



256867

# DERBI Cover Page

## Dow AgroSciences Confidential

Document Type	Scientific Area	Document Status	PTR Number	Pages	Date Report Issued
REG	Analytical	NEW	none	32	1/28/08

## Report Title

Analytical Method and Validation for the Determination of Epoxide Impurities in Telone II Technical; Method NA-AM-98-081 Extension

## Author(s) and ID's

Amy Latham

## Department

Crop Protection R&amp;D

Report / File Number(s)

DAS-AM-06-045

## Materials

DED Code	Formulation No.	Formulation Type	Product Name	Active Ingredient(s)
			TELONE II	1,3-D

## Keywords

EPOXIDE METHOD

Compound Number(s)	Batch/Lot Number(s)	Bayer Codes

## Lab Notebooks

## Language(s)

## Performing Laboratory Name

	ENGLISH	DAS-INDIANAPOLIS
--	---------	------------------

## Data Requirement(s) (Guidelines)

--

Geo Area and Country (s)  
of Study

## Protocol Number(s)

## Study Number(s)

## Published?

## Date Study Completed

NA				YES	1/28/08
				Vertebrate?	GLP?
				N/A	YES

## Method Number

## Method Division

## Method Status

## Validated?

DAS-AM-06-045	N/A	N/A	N/A
---------------	-----	-----	-----

## Analyte

## Method Type

## SMC Code(s)

## Enforcement Method?

	N/A		N/A
--	-----	--	-----

## Method Technique(s)

--

## Reviewer(s)

--

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 Epoxide Impurities in Telone II  
Technical; Method NA-AM-98-081 Extension

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham

STUDY COMPLETED ON

January 28, 2008

PERFORMING LABORATORY

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

LABORATORY STUDY ID

DAS-AM-06-045

## SUMMARY

This report describes the validation of an analytical method for determination of cis and trans epoxide impurities 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 Epoxide Impurities in Telone II  
Technical; Method NA-AM-98-081 Extension

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

A. L. Latham  
317-337-3582

STUDY COMPLETED ON

January 28, 2008

PERFORMING LABORATORY

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

LABORATORY STUDY ID

DAS-AM-06-045

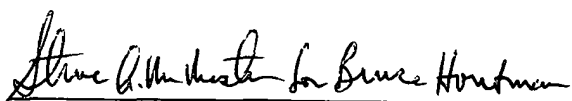
## STATEMENT OF DATA CONFIDENTIALITY CLAIMS

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: 3/8/07

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Study Initiation Date: October 3, 2006

Experimental Start Date: November 21, 2006

Experimental End Date: January 24, 2007

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

Steve Q. Houtman for Bruce Houtman

Bruce Houtman  
Sponsor  
Dow AgroSciences LLC

3/8/07

Date

Steve Q. Houtman for Bruce Houtman

Bruce Houtman  
Submitter  
Dow AgroSciences LLC

3/8/07

Date

Amy Latham

Amy Latham  
Study Director  
Dow AgroSciences LLC

1/28/08

Study Completion Date

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

**Compound:** Epoxide Impurities

**Study ID:** DAS-AM-06-045

**Title:** Analytical Method and Validation for the Determination of Epoxide Impurities in  
Telone II Technical; Method NA-AM-98-081 Extension

**Study Initiation Date:** 3 October 2006

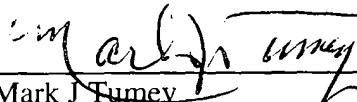
**Study Completion Date:** 28 January 2008

**GLP Quality Assurance Inspections**

<b>Date of GLP Inspection(s)</b>	<b>Date Reported to the Study Director and to Management</b>	<b>Phases of the Study which received a GLP Inspection by the Quality Assurance Unit</b>
2 October 2006	3 October 2006	Protocol review
21,22 November 2006	27 November 2006	Preparation of Recovery Samples for Analysis
25,26,28 March 2007 & 3,4 April 2007	12 April 2007	Report and Raw Data

**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.

  
\_\_\_\_\_  
Mark J. Tumey  
Dow AgroSciences, Quality Assurance

28 - JAN - 2008  
\_\_\_\_\_  
Date

SIGNATURE PAGE



Amy Latham  
Author  
Dow AgroSciences LLC

1/28/08

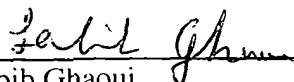
Date



Rose Nelson  
Reviewer  
Dow AgroSciences LLC

9 March 6, 2007

Date



Labib Ghaoui  
Analytical Leader  
Dow AgroSciences LLC

March 7, 2007

Date



Title: Analytical Method and Validation for the Determination of Epoxide Impurities in Telone  
II Technical; Method NA-AM-98-081 Extension

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

CONFIDENTIAL ATTACHMENT

STUDY TITLE

Analytical Method and Validation for the Determination of Epoxide Impurities in Telone II  
Technical; Method NA-AM-98-081 Extension

DATA REQUIREMENT

U.S. EPA OPPTS Test Guideline 830.1800

STUDY DIRECTOR

Amy L. Latham  
317-337-3582

STUDY COMPLETED ON

January 28, 2008

PERFORMING LABORATORY

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

LABORATORY STUDY ID

DAS-AM-06-045

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>
6	Process impurities identified	§10(d)(1)(A)

## TABLE OF CONTENTS

	<u>Page</u>
I. ABSTRACT.....	4
II. INTRODUCTION .....	5
A. Scope.....	5
B. Principle .....	5
III. MATERIALS AND METHODS.....	5
A. Equipment .....	5
B. Reagents and Standards .....	5
C. Safety .....	5
D. Analytical Procedures .....	6
E. Instrumentation .....	7
F. Methods of Calculation.....	7
IV. RESULTS AND DISCUSSION .....	8
A. Linearity.....	8
B. Accuracy .....	8
C. Method Precision .....	9
D. System Precision.....	9
E. Solution Stability .....	9
F. Interferences.....	9
G. Ruggedness .....	9
H. Limit of Quantitation (LOQ) and Limit of Detection (LOD).....	10
V. CONCLUSIONS.....	10
VI. TABLES .....	11
VII. FIGURES .....	15
VIII. APPENDIX.....	22

## I. ABSTRACT

This report describes the validation of an analytical method for determination of cis and trans epoxide impurities 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 0.068 to 0.192 wt. % cis epoxide and 0.076 to 0.214 wt. % trans epoxide in Telone II. The average recovery for cis epoxide in Telone II was 103%, with a relative standard deviation of 8.23%. The average recovery for trans epoxide in Telone II was 108%, with a relative standard deviation of 7.52%. Detector response was shown to be linear for cis epoxide over a range of 0.068 to 0.192 wt. % cis epoxide and 0.076 to 0.214 wt. % trans epoxide in Telone II.

Replicate analyses of Telone II technical formulation on two separate days gave a relative standard deviation of 4.13% for the cis epoxide and 3.88% for the trans epoxide at an average concentration of 0.070 wt. % and 0.075 wt. %, respectively, in Telone II. The analysis is complete in approximately 30 minutes.

## II. INTRODUCTION

### A. Scope

This GC method is applicable to the determination of cis and trans epoxide impurities in Telone II technical formulation. The method was evaluated over the range 0.023 to 0.192 wt. % cis epoxide and 0.026 to 0.214 wt. % trans epoxide in Telone II and was found to be acceptable over the range 0.068 to 0.192 wt. % cis epoxide and 0.076 to 0.214 wt. % trans epoxide 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: Agilent EZChrom Elite.
4. GC Column J & W Scientific DB-1701, 60 m x 0.32 mm x 1  $\mu$ m
5. Autosampler vials and caps: 1.5 mL with screw caps
6. Miscellaneous laboratory glassware.

### B. Reagents and Standards:

1. Test and Reference Substances: cis Telone epoxide, TSN105478, 91.8%, recertification date January 27, 2008 and trans Telone epoxide, TSN105479, 92.9%, recertification date January 27, 2008 .
2. Ethyl acetate: Mallinckrodt, or equivalent.
3. Test systems:  
Telone II technical formulation blank: TSN105896  
Telone II technical formulation blank: TSN105894

### C. Safety

Each analyst should be acquainted with potential hazards of the reagents, products and solvents before beginning laboratory work. Sources of information include: material safety

data sheets, literature and other related data. Disposal of reagents, reactants, and solvents must be in compliance with local, state and federal laws and regulations.

#### D. Analytical Procedures

##### 1. Preparation of calibration solution:

Prepare calibration solutions by weighing accurately, approximately 50 mg of cis epoxide and trans epoxide reference standards 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 cis epoxide and trans epoxide 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 (designated TSN105896S) was prepared by adding 127.2 mg cis epoxide and 139.9 mg trans epoxide to 10 mL Telone II blank.

Recovery samples containing cis and trans epoxides were prepared for Telone II by weighing approximately 4.9 g aliquots of the Telone II (TSN105896) into 10 mL volumetric flasks (Table I). Volumetric aliquots of cis and trans epoxides were then added to each sample via the stock solution TSN105896S of cis and trans epoxides in Telone II, and contents were prepared for GC analysis by adding filling to the mark with 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 approximately 2.4 g aliquots of Telone II (fortified with cis and trans epoxides to ensure technical formulations were in the appropriate range for analysis) into 5 mL volumetric flasks and filling to the mark with ethyl acetate. 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 minute  
25°C/minute to 270°C, hold for 0 minutes  
Injection port: Split at 150°C with a split 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: cis epoxide ~ 23.8 minutes  
trans epoxide ~25.6 minutes

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

F. Methods of Calculation

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

1. Calculation of the response factor for cis epoxide in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(\text{cis epoxide})} \times 50 \text{ mL}}$$

where:

RF = Response factor for cis epoxide  
mg reference std = Weight of cis epoxide reference standard in calibration solution, mg  
P = Purity of reference standard, expressed as a fraction  
Area<sub>(cis epoxide)</sub> = Peak area for cis epoxide in calibration solution



2. Calculation of the weight % of cis epoxide in the sample:

$$\text{Weight \%} = \frac{\text{Area}_{(\text{cis epoxide})} \times \text{RF}}{\text{Sample wt}} \times 100$$

where: Weight % = Weight % of cis epoxide in the sample  
Area<sub>(cis epoxide)</sub> = Peak area for cis epoxide in the sample solution  
RF = Response factor calculated for cis epoxide  
Sample wt = Weight of sample expressed as a concentration (mg/mL)

Note that the calculations above are used to determine the response factor and weight percent of the trans epoxide by replacing all instances of "cis epoxide" with "trans epoxide" in the equations above.

#### IV. RESULTS AND DISCUSSION

##### A. Linearity

The linearity for cis epoxide was evaluated using the GC conditions used for this study. A linear relationship between peak area and concentration ( $r^2 = 0.9952$ ) was noted for cis epoxide from 0.068 wt. % to 0.192 wt. %. A linear relationship between peak area and concentration ( $r^2 = 0.9979$ ) was also noted for the trans epoxide from 0.076 wt. % to 0.214 wt. %. The linearity plots are shown in Figures 3 and 4. A linear relationship was not established for concentrations below the levels mentioned above.

##### 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 0.023 to 0.192 wt. % cis epoxide and 0.026 to 0.214 wt. % trans epoxide in Telone II.

The recovery for cis epoxide in Telone II ranged from 97 % to 122 %, with an average recovery of 103%, and a relative standard deviation of 8.23 %. Recovery data are shown in Table II. The recovery for trans epoxide in Telone II ranged from 97 % to 131 %, with an average recovery of 108%, and a relative standard deviation of 7.52 %. Recovery data are shown in Table III. 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 III 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 Tables IV and V.

The relative standard deviation for day 1 was 1.86 % at an average concentration of 0.067 wt. % cis epoxide in Telone II. The Horwitz RSD<sub>r</sub> value was calculated to be 4.02 for Telone II, therefore, results are acceptable (Table IV). The relative standard deviation for day 1 was 1.78 % at an average concentration of 0.073 wt. % trans epoxide in Telone II. The Horwitz RSD<sub>r</sub> value was calculated to be 3.98 for Telone II; therefore, results are acceptable (Table V). The RSD between day 1 and day 2 precision was 4.13% for cis epoxide and 3.88% trans epoxide.

### D. System Precision

System precision was determined by injecting a prepared solution of Telone II technical formulation five times. Data obtained are shown in Tables VI and Table VII. The relative standard deviation for the area of cis epoxide for all five injections of Telone II was 1.09%. The relative standard deviation for the area of trans epoxide for all five injections of Telone II was 2.37%.

### E. Solution Stability

The solution stability was determined by analyzing sample solutions prepared for the day one precision study four days after the initial analysis. The t-test was used to compare the results. The t-test results indicated that the results obtained four 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 for analysis the same day they are prepared.

### F. Interferences

No interferences were detected for the ethyl acetate solvent, technical formulation inert ingredients, or cis and trans epoxide impurities. Chromatograms of a solvent blank, cis and trans epoxide mixed standard and technical formulation blank for Telone II are shown in Figure 5.

### 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 6. No interferences were observed for any of the components.

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

Limit of quantitation was experimentally demonstrated by preparing five spiked samples using the same procedure as that used in the recovery study at a level of 0.068 wt. % cis epoxide and 0.072 wt. % trans epoxide and demonstrating acceptable precision and recovery on these samples.

Limit of detection (LOD) was determined by reviewing the lowest recovery samples, 0.023 wt. % cis epoxide and 0.026 wt. % trans epoxide, and determining that the epoxide impurities had a greater than three times signal to noise ratio. Additional samples were prepared at concentrations, 0.006 and 0.012 wt. % cis epoxide and 0.006 and 0.0125 wt. % trans epoxide, below the lowest recovery samples to demonstrate the lower limit of detection. These concentrations also had a greater than three times signal to noise ratio, as shown in Figure 7.

#### V. CONCLUSIONS

This method is applicable to the determination of cis and trans epoxide in Telone II technical formulation over a range of 0.068 to 0.192 wt. % cis epoxide and 0.076 to 0.214 wt. % trans epoxide in Telone II. The precision, recovery and linearity data have shown this method to be acceptable for the determination of cis and trans epoxide in Telone II technical formulation over these ranges. 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, linear regression, 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 epoxide added, mg		Weight of Telone II sample plus weight of spike, mg	Volume of spike solution added (uL)	Epoxide Wt. %	
	Cis	Trans			Cis	Trans
Recovery 1	1.2	1.3	4969.1	100	0.023	0.026
Recovery 2	2.3	2.6	5078.1	200	0.046	0.051
Recovery 3	3.5	3.9	5156.0	300	0.068	0.076
Recovery 4	4.7	5.2	5334.7	400	0.088	0.097
Recovery 5	5.8	6.5	5411.5	500	0.108	0.120
Recovery 6	9.3	10.4	5839.0	800	0.160	0.178
Recovery 7	11.7	13.0	6084.8	1000	0.192	0.214

Weight of epoxide added to sample =

$$\frac{\text{Wt. of epoxide in spike solution (mg)} \times \text{Purity} \times \text{vol of spike added (mL)}}{\text{Total volume of solution (mL)}}$$

Epoxide wt. % =

$$\frac{\text{Weight epoxide, mg}}{\text{Weight of Telone II sample plus weight of spike, mg}} \times 100$$

Table II. Recovery Data for Cis Epoxide in Telone II Technical Formulation

Sample	Cis Epoxide wt % added	Cis Epoxide wt % found	Average Recovery %
Recovery 1	0.023	0.028	122
Recovery 2	0.046	0.047	102
Recovery 3	0.068	0.068	99
Recovery 4	0.088	0.086	97
Recovery 5	0.108	0.112	103
Recovery 6	0.160	0.156	98
Recovery 7	0.192	0.195	101
Average			103
Std. Dev.			8.49
R.S.D.			8.23

Table III. Recovery Data for Trans Epoxide in Telone II Technical Formulation

Sample	Trans Epoxide wt % added	Trans Epoxide wt % found	Average Recovery %
Recovery 1	0.026	0.033	125
Recovery 2	0.051	0.057	111
Recovery 3	0.076	0.079	103
Recovery 4	0.097	0.098	101
Recovery 5	0.120	0.129	107
Recovery 6	0.178	0.186	104
Recovery 7	0.214	0.226	106
Average			108
Std. Dev.			8.12
R.S.D.			7.52

Table IV. Method Precision Data for Cis Epoxide in Telone II Technical Formulation

Day 1 Precision		Day 2 Precision	
Sample	Average Wt. % cis epoxide	Sample	Average Wt. % cis epoxide
TSN105894-Prec 1-1	0.068	TSN105894-Prec 2-1	0.072
TSN105894-Prec 1-2	0.066	TSN105894-Prec 2-2	0.072
TSN105894-Prec 1-3	0.068	TSN105894-Prec 2-3	0.073
TSN105894-Prec 1-4	0.067	TSN105894-Prec 2-4	0.073
TSN105894-Prec 1-5	0.069	TSN105894-Prec 2-5	0.073
Average	0.067	Average	0.073
Std. Dev.	0.0013	Std. Dev.	0.00050
R.S.D.	1.86	R.S.D.	0.69
Horwitz RSD <sub>R</sub>	6.00	Horwitz RSD <sub>R</sub>	5.94
Horwitz RSD <sub>r</sub>	4.02	Horwitz RSD <sub>r</sub>	3.98
RSD < Horwitz RSD <sub>r</sub> ?	Acceptable	RSD < Horwitz RSD <sub>r</sub> ?	Acceptable

Table V. Method Precision Data for Trans Epoxide in Telone II Technical Formulation

Day 1 Precision		Day 2 Precision	
Sample	Average Wt. % Trans epoxide	Sample	Average Wt. % Trans epoxide
TSN105894-Prec 1-1	0.071	TSN105894-Prec 2-1	0.077
TSN105894-Prec 1-2	0.073	TSN105894-Prec 2-2	0.079
TSN105894-Prec 1-3	0.072	TSN105894-Prec 2-3	0.078
TSN105894-Prec 1-4	0.073	TSN105894-Prec 2-4	0.078
TSN105894-Prec 1-5	0.075	TSN105894-Prec 2-5	0.079
Average	0.073	Average	0.078
Std. Dev.	0.0013	Std. Dev.	0.00062
R.S.D.	1.78	R.S.D.	0.79
Horwitz RSD <sub>R</sub>	5.94	Horwitz RSD <sub>R</sub>	5.88
Horwitz RSD <sub>r</sub>	3.98	Horwitz RSD <sub>r</sub>	3.94
RSD < Horwitz RSD <sub>r</sub> ?	Acceptable	RSD < Horwitz RSD <sub>r</sub> ?	Acceptable

Table VI. System Precision Data for Cis Epoxide

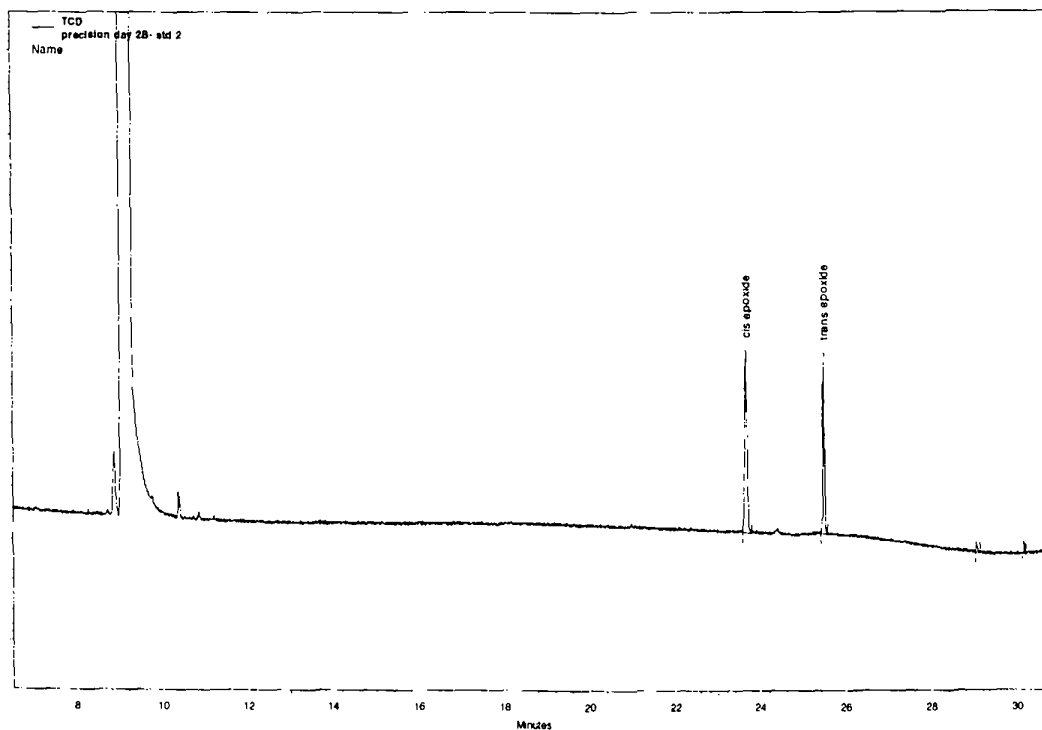
Sample	Peak Area
<b>Telone II</b>	
Injection #1	5339
Injection #2	5245
Injection #3	5303
Injection #4	5363
Injection #5	5397
Average	5329
Std. Dev.	58.33
R.S.D. (%)	1.09

Table VII. System Precision Data for Trans Epoxide

Sample	Peak Area
<b>Telone II</b>	
Injection #1	6011
Injection #2	5795
Injection #3	5687
Injection #4	5671
Injection #5	5748
Average	5782
Std. Dev.	137.03
R.S.D. (%)	2.37

## VII. FIGURES

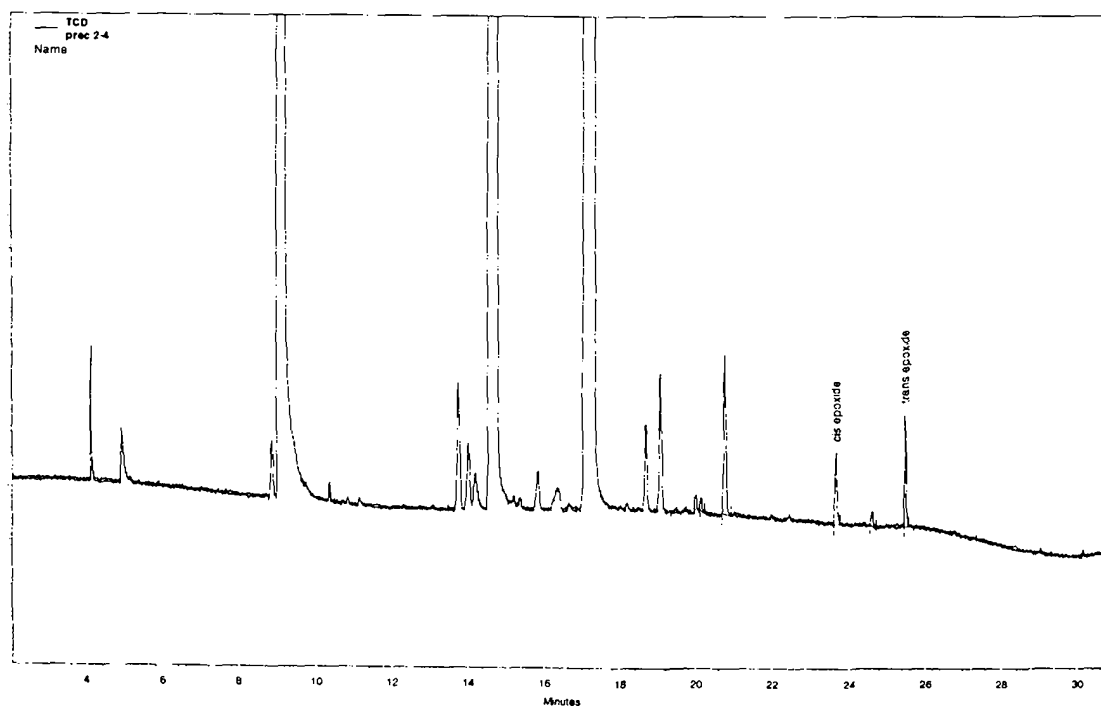
Figure 1. Chromatogram of a Calibration Solution



Datafile: \\Elntnd12\EZChrom\Projects\167gc055\DAS-AM-06-045\FOR-06-045\re-run data-Day2\precision day 2B-std 2-003-rep 2.dat

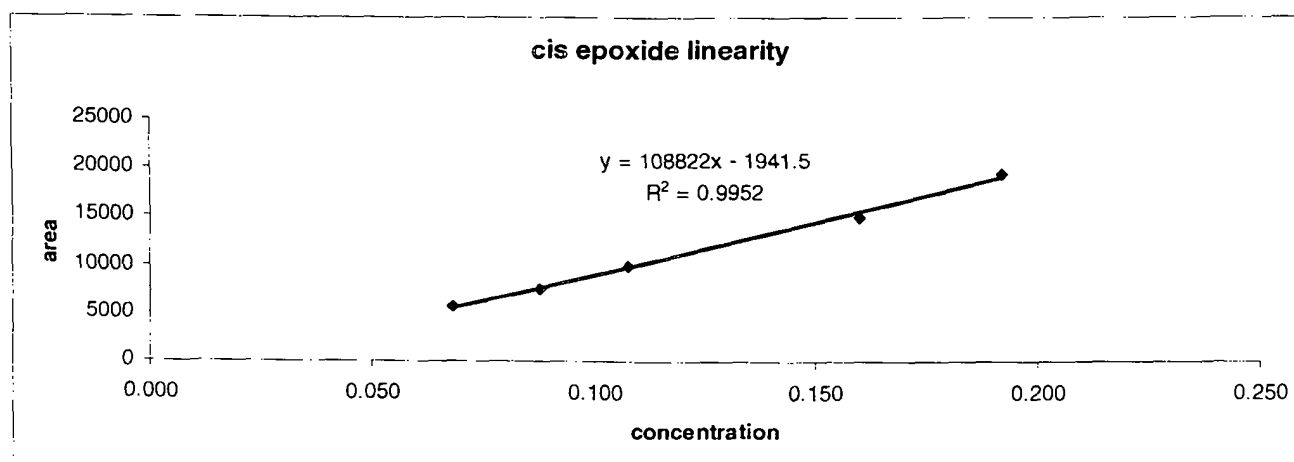


Figure 2. Chromatogram of a Sample Solution of Telone II Technical Formulation



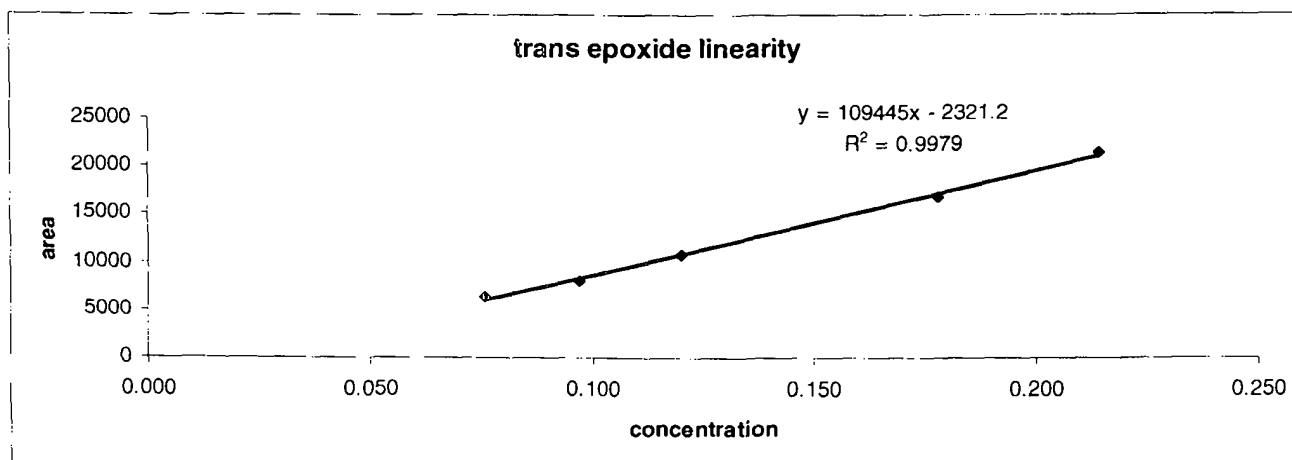
Datafile: \\Elntrd12\EZChrom\Projects\167gc055\ DAS-AM-06-045\FOR-06-045\re-run data-  
Day2\prec 2-4-007-rep 2.dat

Figure 3. Linearity Plot for Cis Epoxide



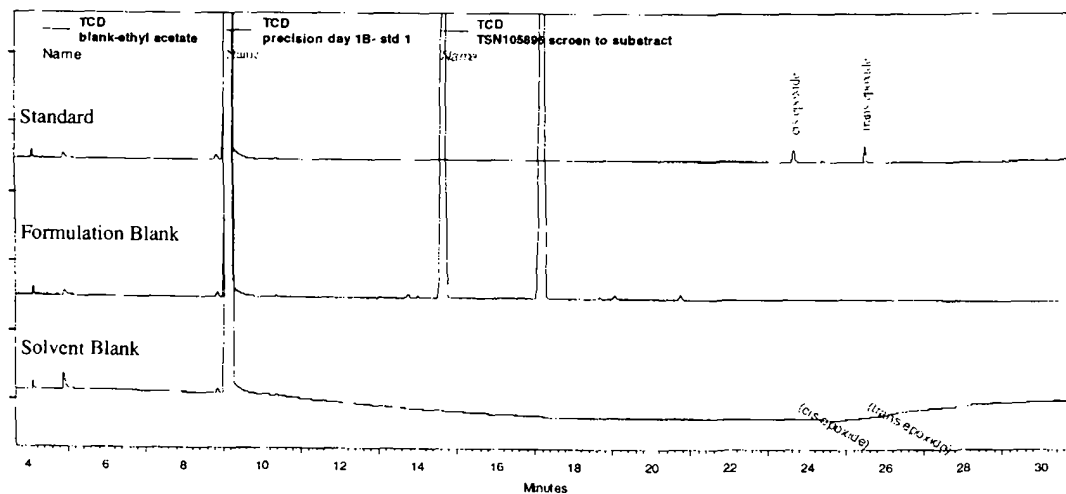
Concentration, <u>Wt. %</u>	Concentration, <u>mg/mL</u>	<u>Peak area</u>
0.068	0.35	5721
0.088	0.47	7466
0.108	0.58	9848
0.160	0.93	14905
0.192	1.17	19387

Figure 4. Linearity Plot for Trans Epoxide



Concentration, Wt. %	Concentration, mg/mL	Peak area
0.076	0.39	6286
0.097	0.52	8077
0.120	0.65	10789
0.178	1.04	16820
0.214	1.30	21392

Figure 5. Chromatograms of Cis and Trans Epoxide Standard, Telone II Technical Formulation Blank, and Solvent Blank



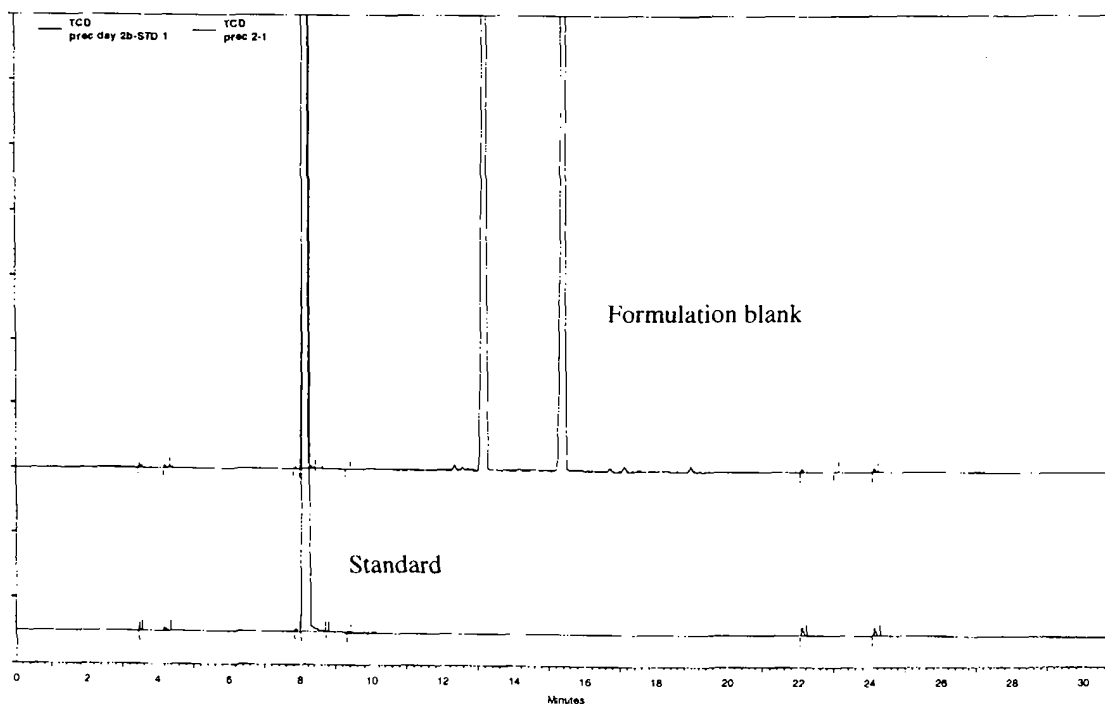
Datafiles:

Solvent Blank = \\Eltrd12\EZChrom\Projects\167gc055\ DAS-AM-06-045\FOR-06-045\re-run data \blank- ethyl acetate-001.dat

Standard = \\Eltrd12\EZChrom\Projects\167gc055\ DAS-AM-06-045\FOR-06-045\re-run data data\precision day 1B-std 1-003-rep 2.dat

Formulation Blank = \\Eltrd12\EZChrom\Projects\167gc055\ DAS-AM-06-045\FOR-06-045\re-run data \TSN 105896 screen to subtract-012-rep 2.dat

Figure 6. Chromatograms for Method Ruggedness of Telone II Technical Formulation Blank and Cis and Trans Epoxide Standard

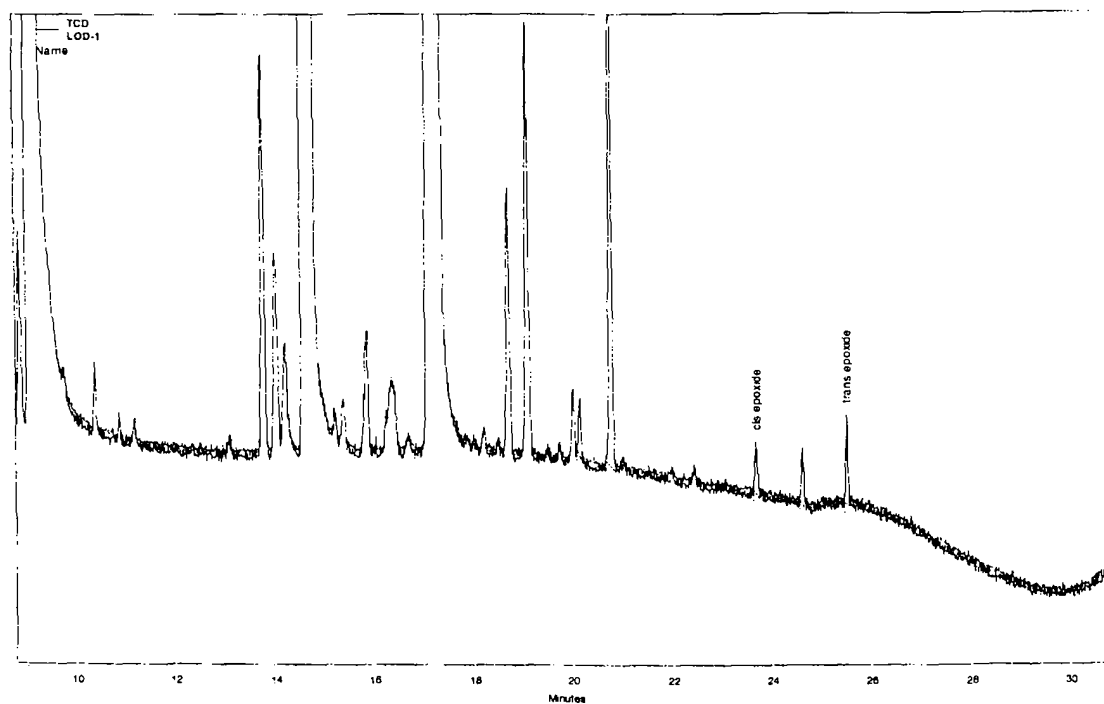


Datafiles:

Standard = \\Elntd12\EZChrom\Projects\167gc055\DAS-AM-06-045\FOR-06-045\ruggedness\prec day 2b-STD 1-002-rep 1.dat

Formulation Blank = \\Elntd12\EZChrom\Projects\167gc055\DAS-AM-06-045\FOR-06-045\ruggedness\prec 2-1-003-rep 2.dat

Figure 7. LOD Chromatogram



Datafile: \\Elntnd12\EZChrom\Projects\167gc055\DAS-AM-06-045\FOR-06-045\re-run  
data\LOD-1-018-Rep 1.dat

## VIII. APPENDIX

### Analytical Method Summary

#### A. Preparation of calibration solution:

Prepare calibration solutions by weighing approximately 50 mg of cis epoxide reference standard and approximately 50 mg of trans epoxide 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 1 $\mu$ m
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 split 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 $\mu$ L
Run Time:	30.9 minutes
Retention Time:	cis epoxide ~ 23.8 minutes trans epoxide ~25.6 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 cis epoxide in the calibration solution:

$$RF = \frac{\text{mg reference std} \times P}{\text{Area}_{(\text{cis epoxide})} \times 50 \text{ mL}}$$

where:

- RF = Response factor for cis epoxide
- mg reference std = Weight of cis epoxide reference standard in calibration solution, mg
- P = Purity of reference standard, expressed as a fraction
- Area<sub>(cis epoxide)</sub> = Peak area for cis epoxide in calibration solution

2. Calculation of the weight % of cis epoxide in the sample:

$$\text{Weight \%} = \frac{\text{Area}_{(\text{cis epoxide})} \times RF}{\text{Sample wt}} \times 100\%$$

where:

- Weight % = Weight % of cis epoxide in the sample
- Area<sub>(cis epoxide)</sub> = Peak area for cis epoxide in the sample solution
- RF = Response factor calculated for cis epoxide
- Sample wt = Weight of sample in mg/mL

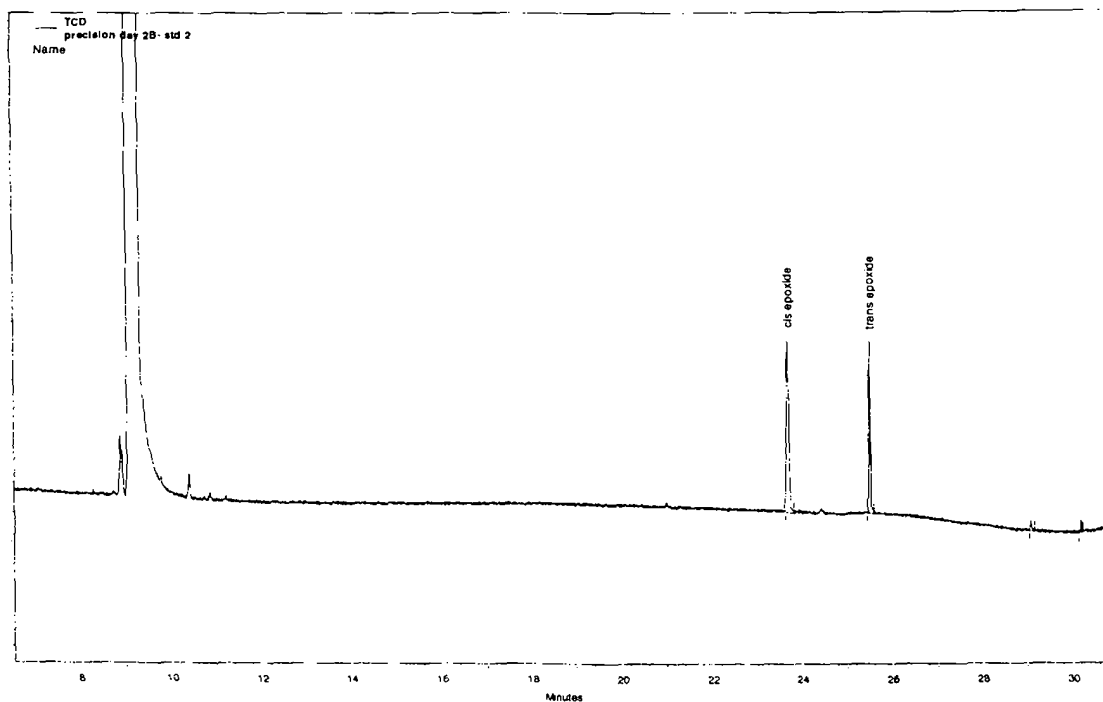
Note that the calculations above are used to determine the response factor and weight percent of the trans epoxide by replacing all instances of "cis epoxide" with "trans epoxide" in the equations above.

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



### Chromatogram of a Sample Solution of Telone II Technical Formulation

