyst: Brandi Thomas

TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION.....	8
II. CONFIDENTIAL ATTACHMENT	
ANALYTICAL METHOD AND VALIDATION FOR THE DETERMINATION OF IMPURITIES IN TELONE II TECHNICAL GRADE MATERIAL – METHOD NA-AM- 98-081.00 EXTENSION	9

I. INTRODUCTION

NA-AM-98-081.00, a gas chromatographic (GC) method, was validated for the determination of additional impurities in Telone II technical grade material. The accuracy, precision, and linearity of the method have been shown to be acceptable.

This report includes the OPPTS 830.1800 Enforcement Analytical Method for these additional impurities. The details for the analysis of the impurities in Telone II TGAI are included in the Confidential Attachment of this report.

Study Title: Analytical Method and Validation for the Determination of Impurities in Telone II
Technical Grade Material – Method NA-AM-98-081.00 Extension

Guidelines Reference OPPTS 830.1800 Enforcement Analytical Method for Impurities
(Information found in the Confidential Attachment under Cross Reference Number 1)

CONFIDENTIAL ATTACHMENT

STUDY TITLE

Analytical Method and Validation for the Determination of Impurities in Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

AMENDED REPORT

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800;
European Commission Regulation (EU) No 544/2011 implementing Regulation (EC) No
1107/2009; and
SANCO/3030/99 rev.4

STUDY DIRECTOR/AUTHOR

Brandi Thomas
317-337-4704
bdthomas@dow.com

STUDY COMPLETED ON
05-June-2014

Amended Report Date: 30-January-2015

PERFORMING LABORATORY

Dow AgroSciences LLC
9330 Zionsville Road
Indianapolis, Indiana 46268

LABORATORY STUDY ID
DAS-AM-G-13-48

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.

<u>Page</u>	<u>Reason for Deletion</u>	<u>FIFRA Reference</u>
9	Process Impurities Identified	§10(d)(1)(A)

TABLE OF CONTENTS – Confidential Attachment

	<u>Page</u>
C_I. ABSTRACT.....	5
C_II. INTRODUCTION.....	6
A. Scope.....	6
B. Principle	6
C_III. MATERIALS AND METHODS	7
A. Equipment	7
B. Reagents and Standards.....	7
C. Safety.....	9
D. Analytical Procedures	9
E. Instrumentation and LC Conditions	11
F. Methods of Calculation	12
C_IV. RESULTS AND DISCUSSION	15
A. Precision.....	15
B. Recovery	15
C. Linearity	15
D. Stability.....	15
E. Interferences	16
F. Method Ruggedness	16
G. LOD/LOQ	17
C_V. CONCLUSIONS	18
C_VI. TABLES.....	19
Table I. Method Precision Data for Impurities in Telone II TGAI	19
Table II. System Precision Data for Impurities in Telone II TGAI.....	20
Table III. Preparation of Recovery Samples (Impurities in Telone II TGAI)	21
Table IV. Equivalent Weight % in Recovery Samples (Impurities in Telone II TGAI)	22
Table V. Recovery Data for 3,3-dichloro-1-propene (M, X191117)	23
Table VI. Recovery Data for 4-chloro-4-methyl-1-pentene (N, X11656876).....	24
Table VII. Recovery Data for 5-chloro-1-hexene (O, X11656878)	25
Table VIII. Recovery Data for (Z)-1-chloro-1,5-hexadiene (P, X11661609)	26
Table IX. Recovery Data for 3-chloro-1-methylcyclopentane (Q, X11726086).....	27
Table X. Recovery Data for 1-chloro-1-methylcyclopentane (R, X11726078)	28
Table XI. Stability Data for Impurities in Telone II TGAI	29
Table XII. Interferences Data	30
Table XII. Interferences Data, cont.	31
Table XIII. Limits of Quantitation for 3,3-dichloropropane (M)	32
Table XIV. Limits of Detection for for 3,3-dichloropropane (M).....	32
Table XV. Limits of Quantitation for 4-chloro-4-methyl-1-pentene (N)	33
Table XVI. Limits of Detection for 4-chloro-4-methyl-1-pentene (N)	33

Table XVII. Limits of Quantitation for 5-chloro-1-hexene (O)	34
Table XVIII. Limits of Detection for 5-chloro-1-hexene (O)	34
Table XIX. Limits of Quantitation for (Z)-1-Chloro-1,5-hexadiene (P)	35
Table XX. Limits of Detection for (Z)-1-Chloro-1,5-hexadiene (P)	35
Table XXI. Limits of Quantitation for 3-chloro-1-methylcyclopentane (Q).....	36
Table XXII. Limits of Detection for 3-chloro-1-methylcyclopentane (Q).....	36
Table XXIII. Limits of Quantitation for 1-chloro-1-methylcyclopentane (R)	37
Table XXIV. Limits of Detection for 1-chloro-1-methylcyclopentane (R)	37
C_VII. FIGURES.....	38
Figure 1. Representative Chromatogram of the Standard Solution.....	38
Figure 2. Representative Chromatogram of a TGAI Solution.....	39
Figure 3. Linearity of 3,3-dichloro-1-propene (M, X191117).....	40
Figure 4. Linearity of 4-chloro-4-methyl-1-pentene (N, X11656876)	41
Figure 5. Linearity of 5-chloro-1-hexene (O, X11656878)	42
Figure 6. Linearity of (Z)-1-chloro-1,5-hexadiene (P, X11661609)	43
Figure 7. Linearity of 3-chloro-1-methylcyclopentane (Q, X11726086)	44
Figure 8. Linearity of 1-chloro-1-methylcyclopentane (R, X11726078)	45
Figure 9. Chromatograms of Impurities from Interferences Evaluation (Region 1, 10-15 min).....	46
Figure 10. Chromatograms of Impurities from Interferences Evaluation (Region 2, 15-20 min).....	47
Figure 11. Chromatograms of Impurities from Interferences Evaluation (Region 3, 20-30.9 min).....	48
Figure 12. Chromatograms of Telone II TGAI from Ruggedness Evaluation	49
APPENDIX I. ANALYTICAL METHOD SUMMARY	50

C_I. ABSTRACT

This report describes the validation extension of NA-AM-98-081.00, a gas chromatographic (GC) method for the determination of impurities in Telone II technical grade active ingredient (TGAII) for additional impurities. NA-AM-98-081.00 consists of a gas chromatographic (GC) system with a DB-1701 60 m x 0.32 mm x 1 μ m column and a thermoconductivity detector (TCD). Concentrations were determined using external standard calibration.

The results for range of precision, recovery, linearity, LOQ and LOD are presented below.

Parameter		3,3-dichloro-1-propene (M, X191117)	4-chloro-4-methyl-1-pentene (N, X11656876)	5-chloro-1-hexene (O, X11656878)	(Z)-1-chloro-1,5-hexadiene (P, X11661609)	3-chloro-1-methyl cyclopentane (Q, X11726086)	1-chloro-1-methyl cyclopentane (R, X11726078)
Method Precision (n=10)	Avg wt %	0.03	0.03	0.09	0.04	0.02	0.003
	% RSD	2.8	1.9	2.3	2.5	3.9	15.2
Accuracy (n = 7)	wt % range	0.009 - 0.858	0.008 - 0.825	0.009 - 0.877	0.008 - 0.824	0.009 - 0.854	0.009 - 0.881
	Recovery range	88.9 - 100.2	93.8 - 106.3	88.9 - 106.8	103.7 - 111.0	83.3 - 100.6	88.9 - 97.7
	Avg Recovery	98	99	99	105	98	95
Linearity (n=7)	mg/mL range	0.027 - 4.175	0.022 - 4.017	0.029 - 4.271	0.030 - 4.014	0.040 - 4.156	0.015 - 4.289
	Equivalent Wt.%	0.006 - 0.858	0.005 - 0.825	0.007 - 0.877	0.007 - 0.824	0.009 - 0.854	0.004 - 0.881
	Coefficient of Determination (r^2)	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
LOQ	Avg wt %	0.006	0.005	0.007	0.007	0.009	0.004
	% RSD	0.0	8.4	16.5	7.7	6.4	14.1
	Avg Recovery	100	100	133	117	113	N/A*
LOD	Avg wt %	0.001	0.001	0.004	0.001	0.005	0.002

* Not applicable because 1-chloro-1-methylpentane was present in the test system at the LOQ (no spiking or dilution needed)

C_II. INTRODUCTION

A. Scope

The gas chromatography method described in this report is applicable for the determination of impurities in Telone II technical grade active ingredient (TGAI) and is an extension of method NA-AM-98-081.00.

This report reflects what was actually performed during the course of this study and includes all amendments and/or deviations to the protocol.

B. Principle

Sample preparation involves adding 2 mL of Telone II to a 5 mL volumetric flask and diluting to volume in ethyl acetate. Sample solutions are then analyzed on a gas chromatographic system using a DB-1701 60 m x 0.32 mm x 1 μ m column and thermocconductivity detection. Sample quantitation is by external standard calculations using peak areas.

C_III. MATERIALS AND METHODS

A. Equipment

1. Analytical balance: capable of measuring to 0.1 mg: Mettler XP205DR
2. Gas Chromatographic System: Agilent model 6890N equipped with a thermoconductivity detector and autosampler
3. Data acquisition and processing system: Agilent EZChrom Elite data system
4. Column: DB-1701 60 m x 0.32 mm x 1 μ m
5. Autosampler vials and caps: 2 mL with screw caps
6. Sonicator and shaker
7. Miscellaneous laboratory glassware and syringes
8. Automatic pipets

B. Reagents and Standards

1. Test Substances

Test substances were received from the Dow AgroSciences Test Substance Coordinator, Indianapolis, Indiana. Additional information for the test substances were as follows:

Common Name (X#)	Chemical Name	Test Substance / Lot Number	Purity	Reference	Recertification Date
M (X191117)	3,3-dichloro-1-propene	TSN030579-0001	97%	FAPC12-000717	22-Aug-2015
O (X11656878)	5-chloro-1-hexene	TSN106329	99%	FAPC13-000407	23-Aug-2015
P (X11661609)	(Z)-1-chloro-1,5-hexadiene	TSN304464	97%	FAPC13-000001	21-Aug-2015
N (X11656876)	4-chloro-4-methyl-1-pentene	TSN303759	96%	FAPC12-000628	12-Aug-2014
R (X11726078)	1-chloro-1-methylcyclopentane	TSN303032	100%	FAPC12-000633	12-Aug-2014
Q (X11726086)	3-chloro-1-methylcyclopentane	TSN303341	97%	FAPC12-000626	12-Aug-2014

The following test substances were used for the evaluation of interferences only:

Common Name (X#)	Chemical Name	Test Substance / Lot Number	Purity	Reference	Recertification Date
A	1,2-Dichloropropane	AGR277102	99%	FAPC13-000368	12-Aug-2017
B (X12314033)	2-Chloro-2-methylpentane	AGR238091	97%	FAPC12-000686	30-Aug-2017
C (X11719553)	2-Chloro-1,5-hexadiene	TSN030278-0001	96%	FAPC12-000245	20-Aug-2014

D (X139475)	2-Chloro-4-methylpentane	TSN106505	98%	FAPC12-000304	23-Aug-2014
E (X11842830)	2-Chloro-2,3-dimethylbutane	TSN028018-0001	100%	FAPC13-000375	12-Aug-2017
F (X11373675)	3-chloro-1,5-hexadiene	TSN303599	98%	FAPC12-00630	12-Aug-2014
G (X11742495)	3-Chloro-2-methylpentane	TSN106504	79%	FAPC12-000296	23-Aug-2016
H	1,3-dichloropropane	AGR238090	99.5%	FAPC12-000685	30-Aug-2017
I (X166920)	1,2,2-trichloropropane	TSN301451	97%	FAPC13-000365	02-Aug-2015
J	Cis-1,3,3-trichloropropene	AGR238088	94%	FAPC13-000366	03-Aug-2017
K-cis K-trans (X11731281)	Cis /Trans-2-chloro-3(chloromethyl)oxirane	TSN106513	83%	FAPC12-000306	24-Aug-2014
L	Trans-1,3,3-trichloropropene	AGR238086	86.2%	FAPC11-000021	23-Aug-2015

2. Reference Substances

The reference substances were obtained from the Test Substance Coordinator for Dow AgroSciences LLC. Additional information on the reference substances are presented below:

Common Name (X#)	Chemical Name	Test Substance / Lot Number	Purity	Reference	Recertification Date
M (X191117)	3,3-dichloro-1-propene	TSN030579-0001	97%	FAPC12-000717	22-Aug-2015
O (X11656878)	5-chloro-1-hexene	TSN106329	99%	FAPC13-000407	23-Aug-2015
P (X11661609)	(Z)-1-chloro-1,5-hexadiene	TSN304464	97%	FAPC13-000001	21-Aug-2015
N (X11656876)	4-chloro-4-methyl-1-pentene	TSN303759	96%	FAPC12-000628	12-Aug-2014
R (X11726078)	1-chloro-1-methylcyclopentane	TSN303032	100%	FAPC12-000633	12-Aug-2014
Q (X11726086)	3-chloro-1-methylcyclopentane	TSN303341	97%	FAPC12-000626	12-Aug-2014

3. Test Systems

Common Name (X#)	Chemical Name	Test Substance / Lot Number	Purity	Reference	Recertification Date
Telone II (X191573)	1,3-dichloropropene technical	TSN106191	98.1% 1,3-dichloropropene	FAPC12-000714	23-Jan-2015

4. Ethyl Acetate: EMD (min 99.9%) and Sigma Aldrich (\geq 99.5%, used for interferences only)
5. Epoxidised Soybean Oil (ESO): Akros

C. **Safety**

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

D. **Analytical Procedures**

1. Preparation of calibration solutions:

A solution of the reference substances was prepared in duplicate by accurately weighing approximately 50 mg into a 50-mL volumetric flask (record weight to the nearest 0.1 or 0.01 mg). The flask was diluted to the mark with ethyl acetate to afford a mixed standard containing all impurities.

2. Calibration procedure:

The calibration solutions were injected at least twice into the gas chromatograph, using the conditions summarized in Section C_III.E, and the response factors were calculated using the equations given in Section C_III.F. The average of the response factors was used for calibration.

3. Sample Preparation and Analysis:

Telone II (2 mLs) was introduced into a 5 mL volumetric flask and diluted to volume with ethyl acetate. Weights were recorded to the nearest 0.1 or 0.01 mg. Samples were mixed well and analyzed using the conditions given in Section C_III.E.

4. Preparation of Precision samples:

Five precision samples were prepared as detailed in section C_III.D.3 and analyzed on each of two days to evaluate method precision. Single injections of the precision samples were made. The first five precision samples were re-analyzed 3 days after preparation to evaluate stability of the analytes in the prepared samples. One of the precision samples, (TSN106191-A2), was used to evaluate system precision by injecting a total of five times.

5. Preparation of Recovery samples:

Seven recovery (accuracy) samples were prepared by adding varying amounts of the test substances (3,3-dichloro-1-propene, 4-chloro-4-methyl-1-pentene, 5-chloro-1-hexene, (Z)-1-chloro-1,5-hexadiene, 3-chloro-1-methylcyclopentane, and 1-chloro-1-methylcyclopentane) to the test system (TSN106191) in a 5 mL volumetric flask and diluting to volume with ethyl acetate. The total weight of the test substances and the test system was approximately 4860 mg (Table I). The solutions were shaken to dissolve and mixed well.

6. Preparation of Linearity samples:

The linearity of 3,3-dichloro-1-propene, 4-chloro-4-methyl-1-pentene, 5-chloro-1-hexene, (Z)-1-chloro-1,5-hexadiene, 3-chloro-1-methylcyclopentane, and 1-chloro-1-methylcyclopentane were determined using the recovery and LOQ samples.

7. Preparation of Samples for the Evaluation of Interferences and Ruggedness:

Test Substance: Test substance samples were prepared by weighing ~ 50 mg of each test substance into separate 50 mL volumetric flasks and diluting to volume with ethyl acetate.

ESO Blank: An ESO blank solution was prepared by weighing 86.4 mg of Epoxidised Soybean Oil into a 5 mL volumetric flask and diluting to volume with ethyl acetate.

Solvent Blank: Solvent blanks were prepared by transferring ethyl acetate (Sigma Aldrich and EMD) into separate autosampler vials

The ESO blank, test substances, and solvent blanks were used for interferences and ruggedness evaluations.

The conditions detailed in section C_III.E, were modified to evaluate method ruggedness. On the first evaluation, the flow rate was changed from 1.9 to 1.7 mL/minute. On the second evaluation, the flow rate was changed from 1.9 to 2.1 mL/minute.

8. Evaluation of Limit of Quantitation (LOQ) and Limit of Detection (LOD):

Since the test system contained all of the test substances, the signal to noise ratio was determined from the analysis of sample solutions prepared as described in Section C_III.D.1 and C_III.D.3.

Samples were diluted to a target signal to noise ratio of 10 for LOQ and 3 for LOD. One solution was prepared for this evaluation and each solution was injected six times.

Samples were analyzed using the conditions given in Section C_III.E. The accuracy and precision of the analysis at these levels were evaluated.

E. Instrumentation and LC Conditions

The following analytical conditions describe the analysis of samples for 1,3-dichloropropene in Telone II technical grade active ingredient (TGAI).

Column:	DB-1701 60 m x 0.32 mm x 1µm	
Inlet temperature:	150°C	
Split ratio:	38:1	
Injection volume:	2 µL	
Column flow:	Constant flow at 1.9 mL/min Helium	
Column temperature:	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	
Detector:	TCD	
Detector temperature:	280°C	
Detector flow rate:	Reference Flow (Helium): 15 mL/min Make-up (Helium): 5 mL/min	
Run Time:	30.9 minutes	
Integrator:	Agilent EZChrom	
Retention Times:	3,3-dichloro-1-propene (M)	~ 11.4 min
	4-chloro-4-methyl-1-pentene (N)	~ 13.4 min
	Cis-1,3-dichloropropene	~ 15.4 min
	(Z)-1-chloro-1,5-hexadiene (P)	~ 16.6 min
	5-chloro-1-hexene (O)	~ 16.9 min
	Trans-1,3-dichloropropene	~ 18.1 min
	1-chloro-1-methylcyclopentane (R)	~ 18.5 min
	3-chloro-1-methylcyclopentane (Q)	~ 20.6 min

Approximate time to prepare and analyze a sample: 4 hours

F. Methods of Calculation

1. Calculation of the weight of the impurity in the calibration solutions:

$$Wt_x = Wt_{RS} \times \text{Purity}_{RS}$$

where:

- Wt_x = Weight of the impurity in the Calibration Standard Solution
 Wt_{RS} = Weight of the reference substance added to the calibration solution
 Purity_{RS} = Purity of the Reference Substance, expressed as a decimal

The following is an example calculation for 3,3-dichloro-1-propene (M) in 13-48-Std-C calibration solution:

$$Wt_M = 66.7 \text{ mg} \times 0.97 = 64.7 \text{ mg}$$

where:

- Wt_M = Weight of 3,3-dichloro-1-propene (M) in the Calibration Standard Solution
 Wt_{RS} = 66.7 mg
 Purity_{RS} = 97% = 0.97

2. Calculation of the response factor for the impurity in the calibration solutions:

$$Rf = \frac{Wt_x}{A_x}$$

where:

- Rf = Response factor
 Wt_x = Weight of impurity in stock standard solution
 A_x = Area of impurity peak obtained during analysis of the calibration solution

The following is an example calculation for 3,3-dichloro-1-propene (M) in 13-48-Std-C calibration solution (\elntrd12\ezchrom\Projects\202gc104\Das-Am\Das-Am-G-13-48\Precision\Day1\b\003_13-48-STD-C.dat):

$$Rf = \frac{64.7 \text{ mg}}{61385} = 0.001054003$$

where:

- Rf = Response factor for 3,3-dichloro-1-propene (M)
 Wt_M = 64.7 mg
 A_M = 61385

The response factors from all standard injections during a run sequence were averaged to calculate $RF_{(avg)}$. The $RF_{(avg)}$ for 3,3-dichloro-1-propene (M) in run sequence \elntrd12\ezchrom\Projects\202gc104\Das-Am\Das-Am-G-13-49\Precision\Day1\b\Das-Am-G-13-48 PrecD1b.seq was 0.00100818.

3. Calculation of the weight % of impurity in the sample:

$$Wt\%_x = \frac{A_x \times RF_{(avg\ x)}}{Wt_{sample} \times \text{Dilution factor}} \times 100$$

where:

Wt% _x	= Weight % of impurity in the sample
A _x	= Area of impurity obtained during analysis of the sample solution
RF _(avg x)	= Average response factor for impurity
Wt _{sample}	= Weight of sample in sample solution
Dilution factor	= Dilution factor to account for the difference in volume between the standards and samples

The following is an example calculation for 3,3-dichloro-1-propene (M) in sample solution, TSN106191-A2 (\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Precision\Day1\b\007_TSN106191-A2-Rep1.dat):

$$Wt\%_M = \frac{8040 \times 0.00100818}{2500.6 \times 10} \times 100 = 0.032\%$$

where:

Wt% _M	= Weight % of 3,3-dichloro-1-propene (M) in the sample
A _M	= 8040
RF _(avg M)	= 0.00100818
Wt _{sample}	= 2500.6 mg
Dilution factor	= 50/5 = 10

4. Statistical Methods

The statistical methods used in this study were means, standard deviations, relative standard deviations, Horwitz equation for acceptable repeatability, linear regression, and t-test.

$$\text{Average (arithmetic mean)} = \frac{\text{sum of all numbers ("x")}}{n}$$

$$\text{Standard deviation} = \sqrt{\frac{\sum (x - \bar{x})^2}{(n-1)}}$$

$$\text{relative standard deviation} = \frac{\text{standard deviation}}{\text{average}} \times 100$$

Horowitz RSD_R= $2^{(1-0.5\log C)}$
where C = concentration of the analyte in the sample as a decimal fraction

$$\text{Horowitz RSD}_r = \text{RSD}_R \times 0.67$$

Linear regression: $y = mx + b$
Where m = the slope of the line and b = the intercept

5. Spreadsheet and Data System Calculations

Calculation of response factors and weight percent values were performed with a chromatographic data system. Calculation of regression analysis and remaining calculations were performed with an Excel spreadsheet. Calculations by hand using the data provided in the examples and tables may have slight differences due to rounding and significant figures used by the EZ Chrom Elite chromatographic data system or an Excel spreadsheet.

C_IV. RESULTS AND DISCUSSION

A. Precision

The precision of the method was evaluated by preparing and analyzing five aliquots of Telone II TGAI on each of two days. Fresh standard solutions were prepared for each group of samples. For each impurity analysis on each day, the average concentration and relative standard deviation (RSD) was calculated. Results are summarized in [Table I](#).

The acceptability of the RSD was assessed in each case by the Horwitz equation. Since the experimental RSD was less than the Horwitz RSD_r for 3,3-dichloro-1-propene, 4-chloro-4-methyl-1-pentene, 5-chloro-1-hexene, (Z)-1-chloro-1,5-hexadiene, and 3-chloro-1-methylcyclopentane the precision of the method was acceptable for these impurities. 1-chloro-1-methylcyclopentane did not pass the Horwitz precision test over both days of testing mainly due to low levels of the impurity in the test system. However, acceptable standard deviation was observed. Taking the low standard deviation into consideration and the low levels of impurities, it can be concluded that acceptable precision has been demonstrated for this method. A typical chromatogram of a calibration solution and a sample solution are shown in Figures [1](#) and [2](#), respectively.

System precision was determined by analyzing a prepared Telone II TGAI sample five times. Results are summarized in [Table II](#).

B. Recovery

The recovery (accuracy) of the method was evaluated by analysis of a series of samples prepared by fortifying the test system with 3,3-dichloro-1-propene, 4-chloro-4-methyl-1-pentene, 5-chloro-1-hexene, (Z)-1-chloro-1,5-hexadiene, 3-chloro-1-methylcyclopentane and 1-chloro-1-methylcyclopentane as described in Section C_III.D.5 and in [Table III](#). The applicable validation ranges (equivalent weight %) are provided in [Table IV](#).

Recovery data are summarized in [Table V-X](#). Recovery results were found to be acceptable based on SANCO guidelines (SANCO/3030/99 rev.4).

C. Linearity

Linearity of detector response was evaluated using the recovery and LOQ samples. The relationship between peak area and concentration was linear for all impurities. Results are summarized in [Figures 3-8](#).

D. Stability

Stability was determined by analyzing method precision samples three days after the initial analysis. Fresh standard solutions were prepared for the reanalysis. The t-test was used to compare the results and indicated that concentrations for 4-chloro-4-methyl-1-pentene, 5-chloro-1-hexene, (Z)-1-chloro-1,5-hexadiene, 3-chloro-1-methylcyclopentane and 1-chloro-

1-methylcyclopentane were statistically equivalent. 3,3-dichloro-1-propene did not pass the t-test. However, the results are not practically different (when reported to 2 decimal places as the impurities are typically reported, both values are 0.03%). Taking the low difference in results between days into consideration and the low levels of impurities, it can be concluded that acceptable stability has been demonstrated for this method for 3,3-dichloro-1-propene. Therefore, the sample solutions are considered to be stable for at least three days.

Results are summarized in [Table XI](#).

E. Interferences

No interferences were observed from the EMD ethyl acetate solvent blank, the ESO blank, and several of the test substances (3,3-dichloro-1-propene, 1,2-dichloropropane, 2-chloro-2-methylpentane, 2-chloro-4-methylpentane, 2-chloro-2,3-dimethylbutane, 1-chloro-1-methylcyclopentene, 1,3-dichloropropane, and 3-chloro-1-methylcyclopentene) in the analytical method.

Acceptable interference (less than 3%) was observed from several test substances (4-chloro-4-methyl-1-pentene, 2-chloro-1,5-hexadiene, 3-chloro-1,5-hexadiene, 3-chloro-2-methylpentane, 1-chloro-1,5-hexadiene, 5-chloro-1-hexene, 1,2,2-trichloropropane, Trans-1,3,3-trichloropropene in the analytical method.

If the purity of the test substances which showed no interferences or acceptable levels of interferences decreases, or a test substance of lower purity is obtained, the test substance should be re-evaluated to determine its suitability of use in a combined standard solution or the test substance should be prepared separately.

Unacceptable interference ($\geq 3\%$) was observed from Cis-1,3,3-trichloropropene and Cis/Trans-2-chloro-3(chloromethyl)oxirane. As a result, standards prepared using these test substances should be prepared separately. If a test substance of higher purity is obtained, the test substance may be re-evaluated to determine its suitability of use in a combined standard solution or may continue to be prepared separately.

Unacceptable interference ($\geq 3\%$) was observed from Sigma Aldrich ethyl acetate ($\geq 99.5\%$). Therefore a higher purity ethyl acetate (such as EMD min 99.9%) must be used for the preparation and analysis of Telone II TGAI samples according to this method. Alternative suppliers and purities of ethyl acetate should be evaluated prior to use.

Results are summarized in [Table XII](#) and [Figures 9-11](#).

F. Method Ruggedness

Ruggedness of the analytical method was tested by changing the nominal flow rate of 1.9 mL/minute in the analytical method to 1.7 and 2.1 mL/minute. The solvent blanks, ESO blank, and test substances were injected. Increasing and decreasing the flow rate primarily

caused retention time shifts for all peaks ([Figure 12](#)). Interference results under the ruggedness conditions were consistent with results at the nominal flow rate.

G. LOD/LOQ

For each analytical evaluation of LOQ the precision of the replicate analyses was evaluated against the expected relative standard deviation calculated by the Horwitz equation and found to be acceptable at the prepared level for 3,3-dichloro-1-propene (M).

The precision for 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O) , (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) and 1-chloro-1-methylcyclopentane (R) were not acceptable by the Horwitz test for the evaluation; however, this is just one method of evaluation of acceptance. Acceptable relative standard deviation (RSD) was observed for 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O) , (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) and 1-chloro-1-methylcyclopentane (R) and therefore, precision was considered acceptable for 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O) , (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) and 1-chloro-1-methylcyclopentane (R). It should also be noted that the Horwitz test considers more significance than necessary for this low level analysis. Therefore the precision data is acceptable for its intended use and the unacceptable Horowitz results do not affect the quality or integrity of the data.

For 3,3-dichloro-1-propene (M), 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O), (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) the recovery was found to be within the acceptable range (75 – 125%). Recovery could not be calculated for 1-chloro-1-methylcyclopentane (R) because it was present in the sample at the LOQ (no dilution or spiking was necessary).

The results for the evaluation of LOQ and LOD are presented in [Tables XIII-XXIV](#).

C_V. CONCLUSIONS

This method is applicable to the determination of 3,3-dichloro-1-propene (M), 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O), (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) and 1-chloro-1-methylcyclopentane (R) in Telone II TGAI.

The recovery, linearity, and precision data have shown this method to be acceptable for the assay of 3,3-dichloro-1-propene (M), 4-chloro-4-methyl-1-pentene (N), 5-chloro-1-hexene (O), (Z)-1-chloro-1,5-hexadiene (P), 3-chloro-1-methylcyclopentane (Q) and 1-chloro-1-methylcyclopentane (R) in Telone II TGAI.

If the method is used with another set of equipment, it is suggested that method precision, linearity, and method limits be re-determined.

This report accurately reflects what was done during the course of the study and includes all amendments and/or deviations to the protocol.

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, Horwitz equation, regression analysis, t-test, and Grubbs test for outliers. The databook(s), 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 9330 Zionsville Road, Indianapolis, Indiana.

C_VI. TABLES

Table I. Method Precision Data for Impurities in Telone II TGAI

Date	Sample Id	3,3-dichloro-1-propene (M)	4-chloro-4-methyl-1-pentene (N)	(Z)-1-chloro-1,5-hexadiene (P)	5-chloro-1-hexene (O)	1-chloro-1-methylcyclopentane (R)	3-chloro-1-methylcyclopentane (Q)
24-Jan-14	TSN106191-A2 Rep 1	0.032	0.027	0.038	0.094	0.004	0.025
	TSN106191-B2	0.032	0.028	0.037	0.088	0.003	0.026
	TSN106191-C2	0.033	0.028	0.038	0.090	0.003	0.025
	TSN106191-D2	0.033	0.027	0.035	0.090	0.004	0.025
	TSN106191-E2	0.032	0.027	0.037	0.089	0.004	0.025
27-Jan-14	TSN106191-F	0.034	0.027	0.037	0.091	0.003	0.025
	TSN106191-G	0.034	0.028	0.037	0.088	0.004	0.023
	TSN106191-H	0.034	0.027	0.036	0.089	0.003	0.025
	TSN106191-I	0.034	0.027	0.038	0.092	0.003	0.023
	TSN106191-J	0.034	0.028	0.037	0.087	0.003	0.024
Overall Average		0.03	0.03	0.04	0.09	0.003	0.02
Std. Dev.		0.001	0.001	0.001	0.002	0.001	0.001
Overall RSD		2.8	1.9	2.5	2.3	15.2	3.9
Horwitz RSD_R		6.7	6.9	6.6	5.7	9.4	7.0
Horwitz RSD_{Dr}		4.5	4.6	4.4	3.9	6.3	4.7
Acceptable (Overall RSD<Horwitz RSD_{Dr})		Acceptable	Acceptable	Acceptable	Acceptable	Not Acceptable	Acceptable

Table II. System Precision Data for Impurities in Telone II TGAI

Sample Id	3,3-dichloro-1-propene (M)	4-chloro-4-methyl-1-pentene (N)	(Z)-1-chloro-1,5-hexadiene (P)	5-chloro-1-hexene (O)	1-chloro-1-methylcyclopentane (R)	3-chloro-1-methylcyclopentane (Q)
TSN106191-A2 Rep 1	0.032	0.027	0.038	0.094	0.004	0.025
TSN106191-A2 Rep 2	0.032	0.028	0.043	0.103	0.004	0.025
TSN106191-A2 Rep 3	0.032	0.028	0.040	0.094	0.004	0.026
TSN106191-A2 Rep 4	0.032	0.028	0.036	0.090	0.004	0.025
TSN106191-A2 Rep 5	0.033	0.027	0.036	0.088	0.003	0.025
Average	0.032	0.028	0.039	0.094	0.004	0.025
Std. Dev.	0.000	0.001	0.003	0.006	0.000	0.000
RSD	1.4	2.0	7.7	6.1	11.8	1.8

Table III. Preparation of Recovery Samples (Impurities in Telone II TGAI)

Sample ID (% nominal)	Actual Weight (mg)							Total Weight (mg) ¹
	3,3-dichloro-1-propene (M)	4-chloro-4-methyl-1-pentene (N)	5-chloro-1-hexene (O)	(Z)-1-chloro-1,5-hexadiene (P)	3-chloro-1-methylcyclopentane (Q)	1-chloro-1-methylcyclopentane (R)	Telone II	
TSN030579-0001	TSN303759	TSN106329	TSN304464	TSN303341	TSN303032	TSN106191		
Rec Lin - A	0	0	0	0	0	0	4857.90	4857.90
Rec Lin - B	0.43	0.42	0.43	0.41	0.43	0.43	4855.46	4858.01
Rec Lin - C	2.15	2.09	2.16	2.07	2.14	2.14	4845.68	4858.44
Rec Lin - D	4.30	4.18	4.31	4.14	4.29	4.29	4833.46	4858.97
Rec Lin - E	4.30	4.18	4.31	4.14	4.29	4.29	4833.46	4858.97
Rec Lin - F	10.76	10.46	10.79	10.35	10.71	10.72	4796.80	4860.59
Rec Lin - G	21.52	20.92	21.57	20.69	21.43	21.45	4735.70	4863.27
Rec Lin - H	43.04	41.84	43.14	41.38	42.85	42.89	4613.50	4868.64

¹The total sample weight is the sum of weights from all of the impurities and Telone II.

Table IV. Equivalent Weight % in Recovery Samples (Impurities in Telone II TGAI)

Sample ID (% nominal)	Equivalent Weight %					
	3,3-dichloro-1-propene (M)	4-chloro-4-methyl-1-pentene (N)	5-chloro-1-hexene (O)	(Z)-1-chloro-1,5-hexadiene (P)	3-chloro-1-methylcyclopentane (Q)	1-chloro-1-methylcyclopentane (R)
Rec Lin - B (0.01%)	0.009	0.008	0.009	0.008	0.009	0.009
Rec Lin - C (0.05%)	0.043	0.041	0.044	0.041	0.043	0.044
Rec Lin - D (0.1%)	0.086	0.083	0.088	0.083	0.086	0.088
Rec Lin - E (0.1%)	0.086	0.083	0.088	0.083	0.086	0.088
Rec Lin - F (0.25%)	0.215	0.207	0.220	0.206	0.214	0.221
Rec Lin - G (0.5%)	0.429	0.413	0.439	0.413	0.427	0.441
Rec Lin - H (1%)	0.858	0.825	0.877	0.824	0.854	0.881

$$\text{Equivalent Wt\%} = \frac{\text{Actual Weight of Impurity} \times \% \text{ purity (as decimal)}}{\text{Total weight}} \times 100$$

Table V. Recovery Data for 3,3-dichloro-1-propene (M, X191117)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.009	8987	1659	0.044	0.008	88.9	88.9
Rec Lin - B	2	0.009	8954	1626	0.044	0.008	88.9	
Rec Lin - C	1	0.043	15997	8669	0.079	0.043	100.0	98.8
Rec Lin - C	2	0.043	15957	8629	0.078	0.042	97.7	
Rec Lin - D	1	0.086	24617	17289	0.121	0.085	98.8	99.4
Rec Lin - D	2	0.086	24773	17445	0.122	0.086	100.0	
Rec Lin - E	1	0.086	24623	17295	0.121	0.085	98.8	98.3
Rec Lin - E	2	0.086	24514	17186	0.120	0.084	97.7	
Rec Lin - F	1	0.215	50582	43254	0.248	0.212	98.6	98.8
Rec Lin - F	2	0.215	50747	43419	0.249	0.213	99.1	
Rec Lin - G	1	0.429	94947	87619	0.466	0.430	100.2	99.9
Rec Lin - G	2	0.429	94305	86977	0.463	0.427	99.5	
Rec Lin - H	1	0.858	181790	174462	0.891	0.855	99.7	99.8
Rec Lin - H	2	0.858	182045	174717	0.893	0.857	99.9	
Average							98	
SANCO guidelines for mean % recovery for <0.1% Impurity							75-125%	
SANCO guidelines for mean % recovery for 0.1-1% Impurity							80-120%	

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table VI. Recovery Data for 4-chloro-4-methyl-1-pentene (N, X11656876)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.008	7547	1693	0.035	0.008	93.8	100.0
Rec Lin - B	2	0.008	7562	1708	0.036	0.009	106.3	
Rec Lin - C	1	0.041	14542	8688	0.068	0.041	98.8	98.8
Rec Lin - C	2	0.041	14441	8587	0.068	0.041	98.8	
Rec Lin - D	1	0.083	23592	17738	0.111	0.084	100.6	100.6
Rec Lin - D	2	0.083	23588	17734	0.111	0.084	100.6	
Rec Lin - E	1	0.083	23589	17735	0.111	0.084	100.6	100.6
Rec Lin - E	2	0.083	23562	17708	0.111	0.084	100.6	
Rec Lin - F	1	0.207	48969	43115	0.23	0.203	97.8	98.3
Rec Lin - F	2	0.207	49497	43643	0.232	0.205	98.8	
Rec Lin - G	1	0.413	93242	87388	0.438	0.411	99.4	99.0
Rec Lin - G	2	0.413	92619	86765	0.435	0.408	98.7	
Rec Lin - H	1	0.825	179386	173532	0.841	0.814	98.6	98.7
Rec Lin - H	2	0.825	179744	173890	0.843	0.816	98.8	
Average							99	
SANCO guidelines for mean % recovery for <0.1% Impurity							75-125%	
SANCO guidelines for mean % recovery for 0.1-1% Impurity							80-120%	

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table VII. Recovery Data for 5-chloro-1-hexene (O, X11656878)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.009	21380	1785	0.097	0.008	88.9	94.4
Rec Lin - B	2	0.009	21597	2002	0.098	0.009	100.0	
Rec Lin - C	1	0.044	28936	9341	0.131	0.042	95.5	101.1
Rec Lin - C	2	0.044	30105	10510	0.136	0.047	106.8	
Rec Lin - D	1	0.088	39354	19759	0.178	0.089	101.1	102.8
Rec Lin - D	2	0.088	39829	20234	0.181	0.092	104.5	
Rec Lin - E	1	0.088	39186	19591	0.178	0.089	101.1	101.1
Rec Lin - E	2	0.088	39213	19618	0.178	0.089	101.1	
Rec Lin - F	1	0.220	67497	47902	0.306	0.217	98.6	98.2
Rec Lin - F	2	0.220	67076	47481	0.304	0.215	97.7	
Rec Lin - G	1	0.439	114988	95393	0.521	0.432	98.4	97.9
Rec Lin - G	2	0.439	114221	94626	0.517	0.428	97.5	
Rec Lin - H	1	0.877	208832	189237	0.945	0.856	97.6	97.6
Rec Lin - H	2	0.877	208835	189240	0.945	0.856	97.6	
Average							99	
SANCO guidelines for mean % recovery for <0.1% Impurity							75-125%	
SANCO guidelines for mean % recovery for 0.1-1% Impurity							80-120%	

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table VIII. Recovery Data for (Z)-1-chloro-1,5-hexadiene (P, X11661609)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.008	11439	2088	0.047	0.009	106.3	106.3
Rec Lin - B	2	0.008	11329	1978	0.047	0.009	106.3	
Rec Lin - C	1	0.041	19785	10434	0.081	0.043	103.7	107.3
Rec Lin - C	2	0.041	20518	11167	0.084	0.046	111.0	
Rec Lin - D	1	0.083	30630	21279	0.126	0.088	105.4	105.4
Rec Lin - D	2	0.083	30631	21280	0.126	0.088	105.4	
Rec Lin - E	1	0.083	30372	21021	0.125	0.087	104.2	104.8
Rec Lin - E	2	0.083	30577	21226	0.126	0.088	105.4	
Rec Lin - F	1	0.206	62010	52659	0.255	0.217	105.1	104.6
Rec Lin - F	2	0.206	61702	52351	0.253	0.215	104.1	
Rec Lin - G	1	0.413	114847	105496	0.471	0.433	104.7	104.5
Rec Lin - G	2	0.413	114186	104835	0.469	0.431	104.2	
Rec Lin - H	1	0.824	219267	209916	0.899	0.861	104.4	104.5
Rec Lin - H	2	0.824	219476	210125	0.900	0.862	104.6	
Average							105	
SANCO guidelines for mean % recovery for <0.1% Impurity							75-125%	
SANCO guidelines for mean % recovery for 0.1-1% Impurity							80-120%	

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table IX. Recovery Data for 3-chloro-1-methylcyclopentane (Q, X11726086)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.009	7692	1679	0.034	0.008	83.3	88.9
Rec Lin - B	2	0.009	7875	1862	0.035	0.009	94.4	
Rec Lin - C	1	0.043	15522	9509	0.069	0.043	98.8	98.8
Rec Lin - C	2	0.043	15346	9333	0.069	0.043	98.8	
Rec Lin - D	1	0.086	24997	18984	0.112	0.086	99.4	100.0
Rec Lin - D	2	0.086	25357	19344	0.113	0.087	100.6	
Rec Lin - E	1	0.086	24952	18939	0.111	0.085	98.3	98.3
Rec Lin - E	2	0.086	24908	18895	0.111	0.085	98.3	
Rec Lin - F	1	0.214	53488	47475	0.239	0.213	99.3	99.5
Rec Lin - F	2	0.214	53805	47792	0.24	0.214	99.8	
Rec Lin - G	1	0.427	101675	95662	0.454	0.428	100.1	99.9
Rec Lin - G	2	0.427	101234	95221	0.452	0.426	99.6	
Rec Lin - H	1	0.854	197261	191248	0.88	0.854	99.9	99.8
Rec Lin - H	2	0.854	196662	190649	0.877	0.851	99.6	
Average								98
SANCO guidelines for mean % recovery for <0.1% Impurity								75-125%
SANCO guidelines for mean % recovery for 0.1-1% Impurity								80-120%

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table X. Recovery Data for 1-chloro-1-methylcyclopentane (R, X11726078)

Sample ID	Rep	Equivalent Weight %	Peak Area	Corrected* Peak Area	Experimental Weight %	Corrected* Experimental Weight %	% Recovery	Average % Recovery
Rec Lin - B	1	0.009	2371	1671	0.011	0.008	88.9	88.9
Rec Lin - B	2	0.009	2456	1756	0.011	0.008	88.9	
Rec Lin - C	1	0.044	9768	9068	0.044	0.041	93.2	93.2
Rec Lin - C	2	0.044	9806	9106	0.044	0.041	93.2	
Rec Lin - D	1	0.088	19210	18510	0.087	0.084	95.5	94.9
Rec Lin - D	2	0.088	19065	18365	0.086	0.083	94.3	
Rec Lin - E	1	0.088	19093	18393	0.086	0.083	94.3	94.3
Rec Lin - E	2	0.088	18945	18245	0.086	0.083	94.3	
Rec Lin - F	1	0.221	47691	46991	0.216	0.213	96.4	96.2
Rec Lin - F	2	0.221	47641	46941	0.215	0.212	95.9	
Rec Lin - G	1	0.441	95907	95207	0.433	0.430	97.5	97.4
Rec Lin - G	2	0.441	95504	94804	0.432	0.429	97.3	
Rec Lin - H	1	0.881	191232	190532	0.863	0.860	97.6	97.7
Rec Lin - H	2	0.881	191390	190690	0.864	0.861	97.7	
Average							95	
SANCO guidelines for mean % recovery for <0.1% Impurity							75-125%	
SANCO guidelines for mean % recovery for 0.1-1% Impurity							80-120%	

* Corrected for the amount of the impurities in Rec Lin - A

See Table IV for Equivalent weight %.

Table XI. Stability Data for Impurities in Telone II TGAI

Sample Id	3,3-dichloro-1-propene (M)		4-chloro-4-methyl-1-pentene (N)		(Z)-1-chloro-1,5-hexadiene (P)		5-chloro-1-hexene (O)		1-chloro-1-methyleclopentane (R)		3-chloro-1-methyleclopentane (Q)	
	24-Jan-14	27-Jan-14	24-Jan-14	27-Jan-14	24-Jan-14	27-Jan-14	24-Jan-14	27-Jan-14	24-Jan-14	27-Jan-14	24-Jan-14	27-Jan-14
TSN106191-A2*	0.032	0.033	0.027	0.029	0.038	0.038	0.094	0.091	0.004	0.003	0.025	0.024
TSN106191-B2	0.032	0.033	0.028	0.027	0.037	0.037	0.088	0.087	0.003	0.003	0.026	0.024
TSN106191-C2	0.033	0.034	0.028	0.027	0.038	0.036	0.090	0.086	0.003	0.003	0.025	0.025
TSN106191-D2	0.033	0.033	0.027	0.028	0.035	0.037	0.090	0.092	0.004	0.003	0.025	0.025
TSN106191-E2	0.032	0.034	0.027	0.027	0.037	0.037	0.089	0.091	0.004	0.003	0.025	0.025
Average	0.032	0.033	0.027	0.028	0.037	0.037	0.090	0.089	0.004	0.003	0.025	0.025
t Stat	-3.162		-0.343		2.194×10^{-15}		0.645		2.449		2.132	
t Critical two-tail	2.776		2.776		2.776		2.776		2.776		2.776	
Statistical outcome (Acceptable if $ t\text{Stat} \leq t\text{critical}$)	Not Acceptable		Acceptable		Acceptable		Acceptable		Acceptable		Acceptable	

*Used Rep 1 for 24-Jan-14

Table XII. Interferences Data

Sample	Purity	Retention Time	Interference Peak	Area of Interference Peak in Sample	Area of Interference Peak in Interference Peak Sample	% Interference
3,3-dichloro-1-propene (M)-Int	97%	11.423	None			
1,2-dichloropropane (A)-Int	99%	12.997	None			
4-chloro-4-methyl-1-pentene (N)-Int	96%	13.423	G	571	44311	1.3
			Trans-1,3-D	3089	10761588	0.0
2-chloro-2-methylpentane (B)-Int	97%	14.137	None			
2-chloro-1,5-hexadiene (C)-Int	96%	14.307	A	329	53786	0.6
			P	317	62472	0.0
			R	365	59697	0.6
2-chloro-4-methylpentane (D)-Int	98%	14.403	None			
2-chloro-2,3-dimethylbutane (E)-Int	100%	14.587	None			
3-chloro-1,5-hexadiene (F)-Int	98%	15.833	M	641	43169	1.5
3-chloro-2-methylpentane (G)-Int	79%	16.343	B	758	45981	1.6
1-chloro-1,5-hexadiene (P)-Int	97%	16.577	C	222	52204	0.4
			R	377	59697	0.6
5-chloro-1-hexene (O)-Int	99%	16.847	Cis-1,3-D	590	11850448	0.0
1-chloro-1-methylcyclopentene (R)-Int	100%	18.330	None			
1,3-dichloropropane (H)-Int	99.5%	19.260	None			
1,2,2-trichloropropane (I)-Int	97%	19.650	A	393	53786	0.7

Table XII. Interferences Data, cont.

Sample	Purity	Retention Time	Interference Peak	Area of Interference Peak in Sample	Area of Interference Peak in Interference Peak Sample	% Interference
3-chloro-1-methylcyclopentene (Q)-Int	97%	20.570 (Q1)	None			
		20.700 (Q2)				
Cis-1,3,3-trichloropropene (J)-Int	94%	21.340	Q1	1048	36172	2.9
			Q2	1025	22129	4.6
Cis/Trans-2-chloro-3(chloromethyl)oxirane (K)-Int	14%	24.227 (K-Cis)	A	2325	53786	4.3
		25.863 (K-Trans)				
Trans-1,3,3-trichloropropene (L)-Int	86.20%	25.097	J	279	68582	0.4
ESO-Int	N/A	N/A	None			
EMD EtAc-Int	N/A	N/A	None			
Sigma EtAc-Int	N/A	N/A	M	9146	43169	21.2

Table XIII. Limits of Quantitation for 3,3-dichloropropane (M)

Sample ID	Height	Actual % wt
TSN106191-Int-D4	442	0.006
TSN106191-Int-D4	443	0.006
TSN106191-Int-D4	427	0.006
TSN106191-Int-D4	446	0.006
TSN106191-Int-D4	447	0.006
TSN106191-Int-D4	438	0.006
Overall Average		0.006
Standard Dev		0.000
Overall RSD		0.0
Horwitz RSD_R		8.6
Horwitz RSD_r		5.8
Acceptable (Overall RSD<Horwitz RSD_r)		Acceptable
Overall Recovery		100
Signal / Noise		21

$$\text{Overall Recovery} = \text{Measured wt.\%} / \text{Theoretical wt.\%} \times 100$$

Table XIV. Limits of Detection for 3,3-dichloropropane (M)

Sample ID	Height	Actual % wt
TSN106191-Int-D6	98	0.001
TSN106191-Int-D6	89	0.001
TSN106191-Int-D6	88	0.001
TSN106191-Int-D6	95	0.001
TSN106191-Int-D6	96	0.001
TSN106191-Int-D6	89	0.001
Overall Average	93	0.001
Signal / Noise		5

Table XV. Limits of Quantitation for 4-chloro-4-methyl-1-pentene (N)

Sample ID	Height	Actual % wt
TSN106191-Int-D4	280	0.005
TSN106191-Int-D4	271	0.005
TSN106191-Int-D4	296	0.005
TSN106191-Int-D4	281	0.005
TSN106191-Int-D4	260	0.004
TSN106191-Int-D4	271	0.005
Overall Average		0.005
Standard Dev		0.000
Overall RSD		8.4
Horwitz RSD_R		8.9
Horwitz RSD_r		6.0
Acceptable (Overall RSD<Horwitz RSD_r)		Not Acceptable
Overall Recovery		100
Signal / Noise		13

Overall Recovery = Measured wt.% / Theoretical wt.% × 100

Table XVI. Limits of Detection for 4-chloro-4-methyl-1-pentene (N)

Sample ID	Height	Actual % wt
TSN106191-Int-D6	69	0.001
TSN106191-Int-D6	61	0.001
TSN106191-Int-D6	78	0.002
TSN106191-Int-D6	70	0.001
TSN106191-Int-D6	70	0.001
TSN106191-Int-D6	76	0.002
Overall Average	71	0.001
Signal / Noise		3

Table XVII. Limits of Quantitation for 5-chloro-1-hexene (O)

Sample ID	Height	Actual % wt
TSN106191-Int-D5	253	0.007
TSN106191-Int-D5	250	0.006
TSN106191-Int-D5	243	0.006
TSN106191-Int-D5	257	0.007
TSN106191-Int-D5	264	0.008
TSN106191-Int-D5	230	0.006
Overall Average		0.007
Standard Dev		0.001
Overall RSD		12.2
Horwitz RSD_R		8.5
Horwitz RSD_r		5.7
Acceptable (Overall RSD<Horwitz RSD_r)		Not Acceptable
Overall Recovery		117
Signal / Noise		12

$$\text{Overall Recovery} = \text{Measured wt.\%} / \text{Theoretical wt.\%} \times 100$$

Table XVIII. Limits of Detection for 5-chloro-1-hexene (O)

Sample ID	Height	Actual % wt
TSN106191-Int-D6	146	0.004
TSN106191-Int-D6	158	0.005
TSN106191-Int-D6	150	0.005
TSN106191-Int-D6	146	0.005
TSN106191-Int-D6	119	0.003
TSN106191-Int-D6	134	0.004
Overall Average	142	0.004
Signal / Noise		7

Table XIX. Limits of Quantitation for (Z)-1-Chloro-1,5-hexadiene (P)

Sample ID	Height	Actual % wt
TSN106191-Int-D4	367	0.007
TSN106191-Int-D4	384	0.007
TSN106191-Int-D4	383	0.007
TSN106191-Int-D4	396	0.007
TSN106191-Int-D4	383	0.006
TSN106191-Int-D4	385	0.006
Overall Average		0.007
Standard Dev		0.001
Overall RSD		7.7
Horwitz RSD_R		8.5
Horwitz RSD_r		5.7
Acceptable (Overall RSD<Horwitz RSD_r)		Not Acceptable
Overall Recovery		117
Signal / Noise		19

$$\text{Overall Recovery} = \text{Measured wt.\%} / \text{Theoretical wt.\%} \times 100$$

Table XX. Limits of Detection for (Z)-1-Chloro-1,5-hexadiene (P)

Sample ID	Height	Actual % wt
TSN106191-Int-D6	64	0.001
TSN106191-Int-D6	95	0.001
TSN106191-Int-D6	75	0.001
TSN106191-Int-D6	76	0.001
TSN106191-Int-D6	99	0.002
TSN106191-Int-D6	72	0.001
Overall Average	80	0.001
Signal / Noise		4

Table XXI. Limits of Quantitation for 3-chloro-1-methylcyclopentane (Q)

Sample ID	Height*	Actual % wt
TSN106191-Int-D3	228	0.009
TSN106191-Int-D3	218	0.008
TSN106191-Int-D3	239	0.008
TSN106191-Int-D3	236	0.009
TSN106191-Int-D3	214	0.008
TSN106191-Int-D3	242	0.009
Overall Average		0.009
Standard Dev		0.001
Overall RSD		6.4
Horwitz RSD_R		8.2
Horwitz RSD_r		5.5
Acceptable (Overall RSD<Horwitz RSD_r)		Not Acceptable
Overall Recovery		113
Signal / Noise		11

* Height corresponding to Q2

Overall Recovery = Measured wt.% / Theoretical wt.% × 100

Table XXII. Limits of Detection for 3-chloro-1-methylcyclopentane (Q)

Sample ID	Height*	Actual % wt
TSN106191-Int-D4	125	0.004
TSN106191-Int-D4	129	0.004
TSN106191-Int-D4	111	0.004
TSN106191-Int-D4	125	0.005
TSN106191-Int-D4	124	0.005
TSN106191-Int-D4	130	0.005
Overall Average	124	0.005
Signal / Noise		6

Table XXIII. Limits of Quantitation for 1-chloro-1-methylcyclopentane (R)

Sample ID	Height	Actual % wt
TSN106191-A2	234	0.004
TSN106191-A2	220	0.004
TSN106191-A2	244	0.004
TSN106191-A2	217	0.004
TSN106191-A2	189	0.003
TSN106191-A2	191	0.003
Overall Average		0.004
Standard Dev		0.001
Overall RSD		14.1
Horwitz RSD_R		9.3
Horwitz RSD_r		6.2
Acceptable (Overall RSD<Horwitz RSD_r)		Not Acceptable
Overall Recovery		N/A*
Signal / Noise		12

* Not applicable because 1-chloro-1-methylpentane was present in the sample at the LOQ
 (no spiking or dilution needed)

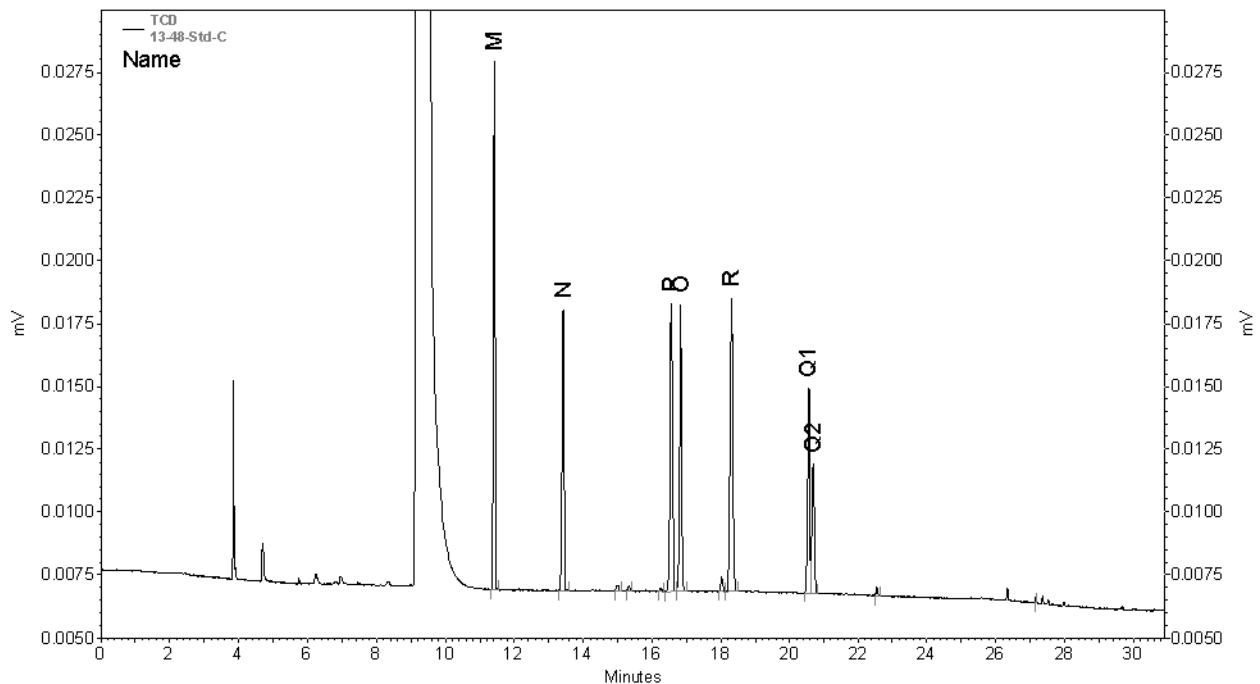
$$\text{Overall Recovery} = \text{Measured wt.\%} / \text{Theoretical wt.\%} \times 100$$

Table XXIV. Limits of Detection for 1-chloro-1-methylcyclopentane (R)

Sample ID	Height	Actual % wt
TSN106191-Int-D2	97	0.002
TSN106191-Int-D2	120	0.002
TSN106191-Int-D2	103	0.002
TSN106191-Int-D2	118	0.002
TSN106191-Int-D2	120	0.002
TSN106191-Int-D2	110	0.002
Overall Average	111	0.002
Signal / Noise		5

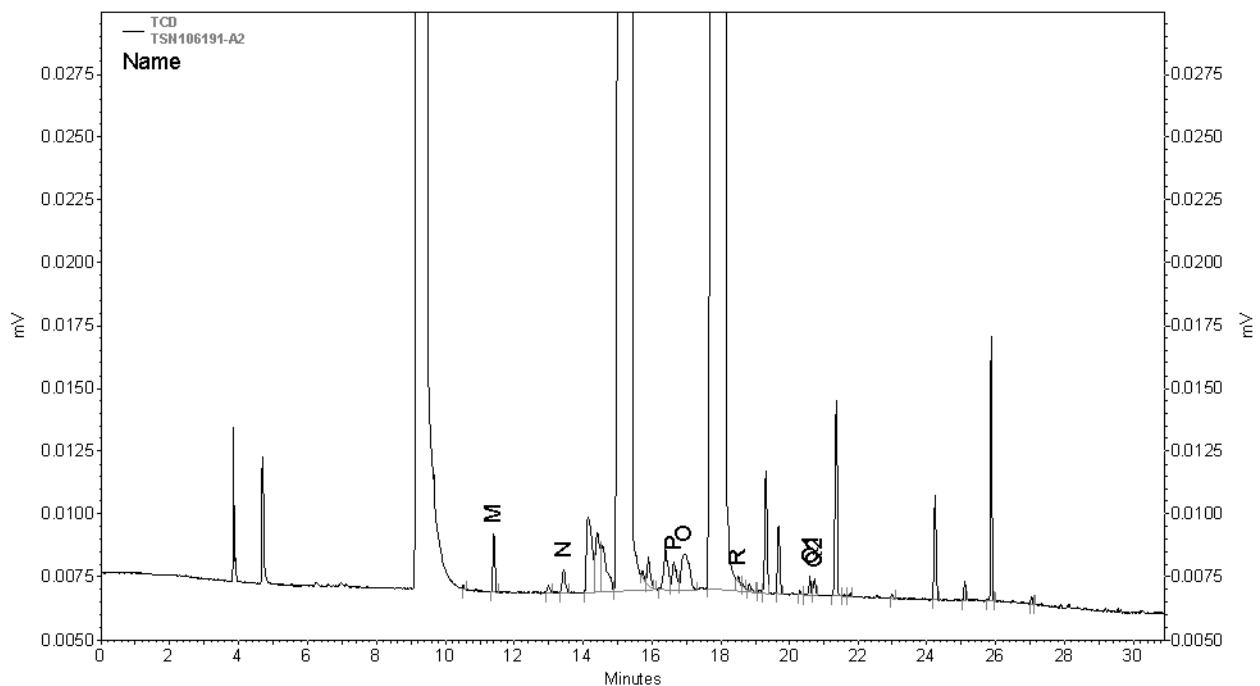
C_VII. FIGURES

Figure 1. Representative Chromatogram of the Standard Solution



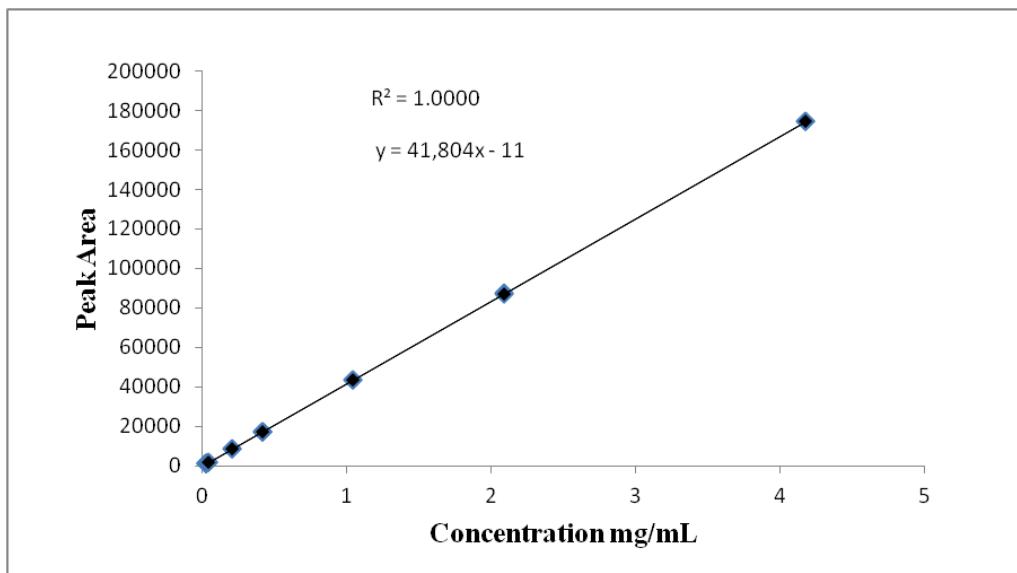
Data file: \\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Precision
|Day1\b\003_13-48-STD-C.dat

Figure 2. Representative Chromatogram of a TGAI Solution



Data file: \\e\lntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Precision\
Day1\b\007_TSN106191-A2-Rep1.dat

Figure 3. Linearity of 3,3-dichloro-1-propene (M, X191117)

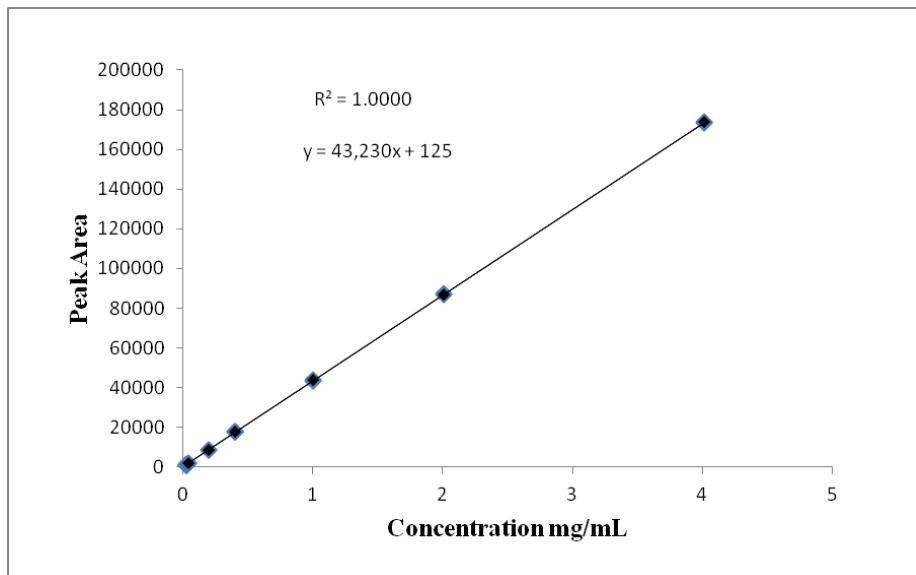


Sample ID	Rep	Weight % ¹	Peak Area ²	Concentration(mg/mL)
TSN106191-Int-D4	1	0.006	1310	0.027
TSN106191-Int-D4	2	0.006	1338	0.027
TSN106191-Int-D4	3	0.006	1288	0.027
TSN106191-Int-D4	4	0.006	1300	0.027
TSN106191-Int-D4	5	0.006	1336	0.027
TSN106191-Int-D4	6	0.006	1301	0.027
Rec Lin - B	1	0.009	1659	0.042
Rec Lin - B	2	0.009	1626	0.042
Rec Lin - C	1	0.043	8669	0.209
Rec Lin - C	2	0.043	8629	0.209
Rec Lin - D	1	0.086	17289	0.417
Rec Lin - D	2	0.086	17445	0.417
Rec Lin - E	1	0.086	17295	0.417
Rec Lin - E	2	0.086	17186	0.417
Rec Lin - F	1	0.215	43254	1.044
Rec Lin - F	2	0.215	43419	1.044
Rec Lin - G	1	0.429	87619	2.087
Rec Lin - G	2	0.429	86977	2.087
Rec Lin - H	1	0.858	174462	4.175
Rec Lin - H	2	0.858	174717	4.175
Slope (m)=				41804
Y-intercept (b) =				-11
Coefficient of determination (r2) =				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 4. Linearity of 4-chloro-4-methyl-1-pentene (N, X11656876)

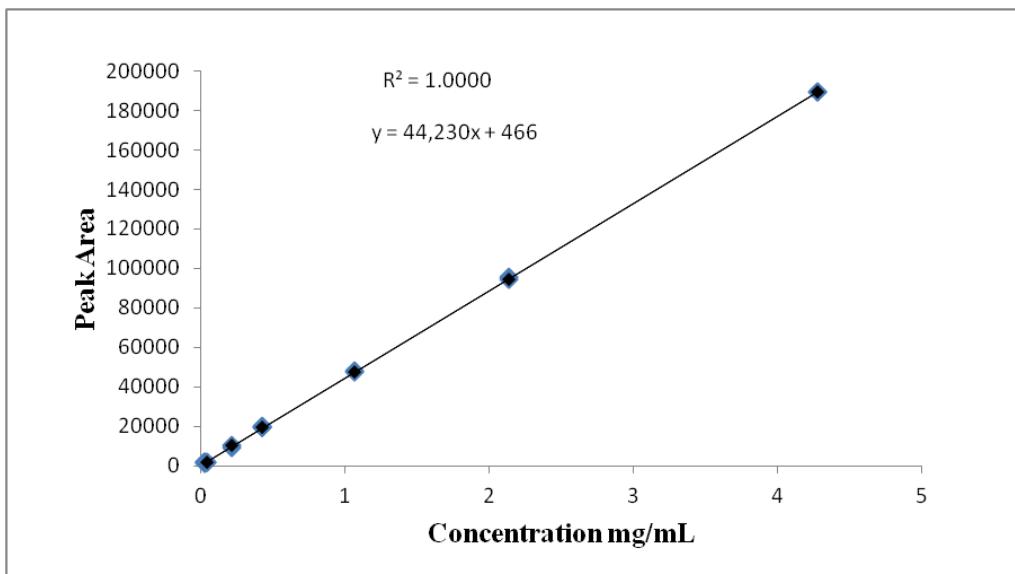


Sample ID	Rep	Weight % ¹	Peak Area ²	Concentration (mg/mL)
TSN106191-Int-D4	1	0.005	1072	0.022
TSN106191-Int-D4	2	0.005	1082	0.022
TSN106191-Int-D4	3	0.005	1129	0.022
TSN106191-Int-D4	4	0.005	1012	0.022
TSN106191-Int-D4	5	0.004	971	0.022
TSN106191-Int-D4	6	0.005	1106	0.022
Rec Lin - B	1	0.008	1693	0.040
Rec Lin - B	2	0.008	1708	0.040
Rec Lin - C	1	0.041	8688	0.201
Rec Lin - C	2	0.041	8587	0.201
Rec Lin - D	1	0.083	17738	0.402
Rec Lin - D	2	0.083	17734	0.402
Rec Lin - E	1	0.083	17735	0.402
Rec Lin - E	2	0.083	17708	0.402
Rec Lin - F	1	0.207	43115	1.004
Rec Lin - F	2	0.207	43643	1.004
Rec Lin - G	1	0.413	87388	2.008
Rec Lin - G	2	0.413	86765	2.008
Rec Lin - H	1	0.825	173532	4.017
Rec Lin - H	2	0.825	173890	4.017
Slope (m)=				43230
Y-intercept (b) =				125
Coefficient of determination (r2) =				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 5. Linearity of 5-chloro-1-hexene (O, X11656878)

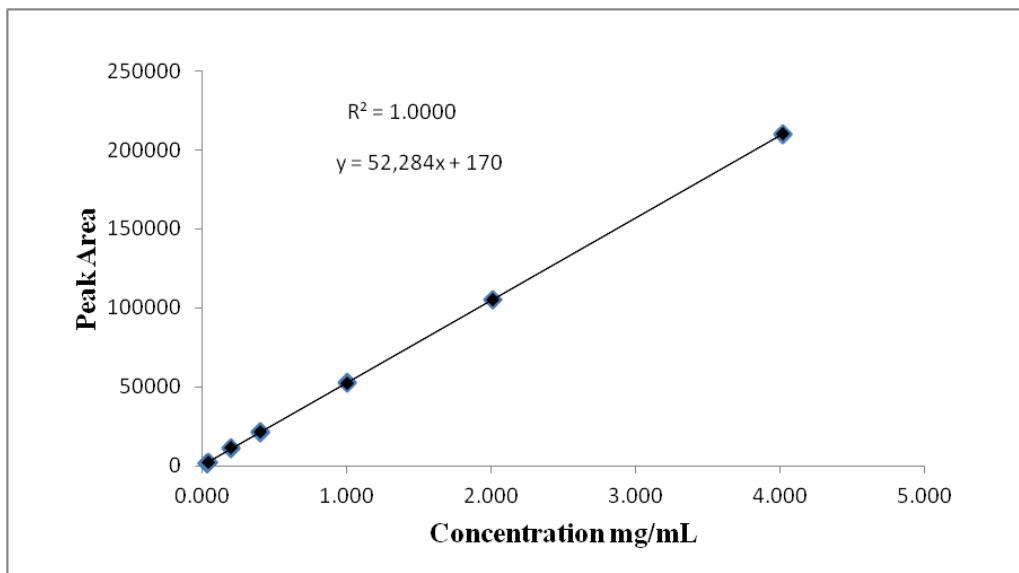


	Rep	Weight % ¹	Peak Area ²	Concentration (mg/mL)
TSN106191-Int-D5	1	0.007	1641	0.029
TSN106191-Int-D5	2	0.006	1541	0.029
TSN106191-Int-D5	3	0.006	1523	0.029
TSN106191-Int-D5	4	0.007	1705	0.029
TSN106191-Int-D5	5	0.008	1857	0.029
TSN106191-Int-D5	6	0.006	1465	0.029
Rec Lin - B	1	0.009	1785	0.043
Rec Lin - B	2	0.009	2002	0.043
Rec Lin - C	1	0.044	9341	0.214
Rec Lin - C	2	0.044	10510	0.214
Rec Lin - D	1	0.088	19759	0.427
Rec Lin - D	2	0.088	20234	0.427
Rec Lin - E	1	0.088	19591	0.427
Rec Lin - E	2	0.088	19618	0.427
Rec Lin - F	1	0.220	47902	1.068
Rec Lin - F	2	0.220	47481	1.068
Rec Lin - G	1	0.439	95393	2.135
Rec Lin - G	2	0.439	94626	2.135
Rec Lin - H	1	0.877	189237	4.271
Rec Lin - H	2	0.877	189240	4.271
Slope (m)=				44230
Y-intercept (b)=				466
Coefficient of determination (r2)=				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 6. Linearity of (Z)-1-chloro-1,5-hexadiene (P, X11661609)

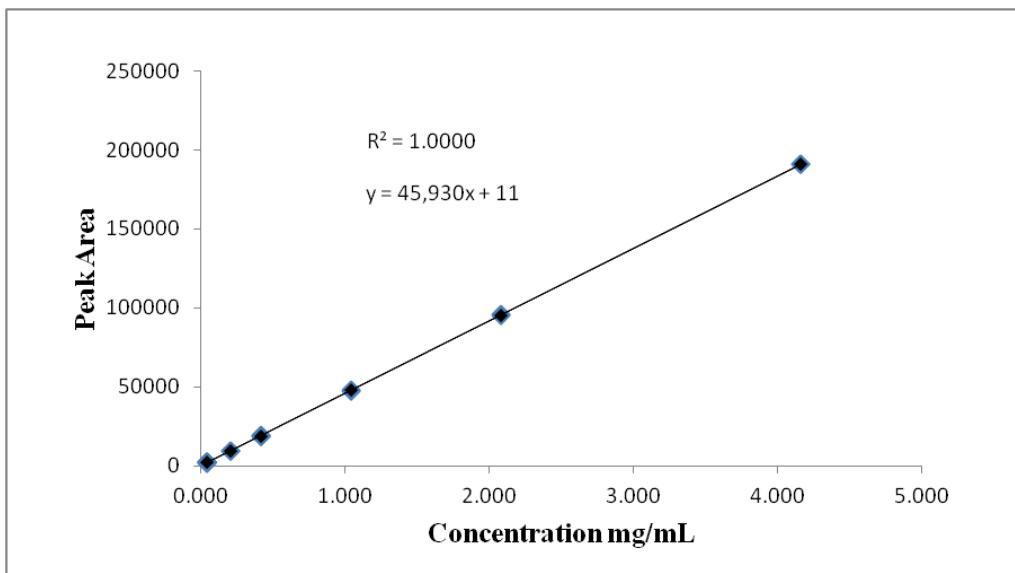


	Rep	Weight % ¹	Peak Area ²	Concentration (mg/mL)
TSN106191-Int-D4	1	0.007	1775	0.030
TSN106191-Int-D4	2	0.007	1817	0.030
TSN106191-Int-D4	3	0.007	1769	0.030
TSN106191-Int-D4	4	0.007	1754	0.030
TSN106191-Int-D4	5	0.006	1705	0.030
TSN106191-Int-D4	6	0.006	1709	0.030
Rec Lin - B	1	0.008	2088	0.040
Rec Lin - B	2	0.008	1978	0.040
Rec Lin - C	1	0.041	10434	0.201
Rec Lin - C	2	0.041	11167	0.201
Rec Lin - D	1	0.083	21279	0.401
Rec Lin - D	2	0.083	21280	0.401
Rec Lin - E	1	0.083	21021	0.401
Rec Lin - E	2	0.083	21226	0.401
Rec Lin - F	1	0.206	52659	1.003
Rec Lin - F	2	0.206	52351	1.003
Rec Lin - G	1	0.413	105496	2.007
Rec Lin - G	2	0.413	104835	2.007
Rec Lin - H	1	0.824	209916	4.014
Rec Lin - H	2	0.824	210125	4.014
Slope (m)=				52284
Y-intercept (b) =				170
Coefficient of determination (r2) =				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 7. Linearity of 3-chloro-1-methylcyclopentane (Q, X11726086)

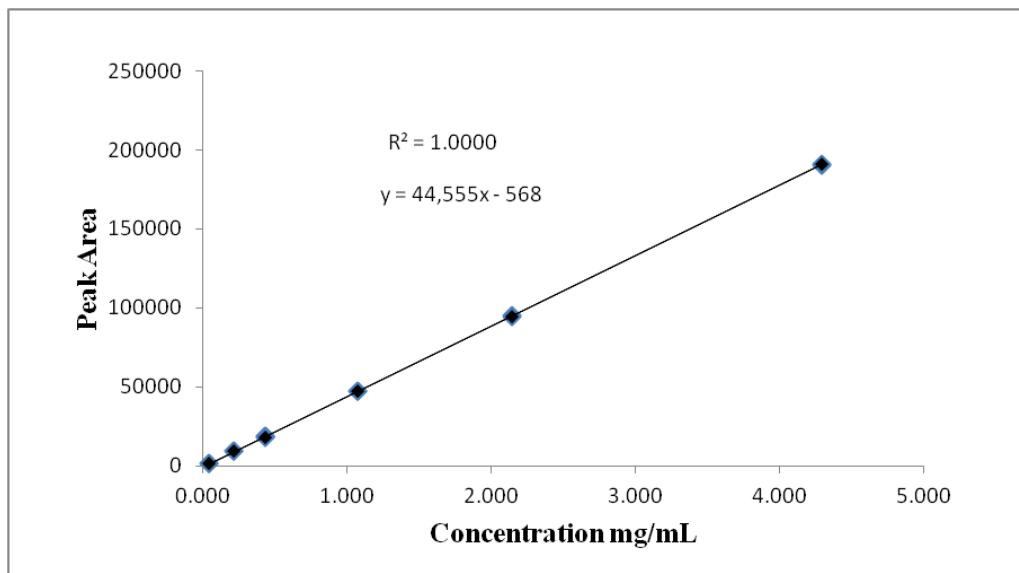


	Rep	Weight % ¹	Peak Area ²	Concentration (mg/mL)
TSN106191-Int-D3	1	0.009	2086	0.040
TSN106191-Int-D3	2	0.008	2004	0.040
TSN106191-Int-D3	3	0.008	1936	0.040
TSN106191-Int-D3	4	0.009	2142	0.040
TSN106191-Int-D3	5	0.008	1877	0.040
TSN106191-Int-D3	6	0.009	2106	0.040
Rec Lin - B	1	0.009	1679	0.042
Rec Lin - B	2	0.009	1862	0.042
Rec Lin - C	1	0.043	9509	0.208
Rec Lin - C	2	0.043	9333	0.208
Rec Lin - D	1	0.086	18984	0.416
Rec Lin - D	2	0.086	19344	0.416
Rec Lin - E	1	0.086	18939	0.416
Rec Lin - E	2	0.086	18895	0.416
Rec Lin - F	1	0.214	47475	1.039
Rec Lin - F	2	0.214	47792	1.039
Rec Lin - G	1	0.427	95662	2.078
Rec Lin - G	2	0.427	95221	2.078
Rec Lin - H	1	0.854	191248	4.156
Rec Lin - H	2	0.854	190649	4.156
Slope (m)=				45930
Y-intercept (b) =				11
Coefficient of determination (r2) =				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 8. Linearity of 1-chloro-1-methylcyclopentane (R, X11726078)

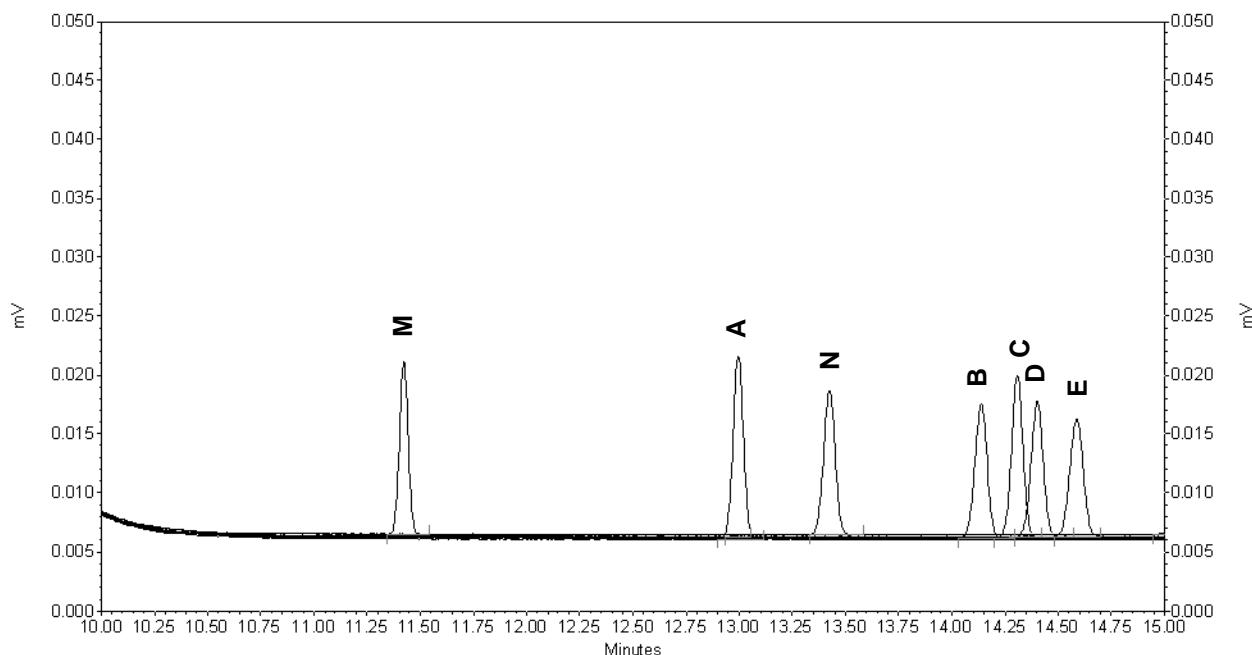


	Rep	Weight % ¹	Peak Area ²	Concentration (mg/mL)
TSN106191-A2	1	0.004	895	0.015
TSN106191-A2	2	0.004	908	0.015
TSN106191-A2	3	0.004	959	0.015
TSN106191-A2	4	0.004	915	0.015
TSN106191-A2	5	0.003	733	0.015
TSN106191-A2	6	0.003	734	0.015
Rec Lin - B	1	0.009	1671	0.043
Rec Lin - B	2	0.009	1756	0.043
Rec Lin - C	1	0.044	9068	0.214
Rec Lin - C	2	0.044	9106	0.214
Rec Lin - D	1	0.088	18510	0.429
Rec Lin - D	2	0.088	18365	0.429
Rec Lin - E	1	0.088	18393	0.429
Rec Lin - E	2	0.088	18245	0.429
Rec Lin - F	1	0.221	46991	1.072
Rec Lin - F	2	0.221	46941	1.072
Rec Lin - G	1	0.441	95207	2.145
Rec Lin - G	2	0.441	94804	2.145
Rec Lin - H	1	0.881	190532	4.289
Rec Lin - H	2	0.881	190690	4.289
Slope (m)=				44555
Y-intercept (b) =				-568
Coefficient of determination (r2) =				1.0000

1 - Weight percent is equivalent weight percent for Rec/Lin samples and actual weight % for LOQ samples

2 - Area is corrected area for Rec/Lin samples and uncorrected area for LOQ samples

Figure 9. Chromatograms of Impurities from Interferences Evaluation (Region 1, 10-15 min)



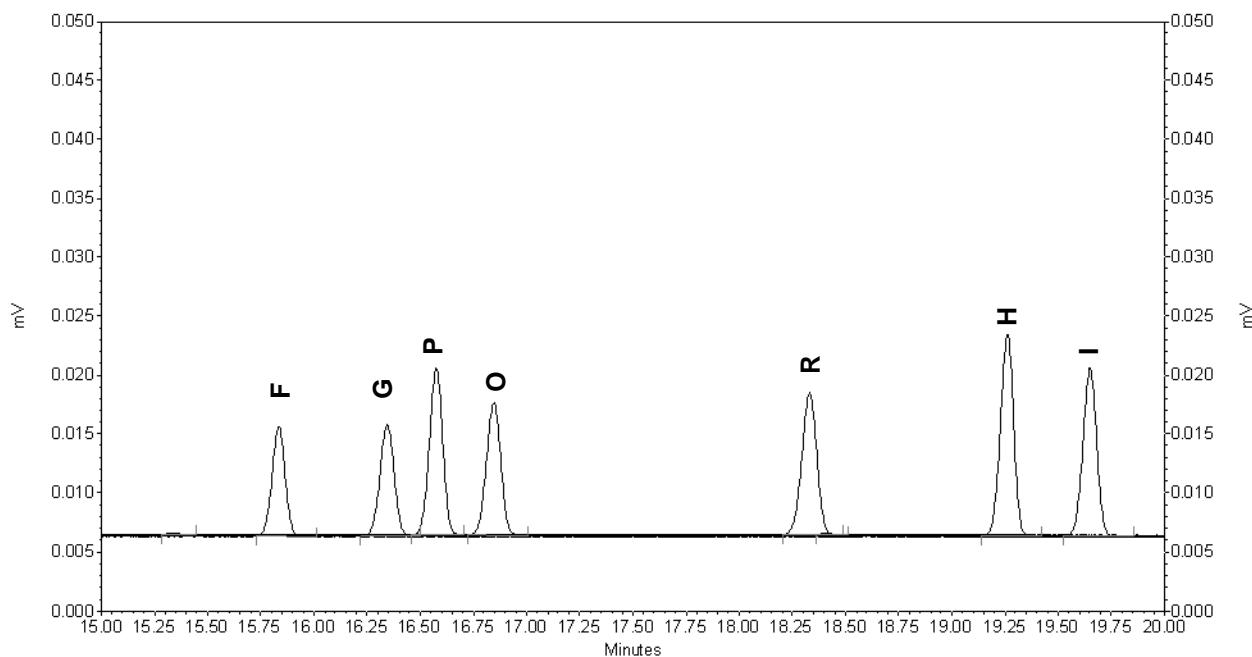
Data path: \\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Interferences\

Trace	Sample	Filename
M	3,3-dichloro-1-propene (M)-Int (TSN030579-0001)	014_3,3-dichloro-1-propene M-Int.dat
A	1,2-dichloropropane (A)-Int (AGR277102)	002_1,2-dichloropropane A-Int.dat
N	4-chloro-4-methyl-1-pentene (N)-Int (TSN303759)	016_4-chloro-4-methyl-1-pentene O-Int.dat**
B	2-chloro-2-methylpentane (B)-Int (AGR238091)	003_2-chloro-2-methylpentane B-Int.dat
C*	2-chloro-1,5-hexadiene (C)-Int (TSN030278-0001)	004_2-chloro-1,5-hexadiene C-Int.dat
D	2-chloro-4-methylpentane (D)-Int (TSN106505)	005_2-chloro-4-methylpentane D-Int.dat
E	2-chloro-2,3-dimethylbutane (E)-Int (TSN028018-0001)	006_2-chloro-2,3-dimethylbutane E-Int.dat

* 2-chloro-1,5-hexadiene (Impurity C) is analyzed in DAS-AM-05-10 due to incomplete resolution from impurity D using the chromatography conditions in this method.

**The incorrect peak ID was used in the filename.

Figure 10. Chromatograms of Impurities from Interferences Evaluation (Region 2, 15-20 min)

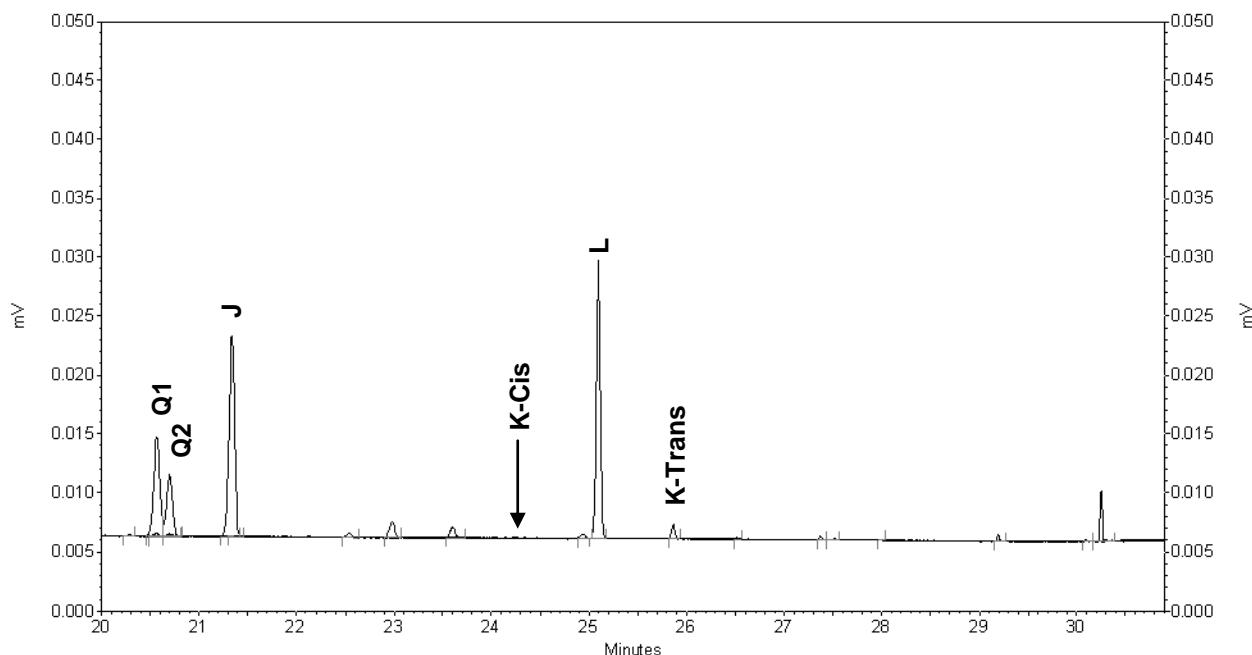


Data path: \\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Interferences\

Trace	Sample	Filename
F	3-chloro-1,5-hexadiene (F)-Int (TSN303599)	007_3-chloro-1,5-hexadiene F-Int.dat
G	3-chloro-2-methylpentane (G)-Int (TSN106504)	008_3-chloro-2-methylpentane G-Int.dat
P	1-chloro-1,5-hexadiene (P)-Int (TSN304464)	017_1-chloro-1,5-hexadiene P-Int.dat
O	5-chloro-1-hexene (O)-Int (TSN106329)	015_5-chloro-1-hexene N-Int.dat*
R	1-chloro-1-methylcyclopentene (R)-Int (TSN303032)	019_1-chloro-1-methylcyclopentene R-Int.dat
H	1,3-dichloropropane (H)-Int (AGR238090)	009_1,3-dichloropropane H-Int.dat
I	1,2,2-trichloropropane (I)-Int (TSN301451)	010_1,2,2-trichloropropane I-Int.dat

*The incorrect peak ID was used in the filename.

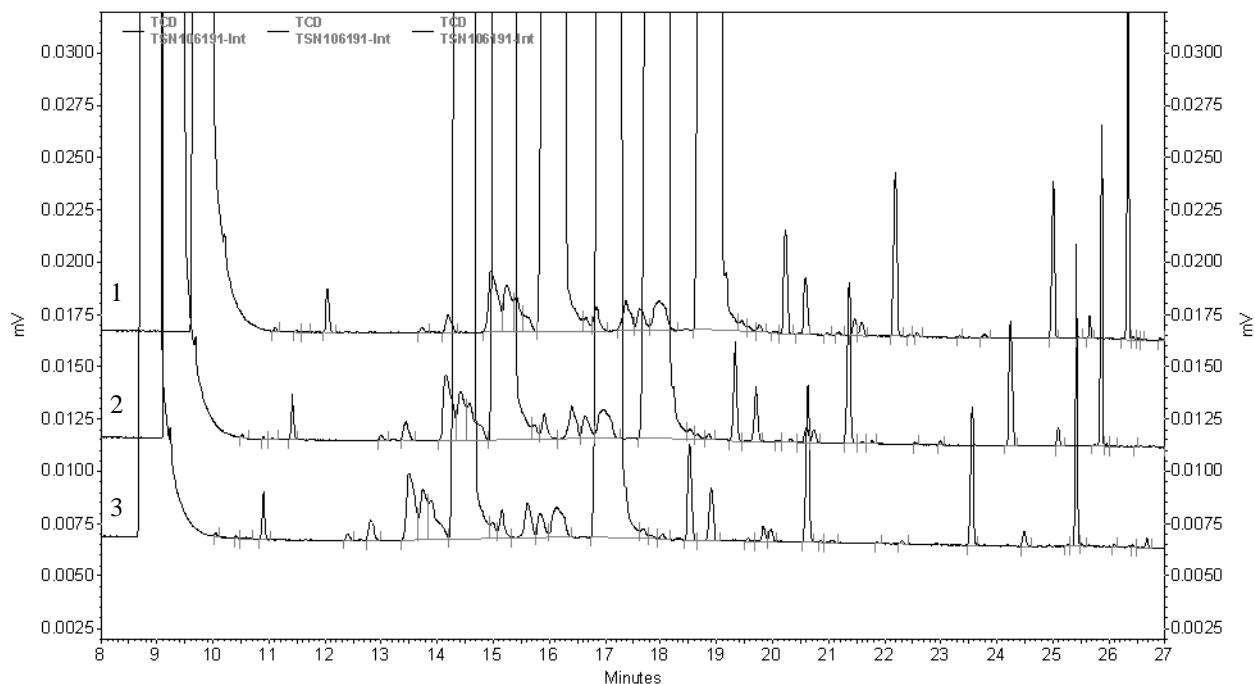
Figure 11. Chromatograms of Impurities from Interferences Evaluation (Region 3, 20-30.9 min)



Data path: \\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\Interferences\

Trace	Sample	Filename
Q	3-chloro-1-methylcyclopentene (Q)-Int (TSN303341)	018_3-chloro-1-methylcyclopentene Q-Int.dat
J	Cis-1,3,3-trichloropropene (J)-Int (AGR238088)	011_Cis-1,3,3-trichloropropene J-Int.dat
K	Cis/Trans-2-chloro-3(chloromethyl)oxirane (K)-Int (TSN106513)	012_Cis/Trans-2-chloro-3(chloromethyl)oxirane K-Int.dat
L	Trans-1,3,3-trichloropropene (L)-Int (AGR238086)	013_Trans-1,3,3-trichloropropene L-Int.dat

Figure 12. Chromatograms of Telone II TGAI from Ruggedness Evaluation



Data path: \\elntrd12\EZChrom\Projects\202gc104\Das-AM\Das-AM-G-13-48\

Trace	Sample	Filename
1	Telone II TGAI at 1.7 mL/min (TSN106191)	Ruggedness\1\020_TSN106191-Int.dat
2	Telone II TGAI at 1.9 mL/min (TSN106191)	Interferences\020_TSN106191-Int.dat
3	Telone II TGAI at 2.1 mL/min (TSN106191)	Ruggedness\2\020_TSN106191-Int.dat

APPENDIX I. ANALYTICAL METHOD SUMMARY

Analytical Method Summary – Analytical Method and Validation for the Determination of Impurities in Telone II Technical Grade Material – Method NA-AM-98-081.00 Extension

1. Preparation of calibration solutions

Prepare a solution of the reference substances in duplicate by accurately weighing approximately 50 mg into a 50-mL volumetric flask (record weight to the nearest 0.1 or 0.01 mg). Dilute to the mark with ethyl acetate to afford a mixed standard containing all impurities.

2. Preparation of sample solutions

Add 2mL of Telone II into a 5 mL volumetric flask (record weight to the nearest 0.1 or 0.01 mg) and dilute to volume with ethyl acetate. Mix well.

3. Instrumentation and Conditions:

GC analysis conditions:

Column:	DB-1701 60 m x 0.32 mm x 1 μ m	
Inlet temperature:	150°C	
Split ratio:	38:1	
Injection volume:	2 μ L	
Column flow:	Constant flow at 1.9 mL/min Helium	
Column temperature:	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	
Detector:	TCD	
Detector temperature:	280°C	
Detector flow rate:	Reference Flow (Helium): 15 mL/min Make-up (Helium): 5 mL/min	
Run Time:	30.9 minutes	
Integrator:	Agilent EZChrom	
Retention Times:	3,3-dichloro-1-propene	~ 11.4 min
	4-chloro-4-methyl-1-pentene	~ 13.4 min
	Cis-1,3-dichloropropene	~ 15.4 min
	Trans-1,3-dichloropropene	~ 16.6 min
	1-chloro-1,5-hexadiene	~ 16.9 min
	5-chloro-1-hexene	~ 18.1 min
	1-chloro-1-methylcyclopentane	~ 18.5 min
	3-chloro-1-methylcyclopentane	~ 20.6 min

Approximate time to prepare and analyze sample: 6 hours

4. Calculations:

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

NOTE: Calculations may be performed as described below (using sample weights and a dilution factor) or using sample concentrations as described in NA-AM-98-081.00.

A. Calculation of the weight of the impurity in the calibration solutions:

$$Wt_x = Wt_{AS} \times \text{Purity}_{AS}$$

where:

Wt_x = Weight of the impurity in the Calibration Standard Solution

Wt_{RS} = Weight of the reference substance added to the calibration solution

Purity_{RS} = Purity of the Reference Substance, expressed as a decimal

B. Calculation of the response factor for the impurity in the calibration solutions:

$$Rf = \frac{Wt_x}{A_x}$$

where:

Rf = Response factor

Wt_x = Weight of impurity in stock standard solution

A_x = Area of impurity peak obtained during analysis of the calibration solution

C. Calculation of the weight % of impurity in the sample:

$$Wt\%_x = \frac{A_x \times RF_{(avg\ x)}}{Wt_{sample} \times \text{Dilution factor}} \times 100$$

where:

$Wt\%_x$ = Weight % of impurity in the sample

A_x = Area of impurity obtained during analysis of the sample solution

$RF_{(avg\ x)}$ = Average response factor for impurity

Wt_{sample} = Weight of sample in sample solution

Dilution factor = Dilution factor to account for the difference in volume between the standards and samples (e.g. 10 when standards are prepared in a 50 mL volumetric flask and samples are prepared in a 5 mL volumetric flask)

Example chromatograms are attached.

Additional details are provided in the body of the report.

Figure 1. Representative Chromatogram of a Calibration Solution

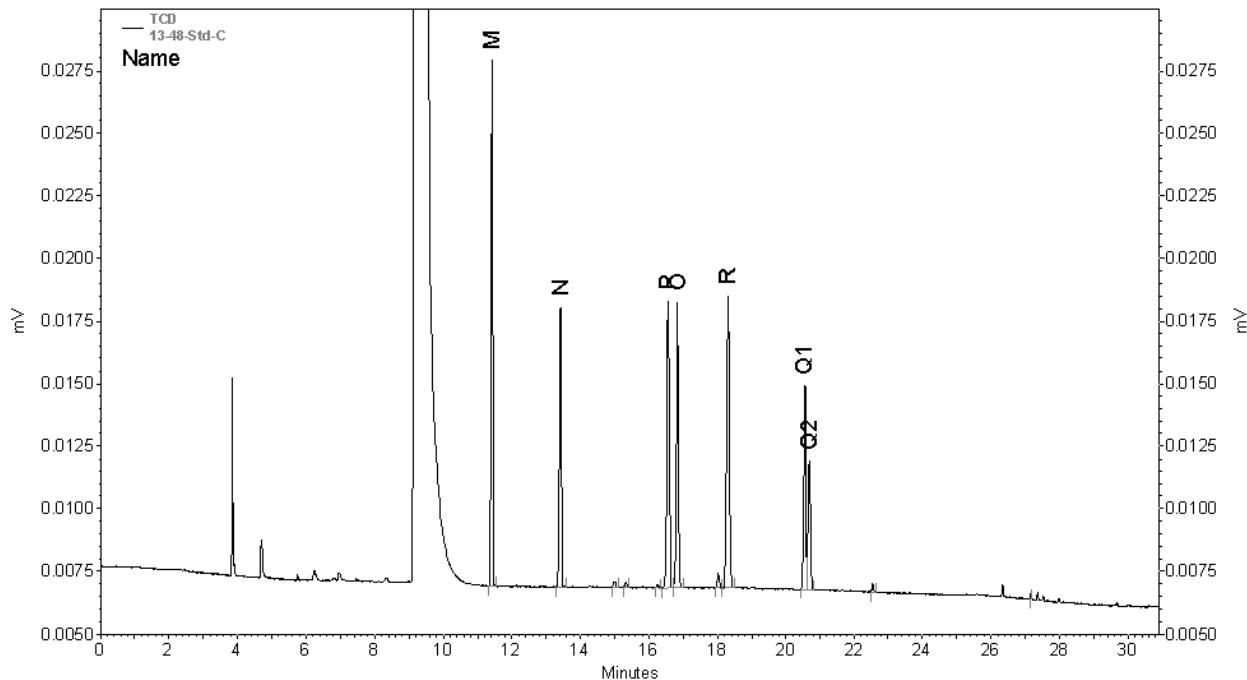


Figure 2. Representative Chromatogram of a Sample Solution of Telone II TGAI

