SUMMARY

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

STUDY TITLE

Analytical Method and Validation for the Determination of Process Impurities in Telone* II by Gas Chromatography

DATA REQUIREMENT

U.S. EPA Product Chemistry
Guideline OPPTS 830.1800- Enforcement Analytical Method

STUDY DIRECTOR

Amy J. Smith

STUDY COMPLETED ON

27-AUG-1999

PERFORMING LABORATORY

Dow AgroSciences LLC
Formulation Science and Technology
9330 Zionsville Road
Indianapolis, Indiana 46268

LABORATORY PROJECT ID

NA-AM-98-081.00

*Trademark of Dow AgroSciences LLC

## SUMMARY

An analytical method was validated for the determination of weight percent of impurities in Telone* II by gas chromatography (GC) with a DB-1701 column and TCD detection. Quantitation of the impurities was achieved by comparing the detector response of the sample to that of a standard using external standard calibration.

*Trademark of Dow AgroSciences LLC

TABLE OF CONTENTS

| | | Page |
|---|---|---|
| I. | ABSTRACT | 7 |
| II. | INTRODUCTION | 8 |
| | A. Scope | 8 |
| | B. Principle | 8 |
| III. | MATERIALS AND METHODS | 8 |
| | A. Safety | 8 |
| | B. Equipment and Materials | 9 |
| | C. Reagents and Standards | 9 |
| | D. Gas Chromatographic Conditions | 10 |
| | E. Preparation of Standard Solutions for Calibration, and Recovery and Linearity Studies | 10 |
| | F. Preparation of Linearity Samples | 11 |
| | G. Preparation of Recovery Samples | 11 |
| | H. Preparation of Impurity Precision Samples | 12 |
| | I. Preparation of System Precision Sample | 12 |
| | J. Method Ruggedness | 12 |
| | K. Methods of Calculation | 12 |
| IV. | RESULTS AND DISCUSSION | 14 |
| | A. Interferences | 14 |
| | B. Relative Response Factors | 14 |
| | C. Recovery (Accuracy) | 14 |
| | D. Precision | 15 |
| | E. Linearity | 16 |
| | F. Stability | 16 |
| | G. Method Ruggedness | 16 |
| | H. Method Limitation and Notes | 17 |
| V. | CONCLUSIONS | 17 |

TABLE OF CONTENTS (continued)

| | | Page |
|---|---|---|
| VI. | TABLES | 19 |
| Table I: | Summary of Telone and Impurity Nomenclature Cross-Reference | 19 |
| Table II: | Preparation of Individual Purity Solutions and Summary of Response Factors and Retention Times | 20 |
| Table III: | Preparation of a Typical Impurity Standard | 21 |
| Table IV: | Preparation of Linearity Samples and Concentrations of Each Impurity Present | 22 |
| Table V: | Preparation of Linearity Samples and Concentrations for TSN101695 (2-Chloro-2-methylpentane and 3-Chloro-2-methylpentane | 23 |
| Table VI: | Stock Solutions used in Preparation of Recovery Samples | 24 |
| Table VII: | Preparation of Recovery Samples | 25 |
| Table VIII: | Stock Solutions and Preparation of Additional Recovery Samples | 26 |
| Table IX: | Preparation of Recovery Samples of TSN101695 (2-Chloro-2-methylpentane and 3-Chloro-2-methylpentane) | 27 |
| Table X: | Preparation of Recovery Samples of TSN101687 | 28 |
| Table XI: | Recovery Data for 2-Chloro-2-methylpentane in Synthetic Samples | 29 |
| Table XII: | Recovery Data for 2-Chloro-4-methylpentane in Synthetic Samples | 30 |
| Table XIII: | Recovery Data for 2-Chloro-2,3-dimethylbutane in Synthetic Samples | 31 |
| Table XIV: | Recovery Data for 3-Chloro-2-methylpentane in Synthetic Samples | 32 |
| Table XV: | Recovery Data for 1,3-Dichloropropane in Synthetic Samples | 33 |
| Table XVI: | Recovery Data for 1,2,2-Trichloropropane in Synthetic Samples | 34 |
| Table XVII: | Recovery Data for cis-1,3,3-Trichloropropene in Synthetic Samples | 35 |
| Table XVIII: | Recovery Data for trans-1,3,3-Trichloropropene in Synthetic Samples | 36 |
| Table XIX: | Summary of Recovery (Accuracy) Data for Impurities in Synthetic Samples | 37 |
| Table XX: | Sample Weights for Telone Precision Samples of TSN101516 and TSN101496 | 38 |
| Table XXI: | Precision Data for 2-Chloro-2-methylpentane in Telone Samples TSN101516 and TSN101496 | 39 |
| Table XXII: | Precision Data for 2-Chloro-4-methylpentane in Telone Samples TSN101516 and TSN101496 | 40 |
| Table XXIII: | Precision Data for 2-Chloro-2,3-dimethylbutane in Telone Samples TSN101516 and TSN101496 | 41 |

VI. Tables, continued

Page

Table XXIV: Precision Data for 3-Chloro-2-methylpentane in Telone Samples TSN101516 and TSN101496 ..... 42
Table XXV: Precision Data for 1,3-Dichloropropane in Telone Samples TSN101516 and TSN101496 ..... 43
Table XXVI: Precision Data for 1,2,2-Trichloropropane in Telone Samples TSN101516 and TSN101496 ..... 44
Table XXVII: Precision Data for cis-1,3,3-Trichloropropene in Telone Samples TSN101516 and TSN101496 ..... 45
Table XXVIII: Precision Data for trans-1,3,3-Trichloropropene in Telone Samples TSN101516 and TSN101496 ..... 46
Table XXIX: Summary of Precision Data for Impurities in Telone II Lots TSN101516 and TSN101496 ..... 47
Table XXX: System Precision Data for Instrument used in Method Validation ..... 48

VII. FIGURES

..... 49
Figure 1: Representative Chromatogram of Epoxidized Soybean Oil: Ethyl Acetate Solvent Blank ..... 49
Figure 2: Representative Chromatogram of Impurity Standards ..... 50
Figure 3: Representative Chromatogram of cis-1,3-Dichloropropene and trans-1,3-Dichloropropene Analytical Standard ..... 51
Figure 4: Representative Chromatogram of Impurities in Telone Sample (TSN101516) ..... 52
Figure 5: Representative Chromatogram of Impurities in Telone Sample (TSN101496) ..... 53
Figure 6: Representative Chromatogram of cis-1,3-Dichloropropene (TSN100275) ..... 54
Figure 7: Representative Chromatogram of trans-1,3-Dichloropropene (TSN100276) ..... 55
Figure 8: Representative Chromatogram of 2-Chloro-2-methylpentane (AGR238091) ..... 56
Figure 9: Representative Chromatogram of 2-Chloro-4-methylpentane (TSN101687) ..... 57
Figure 10: Representative Chromatogram of 2-Chloro-2,3-dimethylbutane (AGR238087) ..... 58
Figure 11: Representative Chromatogram of 3-Chloro-2-methylpentane (minor peak) and 2-Chloro-2-methylpentane (major peak) (TSN101695) ..... 59
Figure 12: Representative Chromatogram of 1,3-Dichloropropane (AGR238090) ..... 60
Figure 13: Representative Chromatogram of 1,2,2-Trichloropropane (AGR238089) ..... 61
Figure 14: Representative Chromatogram of cis-1,3,3-Trichloropropene (AGR238088) ..... 62

VII.Figures, continued

Page

Figure 15: Representative Chromatogram of trans-1,3,3-Trichloropropene (AGR238086) ..... 63
Figure 16: Linearity of 2-Chloro-2-methylpentane ..... 64
Figure 17: Linearity of 2-Chloro-4-methylpentane ..... 65
Figure 18: Linearity of 2-Chloro-2,3-dimethylbutane ..... 66
Figure 19: Linearity of 3-Chloro-2-methylpentane ..... 67
Figure 20: Linearity of 1,3-Dichloropropane ..... 68
Figure 21: Linearity of 1,2,2-Trichloropropane ..... 69
Figure 22: Linearity of cis-1,3,3-Trichloropropene ..... 70
Figure 23: Linearity of trans-1,3,3-Trichloropropene ..... 71
Figure 24: Representative Chromatogram using DB-1 Column for Separation of Telone Impurities ..... 72

VIII. APPENDIX: Analytical Method Summary: Telone II ..... 73

# I. ABSTRACT

An analytical method was validated for the determination of weight percent of process impurities in Telone II* by gas chromatography (CC) with a DB-1701 column and TCD detection. Quantitation of the impurities was achieved by comparing the detector response of the sample to that of a standard using external standard calibration. Detector response was found to be linear for each impurity over the range of at least 0.38 mg/mL to 11.8 mg/mL. Recovery samples were prepared covering a weight percent range of at least 0.050 to 4.65% wt. The average recoveries for the impurities in the synthetic samples ranged from 100.6 to 107.8%, with relative standard deviations ranging from 7.2 to 12.3%.

The precision of the method was determined by repetitive analysis of two lots of Telone II at least 10 times on different days. The precision for the impurities ranged from 1.1 to 17.8% RSD. The stability of Telone in ethyl acetate was examined by re-analyzing the prepared solutions 10 days after the initial preparation. Solutions had been stored refrigerated or at ambient temperature. All impurities were stable for ten days when stored in screw-capped jars at refrigerated or ambient temperatures.

*Trademark of Dow AgroSciences LLC

## II. INTRODUCTION

A. Scope

This method is applicable for the determination of process impurities potentially present in Telone* II. The CAS names with synonyms, CAS registration numbers, and structures of the active ingredients, cis-1,3-dichloropropene and trans-1,3-dichloropropene, and associated process impurities are presented in Table I. This study was performed under protocol NA-AM-98-081.

B. Principle

Solutions of Telone II are prepared in ethyl acetate and injected into a gas chromatograph equipped with a DB-1701 column and TCD detection. Quantitation of the impurities is achieved by comparing the detector response of the sample to that of a standard using external standard calibration. The active ingredients, cis-1,3-dichloropropene and trans-1,3-dichloropropene, and the process impurities, are assayed in the same sample. Statistical methods of means, standard deviations, relative standard deviations, and t-test were used in the study.

## III. MATERIALS AND METHODS

A. Safety

Each analyst should be acquainted with potential hazards of the reagents, products, and solvents before starting laboratory work. Sources of information include: material safety data sheets, literature and other related data. Safety information on non-Dow AgroSciences LLC products should be requested from the supplier. Disposal of reagents, reactants, and solvents must be in compliance with local, state, and federal laws and regulations.

*Trademark of Dow AgroSciences LLC

B. Equipment and Materials

1. Balance: analytical, capable of measuring 0.1 mg, Model AE200, Mettler Instrument Corporation, or equivalent.
2. Column: J&W Scientific DB-1701 60 m x 0.32 mm; 1.0 micron film thickness.
3. Gas Chromatograph: Hewlett Packard 6890, or equivalent, equipped with a split injector and TCD detector.
4. Data System: Hewlett Packard ChemServer, or equivalent.
5. Appropriate laboratory glassware, to include volumetric flasks and pipettes.
6. Gas-tight and Hamilton syringes capable of delivering 50 µL to 500 µL, or equivalent.

C. Reagents and Standards

All standards and lots of Telone II were obtained from Test Substance Coordinator, Dow AgroSciences LLC, Indianapolis, IN 46268.

1. (Z)-1,3-dichloropropene analytical standard, cis-1,3-D, Lot TSN100275, 98.9% purity.
2. (E)-1,3-dichloropropene analytical standard, trans-1,3-D, Lot TSN100276, 97.8% purity.
3. AGR238091: 2-chloro-2-methylpentane, 100% purity.
4. AGR238087: 2-chloro-2,3-dimethylbutane, 100% purity.
5. AGR238090: 1,3-dichloropropane, 99.5% purity.
6. AGR238089: 1,2,2-trichloropropane, 96% purity.
7. AGR238088: cis-1,3,3-trichloropropene, 96% purity.
8. AGR238086: trans-1,3,3-trichloropropene, 95% purity.
9. TSN101687: 2-chloro-4-methylpentane, 75% purity.
10. TSN101695: mixture of 3-chloro-2-methylpentane (23% purity) and 2-chloro-2-methylpentane (76% purity).
11. Telone II, Lots TSN101496 and TSN101516.
12. Epoxidized Soybean Oil (ESO), Witco Drapex 6.8.
13. Ethyl Acetate, EM Science, GC Grade or equivalent.

D. Gas Chromatographic Conditions

| | | | | |
|---|---|---|---|---|
| Instrument: | Hewlett-Packard 6890 or equivalent | | | |
| Column: | J&W DB-1701 60 m x 0.32 mm 1.0 micron film | | | |
| Inlet: | Split | | | |
| Split Ratio: | 38:1 | | | |
| Purge flow: | 1.2 mL/min | | | |
| Inlet Temperature: | 150°C | | | |
| Oven Temperature: | Initial 40°C | | | |
| Initial Time: | 2.0 min | | | |
| Oven Gradient: | Level | Rate °C/min | Final °C | Final Time (min) |
| | 1 | 5.0 | 80 | 7.5 |
| | 2 | 5.0 | 110 | 1.0 |
| | 3 | 25.0 | 270 | 0 |
| Detector: | TCD | | | |
| Detector Temperature: | 280°C | | | |
| Back Inlet: | Constant Flow | | | |
| Carrier Flow: | 72 mL/min | | | |
| Column Flow Rate: | 1.9 mL/min | | | |
| Injection Volume: | 2 µL | | | |
| Run Time: | approximately 30 min. | | | |
| Time to prepare and analyze one sample: | approximately 40 min | | | |

E. Preparation of Standard Solutions for Calibration, Recovery, and Linearity Studies

Note: For routine impurity analysis, a simplified optional procedure for preparation of the analytical and impurity standards is presented in Appendix I. The stock solutions listed below were prepared for fortification of samples to examine recovery and linearity; the concentrations are therefore higher than stock concentrations for routine assay purposes.

Preparation of Stocks for Determining Relative Retention Times and Response Factors:

A solution of each standard was prepared for determining relative retention time and relative response factor, as designated in Table II. Typically, 50 µL of the individual standard, recording the weight to the nearest 0.1 mg, was transferred to a tared vial and diluted to 1 mL final volume by adding 950 µL ethyl acetate with a gas-tight syringe.

Preparation of an Impurity Stock Solution:

An impurity stock solution containing approximately 150 mg/mL of each impurity, depending upon its density and purity (Table III) was prepared. Typically, 100 µL of each standard, recording the weight to the nearest 0.1 mg, was transferred to a tared vial. If needed, add ethyl acetate was added to obtain the desired final concentration.

Preparation of an Impurity Standard:

An impurity standard was prepared containing approximately 1.5 mg/mL of each component (Table III) by volumetrically transferring 100 µL of the impurity stock solution with a gas-tight syringe to a 10-mL volumetric flask and diluting to volume with ethyl acetate.

F. Preparation of Linearity Samples

Linearity samples were prepared by transferring 300 µL of the impurity stock (containing ~150 mg/mL of each impurity, depending upon its density and purity), to a tared vial and adding 1700 µL ethyl acetate with a gas-tight or Hamilton syringe, yielding a final volume of 2000 µL (Table IV). Subsequent dilutions were prepared by volumetrically transferring the appropriate solution with a gas-tight or Hamilton syringe, and adding the appropriate volume of ethyl acetate. Linearity samples for TSN101695, which contains two impurities, were prepared by making a stock solution in ethyl acetate (Table V) and subsequent dilutions.

G. Preparation of Recovery Samples

Recovery samples were prepared by preparing stock solutions of cis-1,3-D, trans-1,3-D and an impurity standard solution (Table VI). The recovery samples were then prepared by combining the appropriate volume of the impurity standard solution with cis-1,3-D or trans-1,3-D (Tables VII). Additional

recovery samples were prepared with new stocks to ensure complete coverage of the potential wt% assay range (Table VIII).

Recovery samples of TSN101695 and TSN101687 were prepared by diluting the standards with cis-1,3-D or trans-1,3-D stock solutions (Tables IX and X).

H. Preparation of Impurity Precision Samples

Precision measurements were performed utilizing two lots of Telone II, TSN101496 and TSN101516. The samples were analyzed at least 5 times on two different days to determine the precision of the method. Samples were prepared by volumetrically pipetting 4 mL Telone into a 10 mL volumetric flask, recording the weight to the nearest 0.1 mg, and diluting to volume with ethyl acetate.

I. Preparation of System Precision Sample

System precision was evaluated by injecting a sample of Telone six consecutive times. To prepare the sample, 4 mL of Telone II was pipetted into a 10 mL volumetric flask, recording the weight of the sample. The sample was diluted to the final volume of 10 mL with ethyl acetate.

J. Method Ruggedness

The ruggedness of the method was determined by injecting standards and technical samples using varying chromatographic conditions, including the use of a DB-1 column (conditions are listed on Figure 24) and a higher injector temperature (270°C) with the DB-1701 column.

K. Methods of Calculation

Calculations were performed by the ChemServer system using external standard calibration or by using manual calculations and the following equations:

Response Factor for Calibration Standard:

$$Rf = \frac{A \times P}{B}$$

Where: Rf = response factor for standard
A = weight (mg), or concentration (mg/mL) of standard
P = purity of standard, expressed as decimal
B = peak area of standard in calibration standard

Concentration of Component in Sample:

$$\% \text{ Component} = \frac{Rf \times D \times E}{F}$$

Where: Rf = response factor for component in standard, calculated above
D = peak area of component in sample
E = multiplication factor (used to adjust for difference in dilution of standard and sample and to express value as weight percent)
F = sample weight, expressed in mg, or concentration (mg/mL)

Determination of Linearity:

The general equation for calculating a least square linear fit for the standard curve is as follows:

$$PR = mC + b$$

where PR = detector response for component
m = slope of the line.
C = concentration of component, in mg/mL.
b = y-axis intercept

Where applicable, corrections were made in the areas of the recovery samples for interferences from minor components within the impurity standards. This was achieved by using the ratio of the interfering impurity to the ratio of the major impurity to calculate the corresponding area due to interference.

# IV. RESULTS AND DISCUSSION

## A. Interferences

The Telone technical sample matrix was evaluated for interference. No impurities were found to elute with cis-1,3-dichloropropene or trans-1,3-dichloropropene. Interferences were corrected for in recovery samples in which minor impurities in one component interfered with quantitation of another component at varying levels. Under normal standard preparation conditions, these corrections will be unnecessary. Representative chromatograms of the solvent blank, impurity standard, cis-1,3-D and trans-1,3-D calibration standard, and the two lots of Telone are presented in Figures 1 through 5, respectively. Chromatograms of the individual impurity standards are presented in Figures 6 through 15.

## B. Relative Response Factors

The relative detector response for each impurity is summarized in Table II. As shown by the similarity of the responses, the impurities can be assigned to groups. If certain standards are not available for some impurities, it is permissible to quantitate them using a standard from within the same group of response factors.

## C. Recovery (Accuracy)

A series of synthetic recovery samples were prepared by mixing the impurities, or a mixture of the impurities, with cis-1,3-D or trans-1,3-D stock solutions. The spiked synthetic samples were corrected for interference from minor impurities in the process impurities before determining recovery.

Analysis of a series of recovery samples yielded acceptable recovery data over the range of at least 0.050 wt% to 4.65 wt%. The average recovery across the weight percent range for each impurity ranged from 100.6 to 107.8% with an

overall average of 103.0% (Table XIX). The recovery data for each impurity are summarized in Tables XI through XVIII. A summary is presented in Table XIX.

D. Precision

Eleven samples of Telone II, lots TSN101516 and TSN101496, were prepared and assayed on two different days. The samples were prepared by volumetrically pipetting 4 mL of the Telone, recording the weight of the sample to the nearest 0.1 mg, and volumetrically diluting to the final volume of 10 mL. As shown in Table XX, the average sample weight for the 4-mL volume is 4.85 g ± 0.01 g. Based on the low variability of the sample weights, samples may be prepared by volumetrically pipetting the 4 mL of Telone sample and diluting to 10 mL, using a density of 1.212 g/mL to calculate the sample weight.

The precision data for each impurity in lots TSN101516 and TSN101496 are summarized in Tables XXI through XXVIII. A summary is presented in Table XXIX. Eleven samples of TSN101516 were prepared and assayed on two different days. The impurity precision ranged from 1.5% to 12.0% RSD with an average of 4.4% in the analysis of TSN101516. The impurity precision in TSN101496 ranged from 1.1% to 17.8% RSD, with an average of 6.2%.

Limits of detection and limits of quantitation were determined based on the precision data for Telone lot TSN101516. The limit of detection was calculated as three times the standard deviation while the limit of quantitation was ten times the standard deviation of the precision data, as summarized in Table XXIX.

The precision of the analytical system was evaluated by injecting a precision sample six consecutive times. The relative standard deviation of the peak area of cis-1,3-D and trans-1,3-D was 0.4%. A third peak, with an area of less than

10% of the primary components, had a relative standard deviation of 1.3%. System precision data is presented in Table XXX.

E. Linearity

Detector response was determined for a series of diluted samples, corresponding to a minimal weight percent of at least 0.0078%wt. The responses for each impurity were linear over the range of at least 0.38 mg/mL to 11.8 mg/mL. Individual linearity plots and the summary of the data for the linearity of each impurity are shown in Figures 16 through Figure 23. All impurities had a regression coefficient of at least 0.9994, with a minimum of nine observations.

F. Stability

Stability of the sample solutions stored in glass jars at ambient and refrigerated temperatures were evaluated by re-analyzing the precision samples from the first day's precision samples for at least one of the technical lots 10 days after initial preparation. The same calibration solutions, used in the initial determination, which had been stored in vials following preparation, were also included in the re-analysis. No differences in concentration was observed between the initial and stored solutions, indicating stability for up to 10 days.

G. Method Ruggedness

Initial chromatographic conditions were tested using a J&W Scientific DB-1 column (60 m x 0.25 mm x 1.0 μ film thickness) with the inlet temperature at 150°C. The early-eluting impurities were not as well resolved in these samples. An example chromatogram is shown in Figure 24.

It was also found that precision was improved using the lower injector temperature, as described in the method. It appeared that some impurities may have degraded at the higher injector temperature.

H. Method Limitation and Notes

1. Initial method development studies showed that injector temperature was important in determining the impurity quantitation. When the injector temperature was too high (270°C), additional impurities were seen, indicating decomposition of the sample in the injector.
2. Equipment, glassware, reagents, and chemicals considered to be equivalent to those specified may be substituted with the understanding that their performance must be confirmed by appropriate tests. Common laboratory supplies are not listed here.
3. The data presented in the report has been calculated in Excel electronic spreadsheets using more figures than shown in the report. Calculation of the values by hand may result in slight differences due to differences in rounding.
4. If volumetric pipettes are used, the weight percent technical may be calculating using the density of 1.2 g/mL to calculate the sample weight with minimal effect on the final weight percent Telone. The concentration of standards should be calculated by weight for improved accuracy.
5. Impurities may be quantitated using a representative impurity standard with a similar relative response factor, as summarized in Table II.

## V. CONCLUSIONS

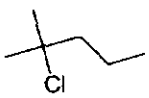
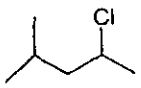
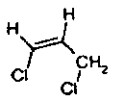
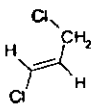
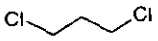
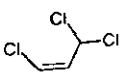
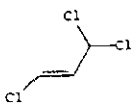
This method is applicable to the determination of eight impurities in Telone over the concentration range of at least 0.38 mg/mL to 11.8 mg/mL. The linearity, precision, and recovery of the analysis results in the validation study were acceptable. This report satisfies the requirement guideline OPPTS 830.1800. In accordance with good laboratory practices, it is recommended that precision and linearity of the method be determined if equipment other than that specified is used.

Raw data generated for this report and the final report will be stored in the Dow AgroSciences LLC Archives, Building 306, 9330 Zionsville Road, Indianapolis, IN.

# VI. TABLES

## Table I

### Summary of Telone and Impurity Nomenclature Cross-Reference

| Impurity Name | Impurity Name | CAS # | Reference Number | Structure |
|---|---|---|---|---|
| 2-Chloro-2-methylpentane | 2-Chloro-2-methylpentane | 4325-48-8 | AGR238091<br>TSN101695 |  |
| 2-Chloro-4-methylpentane | 2-Chloro-4-methylpentane | | TSN101687 |  |
| 2-Chloro-2,3-dimethylbutane | 2-Chloro-2,3-dimethylbutane | 594-57-0 | AGR238087 |  |
| (Z)-1,3-Dichloropropene | cis-1,3-D<br>cis-1,3-dichloropropene | 542-75-6 | TSN100275 |  |
| 3-Chloro-2-methylpentane | 3-Chloro-2-Methylpentane | | TSN101695 |  |
| (E)-1,3-Dichloropropene | trans-1,3-dichloropropene<br>Trans-1,3-D | 542-75-6 | TSN100276 |  |
| 1,3-Dichloropropane | 1,3-Dichloropropane | 142-28-9 | AGR238090 |  |
| 1,2,2-Trichloropropane | 1,2,2-Trichloropropane | 3175-23-3 | AGR238089 |  |
| (Z)-1,3,3-Trichloropropene | Cis-1,3,3-Trichloropropene | 2953-50-6 | AGR238088 |  |
| (E)-1,3,3-Trichloropropene | Trans-1,3,3-Trichloropropene | 2598-01-8 | AGR238086 |  |

## VI. TABLES

### TABLE II
Preparation of Individual Purity Solutions and Summary of Response Factors and Retention Times

| | | Purity (%) | Vol (µL) | Wt (mg) | Vol Et Ac (µL) | Total Vol. (mL) | Conc. (mg/mL) | Approx. Retention Time (min) | Peak Area | Response Factor | Response Factor relative to cis-1,3-D | Group |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TSN100275<br>cis-1,3-dichloropropene | P68I | 98.9 | 50 | 60.5 | 950 | 1.0 | 59.8345 | 14.9 | 265261 | 2.256E-04 | 1.00 | A |
| TSN100276<br>trans-1,3-dichloropropene | P68J | 97.8 | 50 | 60.5 | 950 | 1.0 | 59.169 | 17.6 | 271337 | 2.181E-04 | 0.97 | A |
| TSN101695 | | | | | | | | | | | | |
| 3-Cl-2-methylpentane | P68H | 23 | 200 | 172.8 | 800 | 1.0 | 39.744 | 16.2 | 169373 | 2.347E-04 | 1.04 | A |
| 2-Cl-2-methylpentane | P68H | 76 | 200 | 172.8 | 800 | 1.0 | 131.328 | 14.1 | 559457 | 2.347E-04 | 1.04 | A |
| TSN101687<br>2-Cl-4-methylpentane | P68A | 75 | 50 | 41.8 | 950 | 1.0 | 31.35 | 14.26 | 139280 | 2.251E-04 | 1.00 | A |
| AGR238091<br>2-Cl-2-methylpentane | P68G | 100 | 50 | 42.6 | 950 | 1.0 | 42.6 | 14.01 | 184708 | 2.306E-04 | 1.02 | A |
| AGR238090<br>1,3-Dichloropropane | P68E | 99.5 | 50 | 58.6 | 950 | 1.0 | 58.307 | 19.18 | 258857 | 2.252E-04 | 1.00 | A |
| AGR238087<br>2-Cl-2,3-dimethylbutane | P68F | 100 | 50 | 43.2 | 950 | 1.0 | 43.2 | 14.45 | 173673 | 2.487E-04 | 1.10 | B |
| AGR238086<br>trans-1,3,3-trichloropropene | P68B | 95 | 50 | 69.4 | 950 | 1.0 | 65.93 | 25.01 | 247406 | 2.665E-04 | 1.18 | C |
| AGR238088<br>cis-1,3,3-trichloropropene | P68C | 96 | 50 | 67.4 | 950 | 1.0 | 64.704 | 21.24 | 240372 | 2.692E-04 | 1.19 | C |
| AGR238089<br>1,2,2-Trichloropropane | P68D | 96 | 50 | 65.4 | 950 | 1.0 | 62.784 | 19.54 | 231444 | 2.713E-04 | 1.20 | C |

# VI. TABLES

## Table III

### Preparation of a Typical Impurity Standard

| | P66 A Imp[1] | | | | P66 B[2] |
|---|---|---|---|---|---|
| | µL | mg | Purity | mg/mL | mg/mL |
| **AGR238091** (2-Cl-2-methylpentane) | 100 | 85.7 | 100 | 107.13 | 1.071 |
| **AGR238087** (2-Cl-2,3-dimethylbutane) | 100 | 87.3 | 100 | 109.13 | 1.091 |
| **AGR238090** (1,3-dichloropropane) | 100 | 118.0 | 99.5 | 146.76 | 1.468 |
| **AGR238089** (1,2,2-trichloropropane) | 100 | 131.9 | 96 | 158.28 | 1.583 |
| **AGR238088** (cis-1,3,3-trichloropropene) | 200 | 269.2 | 96 | 323.04 | 3.230 |
| **AGR238086** (trans-1,3,3-trichloropropene) | 100 | 136.9 | 95 | 162.57 | 1.626 |
| **TSN101687** (2-Cl-4-methylpentane) | 100 | 84.1 | 75 | 78.84 | 0.788 |
| **TSN100275** (cis-1,3-D) | 0 | | 98.9 | | |
| **TSN100276** (trans-1,3-D) | 0 | | 97.8 | | |
| **Ethyl Acetate** | | | | | |
| Final vol. (µL) | 800 | | | | |

[1]Individual standards were volumetrically transferred with a gas-tight syringe to a tared, capped vial. Solution is further diluted prior to assay

[2]P66B is prepared by volumetrically transferring 100 µL of P66A Imp with a gas-tight syringe to a 10-mL volumetric flask and diluting to volume with ethyl acetate.

## VI. TABLES

### Table IV

Preparation of Linearity Samples and Concentrations of Each Impurity Present

| Sample # | Dilution | 2-Cl-2-methylpentane AGR238091 | 2-Cl-4-methylpentane TSN101687 | 2-Cl-2,3-Dimethylbutane AGR238087 | 1,3-Dichloro-propane AGR238090 | 1,2,2-Trichloro-propane AGR238089 | cis-1,3,3-trichoro-propene AGR238088 | trans-1,3,3-trichloro-propene AGR238086 |
|---|---|---|---|---|---|---|---|---|
| P67A | 300uL P66AIMP to 2 ml | 16.069 | 11.827 | 16.369 | 22.014 | 23.742 | 48.456 | 24.385 |
| P67B | 500 uL P67A to 1mL total | 8.034 | 5.913 | 8.184 | 11.007 | 11.871 | 24.228 | 12.193 |
| P67C | 500 uL P67B to 1 mL total | 4.017 | 2.957 | 4.092 | 5.504 | 5.936 | 12.114 | 6.096 |
| P67D | 500 uL P67C to 1 mL total | 2.009 | 1.478 | 2.046 | 2.752 | 2.968 | 6.057 | 3.048 |
| P67E | 500 uL p67D to 1 mL total | 1.004 | 0.739 | 1.023 | 1.376 | 1.484 | 3.029 | 1.524 |
| P67F | 500 uL P67E to 1 mL total | 0.502 | 0.370 | 0.512 | 0.688 | 0.742 | 1.514 | 0.762 |
| P67G | 500 uL P67F to 1 mL total | 0.251 | 0.185 | 0.256 | 0.344 | 0.371 | 0.757 | 0.381 |
| P67H | 500 uL P67G to 1 mL total | 0.126 | 0.092 | 0.128 | 0.172 | 0.185 | 0.379 | 0.191 |
| P67I | 200 uL P67A to 2 mL total | 1.607 | 1.183 | 1.637 | 2.201 | 2.374 | 4.846 | 2.439 |
| P67J | 500 uL P67I to 1 mL total | 0.803 | 0.591 | 0.818 | 1.101 | 1.187 | 2.423 | 1.219 |
| P67K | 500 uL P67J to 1 mL total | 0.402 | 0.296 | 0.409 | 0.550 | 0.594 | 1.211 | 0.610 |

# VI. TABLES

## Table V

Preparation of Linearity Samples and Concentrations for TSN101695
(2-Chloro-2-methylpentane and 3-Chloro-2-methylpentane)

(a) Preparation of P68H Stock Solution for TSN101695

| | µL | mg | Impurity | % Purity | Conc. (mg/mL) |
|---|---|---|---|---|---|
| TSN101695 | 200 | 172.8 | 2-Chloro-2-methylpentane | 76 | 131.328 |
| | | | 3-Chloro-2-methylpentane | 23 | 39.744 |
| Ethyl Acetate | 800 | | | | |
| Final Volume: | 1.0 mL | | | | |

(b) Preparation of Additional Dilutions

| Sample # | Dilution | 2-Cl-2-methylpentane mg/mL | 3-Chloro-2-methylpentane mg/mL |
|---|---|---|---|
| P68H | see above | 131.328 | 39.744 |
| P68K | 200 µL P68H + 1800 µL Et. Ac. | 13.133 | 3.974 |
| P68L | 100 µL P68K + 100 µL Et. Ac. | 6.566 | 1.987 |
| P68M | 100 µL P68K + 300 µL Et. Ac. | 3.283 | 0.994 |
| P68N | 100 µL P68K + 450 µL Et. Ac. | 2.388 | 0.723 |
| P68O | 80 µL P68K + 520 µL. Et. Ac. | 1.751 | 0.530 |
| P68P | 100 µL P68O + 100 µL Et. Ac. | 0.876 | 0.265 |
| P68Q | 20 µL P68M + 180 µL Et. Ac. | 0.328 | 0.0994 |
| P68R | 40 µL P68Q + 40 µL Et. Ac. | 0.164 | 0.0497 |

# VI. TABLES

## Table VI

### Stock Solutions used in Preparation of Recovery Samples

| | Volume TSN100275 | Weight TSN100275 | Volume TSN100276 | Weight TSN100276 | ESO:EtAc[1] | Final volume[2] |
|---|---|---|---|---|---|---|
| P74A | 5 mL | 6.1168 g | -- | -- | 1 mL | 10 mL |
| P74B | -- | -- | 5 mL | 6.1394 g | 1 mL | 10 mL |

[1]ESO:EtAc was prepared by making a 20% wt/vol solution of Drapex 6.8 Epoxidized Soybean oil (ESO). 10.479 g ESO was weighed into a 50-mL volumetric flask and diluted to volume with ethyl acetate.
[2]Samples were adjusted to final volume with ethyl acetate (EtAc).

| P74 F | Standard | Volume | Weight |
|---|---|---|---|
| | TSN100275 | 2 mL | 2.4399 g |
| | AGR238091 | 200 µL | 169.0 mg |
| | AGR238087 | 200 µL | 173.6 mg |
| | AGR238090 | 200 µL | 235.9 mg |
| | AGR238089 | 200 µL | 260.5 mg |
| | AGR238088 | 400 µL | 564.2 mg |
| | AGR238086 | 200 µL | 276.0 mg |
| | ESO:EtAc | 1 mL | |
| | Et Ac | to 10 mL final volume | |

# VI. TABLES

## Table VII

### Preparation of Recovery Samples

| Sample # | Stock 1 | Vol 1 (µL) | Wt 1 (mg) | Stock 2 | Vol 2 (µL) | Wt 2 (mg) | Final Vol (µL) |
|---|---|---|---|---|---|---|---|
| 74C | 74A | 100 | 105.8 | 74B | 100 | 105.9 | 200 |
| 74D | 74A | 50 | 52.2 | 74B | 100 | 106.5 | 150 |
| 74E | 74A | 100 | 105.8 | 74B | 50 | 53.2 | 150 |
| 74G | 74F | 180 | 179.4 | 74A | 20 | 20.4 | 200 |
| 74H | 74F | 150 | 148.8 | 74A | 50 | 52.6 | 200 |
| 74I | 74F | 120 | 119.2 | 74A | 80 | 84.2 | 200 |
| 74J | 74F | 100 | 99.3 | 74A | 100 | 106.4 | 200 |
| 74M | 74F | 80 | 78.8 | 74A | 120 | 127.8 | 200 |
| 74N | 74F | 50 | 49.4 | 74A | 150 | 159.3 | 200 |
| 74O | 74F | 20 | 19.6 | 74A | 180 | 189.9 | 200 |
| 74P | 74F | 100 | 100.1 | 74A | 900 | 947.9 | 200 |
| 74Q | 74B | 50 | 52.6 | 74A | 800 | 833.2 | 850 |
| 74R | 74B | 800 | 838.3 | 74A | 50 | 52.7 | 850 |
| 74S | 74B | 30 | 30.7 | 74A | 1800 | 1870.0 | 1830 |
| 74T | 74B | 1800 | 1890.1 | 74A | 30 | 31.0 | 1830 |

# VI. TABLES

## Table VIII

### Stock Solutions and Preparation of Additional Recovery Samples

| | Volume TSN100275 | Weight TSN100275 | Volume TSN100276 | Weight TSN100276 | ESO:EtAc[1] | Final volume[2] |
|---|---|---|---|---|---|---|
| P79B | 5 mL | 5.9586 g | | | 1 mL | 10 mL |

[1]ESO:EtAc was prepared by making a 20% wt/vol solution of Drapex 6.8 Epoxidized Soybean oil(ESO). 10.479 g was weighed into a 50-mL volumetric flask and diluted to volume with ethyl acetate.
[2]Samples are adjusted to final volume with ethyl acetate (EtAc).

| P78B | Standard | Volume | Weight |
|---|---|---|---|
| | TSN100275 | 2 mL | 2.4396 g |
| | AGR238091 | 200 µL | 169.0 mg |
| | AGR238087 | 200 µL | 168.7 mg |
| | AGR238090 | 200 µL | 228.5 mg |
| | AGR238089 | 200 µL | 257.4 mg |
| | AGR238088 | 200 µL | 267.2 mg |
| | AGR238086 | 200 µL | 268.1 mg |
| | ESO:EtAc | 1 mL | |
| | Et Ac | to 10 mL final volume | |

### Preparation of Additional Recovery Samples

| Sample # | Stock 1 | Vol 1 (µL) | Stock 2 | Vol 2 (µL) | Final Vol (µL) |
|---|---|---|---|---|---|
| P79C | P78B | 100 | P79B | 900 | 1000 |
| P79D | P78B | 50 | P79B | 950 | 1000 |
| P79E | P78B | 30 | P79B | 1000 | 1030 |
| P79F | P78B | 20 | P79B | 1000 | 1020 |
| P79G | P78B | 10 | P79B | 1000 | 1010 |
| P79H | P79E | 100 | P79B | 100 | 200 |
| P79I | P79F | 100 | P79B | 100 | 200 |
| P79J | P78B | 200 | P79B | 800 | 1000 |

## VI. TABLES

### Table IX

Preparation of Recovery Samples of TSN101695 (2-Chloro-2-methylpentane and 3-Chloro-2-methylpentane)

| Sample # | Stock 1 | Vol 1 (µL) | Wt 1 (mg) | Stock 2 | Vol 2 (µL) | Wt 2 (mg) | Final Vol (µL) |
|---|---|---|---|---|---|---|---|
| P75A | TSN101695 | 90 | 77.8 | 74A | 300 | 313.1 | 390 |
| P75B | P75A | 20 | 19.7 | 74A | 100 | 102.6 | 120 |
| P75C | P75A | 15 | 15.0 | 74A | 200 | 212.2 | 215 |
| P75D | P75A | 20 | 19.8 | 74A | 900 | 944.1 | 920 |
| P75E | P75D | 100 | 105.7 | 74A | 900 | 941.2 | 1000 |
| P75F | P75D | 50 | 52.7 | 74A | 150 | 158.8 | 200 |
| P75G | P75D | 50 | 52.9 | 74A | 900 | 937.7 | 950 |

# VI. TABLES

## Table X

### Preparation of Recovery Samples of TSN101687

| Sample # | Stock 1 | Vol 1 (µL) | Wt 1 (mg) | Stock 2 | Vol 2 (µL) | Wt 2 (mg) | Final Vol (µL) |
|---|---|---|---|---|---|---|---|
| P77A | TSN101687 | 60 | 50.4 | 74B | 600 | 627.4 | 660 |
| P77B | P77A | 20 | 20.5 | 74B | 100 | 107.9 | 120 |
| P77C | P77A | 15 | 15.0 | 74B | 200 | 202.4 | 215 |
| P77D | P77A | 20 | 20.3 | 74B | 900 | 946.7 | 920 |
| P77E | P77D | 100 | 106.9 | 74B | 900 | 939.9 | 1000 |
| P77F | P77D | 50 | 52.5 | 74B | 150 | 161.3 | 200 |
| P77G | P77D | 20 | 21.1 | 74B | 900 | 942.9 | 920 |

# VI. TABLES

## Table XI

Recovery Data for 2-Chloro-2-methylpentane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.050 | 0.30 | 0.34 | 112.2 |
| 0.55 | 3.30 | 3.49 | 106.0 |
| 1.84 | 10.58 | 10.00 | 94.6 |
| 4.80 | 25.27 | 25.79 | 102.1 |
| 22.63 | 151.61 | 144.88 | 95.6 |
| | | Average, % | 102.1 |
| | Standard Deviation, %: | | 7.4 |
| | | RSD,% | 7.2 |
| | | n | 5 |

# VI. TABLES

## Table XII

Recovery Data for 2-Chloro-4-methylpentane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.020 | 0.13 | 0.16 | 125.8 |
| 0.051 | 0.31 | 0.33 | 105.0 |
| 0.203 | 1.25 | 1.20 | 96.5 |
| 0.649 | 4.00 | 3.85 | 96.4 |
| 1.546 | 9.54 | 8.97 | 94.0 |
| 9.026 | 57.27 | 53.41 | 93.2 |
| | | Average, %: | 101.8 |
| | | Standard Deviation, %: | 12.5 |
| | | RSD, %: | 12.3 |
| | | n | 6 |

## VI. TABLES

### Table XIII

Recovery Data for 2-Chloro-2,3-dimethylbutane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.028 | 0.17 | 0.13 | 78.4 |
| 0.028 | 0.17 | 0.19 | 111.0 |
| 0.041 | 0.25 | 0.27 | 110.5 |
| 0.056 | 0.33 | 0.34 | 102.3 |
| 0.083 | 0.49 | 0.57 | 115.8 |
| 0.11 | 3.37 | 3.25 | 96.3 |
| 0.14 | 0.84 | 0.87 | 103.6 |
| 0.29 | 1.74 | 1.98 | 114.3 |
| 0.29 | 1.74 | 1.90 | 109.4 |
| 0.29 | 1.69 | 1.65 | 97.7 |
| 0.77 | 4.34 | 4.54 | 104.6 |
| 1.70 | 8.68 | 9.04 | 104.1 |
| 2.12 | 10.42 | 10.79 | 103.6 |
| 2.82 | 13.02 | 13.65 | 104.9 |
| 3.62 | 13.62 | 16.10 | 118.2 |
| 4.22 | 17.36 | 18.29 | 105.3 |
| 4.65 | 16.87 | 15.94 | 94.5 |
| | | Average, %: | 104.4 |
| | | Standard Deviation, %: | 9.4 |
| | | RSD, %: | 9.0 |
| | | n | 17 |

# VI. TABLES

## Table XIV

Recovery Data for 3-Chloro-2-methylpentane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.015 | 0.09 | 0.11 | 119.1 |
| 0.17 | 1.00 | 1.02 | 102.4 |
| 0.56 | 3.20 | 3.00 | 93.6 |
| 1.41 | 7.65 | 7.38 | 96.5 |
| 6.85 | 45.88 | 43.59 | 95.0 |
| | | Average, % | 101.3 |
| | | Standard Deviation, %: | 10.5 |
| | | RSD,% | 10.4 |
| | | n | 5 |

# VI. TABLES

## Table XV

Recovery Data for 1,3-Dichloropropane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.04 | 0.22 | 0.24 | 109.8 |
| 0.04 | 0.23 | 0.27 | 120.5 |
| 0.06 | 0.33 | 0.38 | 114.7 |
| 0.08 | 0.45 | 0.44 | 98.2 |
| 0.11 | 0.66 | 0.67 | 101.4 |
| 0.15 | 4.5 | 4.2 | 93.2 |
| 0.19 | 1.1 | 1.1 | 93.7 |
| 0.40 | 2.3 | 2.3 | 99.7 |
| 0.40 | 2.3 | 2.4 | 101.5 |
| 0.40 | 2.3 | 2.1 | 91.0 |
| 1.05 | 5.9 | 5.8 | 98.4 |
| 2.29 | 11.7 | 11.5 | 98.3 |
| 2.86 | 14.1 | 13.9 | 98.7 |
| 3.81 | 17.6 | 17.5 | 99.3 |
| 4.89 | 21.1 | 21.2 | 100.5 |
| 5.70 | 23.5 | 23.3 | 99.3 |
| 6.26 | 22.7 | 21.1 | 92.8 |
| | | Average, %: | 100.6 |
| | | Standard Deviation, %: | 7.8 |
| | | RSD, %: | 7.7 |
| | | n | 17 |

## VI. TABLES

### Table XVI

Recovery Data for 1,2,2-Trichloropropane in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.041 | 0.24 | 0.27 | 111.7 |
| 0.041 | 0.24 | 0.29 | 118.9 |
| 0.061 | 0.36 | 0.43 | 118.8 |
| 0.082 | 0.48 | 0.48 | 99.3 |
| 0.122 | 0.72 | 0.74 | 102.6 |
| 0.164 | 4.94 | 4.81 | 97.4 |
| 0.211 | 1.24 | 1.18 | 95.2 |
| 0.432 | 2.47 | 2.29 | 92.8 |
| 2.443 | 12.50 | 12.11 | 96.9 |
| 3.051 | 15.01 | 14.50 | 96.6 |
| 4.061 | 18.76 | 18.10 | 96.5 |
| 5.211 | 22.51 | 22.35 | 99.3 |
| 6.071 | 25.01 | 24.49 | 97.9 |
| 6.808 | 24.71 | 24.56 | 99.4 |
| | | Average, %: | 101.7 |
| | | Standard Deviation, %: | 8.5 |
| | | RSD, %: | 8.4 |
| | | n | 14 |

## VI. TABLES

### Table XVII

Recovery Data for cis-1,3,3-Trichloropropene in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.042 | 0.25 | 0.28 | 111.8 |
| 0.043 | 0.25 | 0.32 | 126.5 |
| 0.063 | 0.37 | 0.45 | 121.0 |
| 0.085 | 0.50 | 0.56 | 110.9 |
| 0.13 | 0.75 | 0.83 | 111.2 |
| 0.17 | 5.1 | 5.2 | 101.8 |
| 0.22 | 1.3 | 1.3 | 101.8 |
| 0.45 | 2.6 | 2.5 | 98.4 |
| 0.92 | 5.4 | 5.3 | 97.8 |
| 0.92 | 5.4 | 5.5 | 100.9 |
| 2.41 | 13.5 | 13.2 | 97.7 |
| 5.29 | 27.1 | 26.3 | 97.0 |
| 6.61 | 32.5 | 31.4 | 96.5 |
| 7.07 | 25.7 | 26.3 | 102.7 |
| 8.80 | 40.6 | 39.6 | 97.4 |
| 11.29 | 48.7 | 48.6 | 99.6 |
| 13.15 | 54.2 | 53.2 | 98.2 |
| | | Average, %: | 104.2 |
| | | Standard Deviation, %: | 9.0 |
| | | RSD, %: | 8.6 |
| | | n | 17 |

## VI. TABLES

### Table XVIII

Recovery Data for trans-1,3,3-Trichloropropene in Synthetic Samples

| wt% Assayed | Theoretical mg/mL in Sample | Recovered mg/mL | % Recovered |
|---|---|---|---|
| 0.042 | 0.25 | 0.28 | 113.5 |
| 0.042 | 0.25 | 0.34 | 136.6 |
| 0.063 | 0.37 | 0.50 | 134.8 |
| 0.084 | 0.50 | 0.56 | 112.5 |
| 0.13 | 0.74 | 0.85 | 114.5 |
| 0.17 | 5.09 | 5.55 | 109.0 |
| 0.22 | 1.27 | 1.35 | 105.9 |
| 0.44 | 2.62 | 2.53 | 96.5 |
| 0.44 | 2.62 | 2.59 | 98.9 |
| 0.44 | 2.55 | 2.63 | 103.3 |
| 1.17 | 6.56 | 6.82 | 104.0 |
| 2.56 | 13.11 | 12.74 | 97.2 |
| 3.20 | 15.73 | 15.65 | 99.5 |
| 4.26 | 19.67 | 19.55 | 99.4 |
| 5.46 | 23.60 | 23.45 | 99.4 |
| 6.36 | 26.22 | 25.74 | 98.2 |
| 7.02 | 25.47 | 28.06 | 110.2 |
| | | Average, %: | 107.8 |
| | | Standard Deviation, %: | 12.1 |
| | | RSD, %: | 11.2 |
| | | n | 17 |

# VI. TABLES

## Table XIX

### Summary of Recovery (Accuracy) Data for Impurities in Synthetic Samples

| Impurity | Range of Wt % Assayed | n | Average Recovery (%) | Standard Deviation (%) | Relative Standard Deviation (%) |
|---|---|---|---|---|---|
| 2-Chloro-2-methylpentane | 0.050 – 22.63 | 5 | 102.1 | 7.4 | 7.2 |
| 2-Chloro-4-methylpentane | 0.020 – 9.03 | 6 | 101.8 | 12.5 | 12.3 |
| 2-Chloro-2,3-dimethylbutane | 0.028 – 4.65 | 17 | 104.4 | 9.4 | 9.0 |
| 3-Chloro-2-methylpentane | 0.015 – 6.85 | 5 | 101.3 | 10.5 | 10.4 |
| 1,3-Dichloropropane | 0.04 – 6.26 | 17 | 100.6 | 7.8 | 7.7 |
| 1,2,2-Trichloropropane | 0.041 – 6.81 | 14 | 101.7 | 8.5 | 8.4 |
| cis-1,3,3-Trichloropropene | 0.042 – 13.15 | 17 | 104.2 | 9.0 | 8.6 |
| trans-1,3,3-Trichloropropene | 0.042 – 7.02 | 17 | 107.8 | 12.1 | 11.2 |
| Overall Average | | | 103.0 | | |
| Minimum | | | 100.6 | | |
| Maximum | | | 107.8 | | |
| Range Included in all Standards | 0.050 – 4.65 | | | | |

# VI. TABLES

## Table XX

Sample Weights for Telone Precision Samples of TSN101516 and TSN101496

| | Run # | Volume mL | TSN101516 g | TSN101496 g |
|---|---|---|---|---|
| Day 1 | 1 | 4 | 4.8552 | 4.8567 |
| | 2 | 4 | 4.8532 | 4.8673 |
| | 3 | 4 | 4.8498 | 4.8391 |
| | 4 | 4 | 4.8468 | 4.8634 |
| | 5 | 4 | 4.8613 | 4.8458 |
| Day 2 | 1 | 4 | 4.8712 | 4.8625 |
| | 2 | 4 | 4.8595 | 4.8394 |
| | 3 | 4 | 4.8711 | 4.8539 |
| | 4 | 4 | 4.8425 | 4.8326 |
| | 5 | 4 | 4.8578 | 4.8525 |
| | 6 | 4 | 4.8396 | 4.8465 |
| Average, % | | | 4.855 | 4.851 |
| Standard Deviation, % | | | 0.010 | 0.011 |
| Relative Standard Deviation, % | | | 0.2 | 0.2 |

# VI. TABLES

## Table XXI

Precision Data for 2-Chloro-2-methylpentane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.37 | 0.12 |
| | 2 | 0.37 | 0.12 |
| | 3 | 0.37 | 0.13 |
| | 4 | 0.36 | 0.12 |
| | 5 | 0.38 | 0.12 |
| Day 2 | 1 | 0.37 | 0.13 |
| | 2 | 0.37 | 0.13 |
| | 3 | 0.37 | 0.12 |
| | 4 | 0.38 | 0.12 |
| | 5 | 0.38 | 0.12 |
| | 6 | 0.38 | 0.12 |
| Average, % | | 0.37 | 0.12 |
| Standard Deviation, % | | 0.005 | 0.005 |
| Relative Standard Deviation, % | | 1.5 | 4.3 |

# VI. TABLES

## Table XXII

Precision Data for 2-Chloro-4-methylpentane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.15 | 0.069 |
| | 2 | 0.15 | 0.065 |
| | 3 | 0.15 | 0.079 |
| | 4 | 0.14 | 0.070 |
| | 5 | 0.15 | 0.074 |
| Day 2 | 1 | 0.14 | 0.081 |
| | 2 | 0.15 | 0.079 |
| | 3 | 0.15 | 0.069 |
| | 4 | 0.15 | 0.071 |
| | 5 | 0.15 | 0.073 |
| | 6 | 0.15 | 0.066 |
| Average, % | | 0.15 | 0.072 |
| Standard Deviation, % | | 0.003 | 0.005 |
| Relative Standard Deviation, % | | 2.0 | 7.5 |

# VI. TABLES

## Table XXIII

Precision Data for 2-Chloro-2,3-dimethylbutane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.13 | 0.049 |
| | 2 | 0.13 | 0.042 |
| | 3 | 0.13 | 0.068 |
| | 4 | 0.12 | 0.049 |
| | 5 | 0.14 | 0.056 |
| Day 2 | 1 | 0.12 | 0.068 |
| | 2 | 0.13 | 0.064 |
| | 3 | 0.13 | 0.050 |
| | 4 | 0.13 | 0.051 |
| | 5 | 0.13 | 0.054 |
| | 6 | 0.13 | 0.040 |
| Average, % | | 0.13 | 0.054 |
| Standard Deviation, % | | 0.004 | 0.010 |
| Relative Standard Deviation, % | | 3.4 | 17.8 |

## VI. TABLES

### Table XXIV

Precision Data for 3-Chloro-2-methylpentane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.091 | 0.032 |
| | 2 | 0.089 | 0.036 |
| | 3 | 0.089 | 0.033 |
| | 4 | 0.084 | 0.033 |
| | 5 | 0.092 | 0.036 |
| Day 2 | 1 | 0.085 | 0.036 |
| | 2 | 0.088 | 0.037 |
| | 3 | 0.090 | 0.033 |
| | 4 | 0.090 | 0.034 |
| | 5 | 0.091 | 0.035 |
| | 6 | 0.092 | 0.038 |
| Average, % | | 0.089 | 0.035 |
| Standard Deviation, % | | 0.003 | 0.002 |
| Relative Standard Deviation, % | | 3.0 | 6.0 |

## VI. TABLES

### Table XXV

Precision Data for 1,3-Dichloropropane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.047 | 0.086 |
| | 2 | 0.048 | 0.080 |
| | 3 | 0.045 | 0.082 |
| | 4 | 0.046 | 0.085 |
| | 5 | 0.044 | 0.080 |
| Day 2 | 1 | 0.047 | 0.083 |
| | 2 | 0.046 | 0.082 |
| | 3 | 0.049 | 0.086 |
| | 4 | 0.048 | 0.085 |
| | 5 | 0.046 | 0.092 |
| | 6 | 0.042 | 0.085 |
| Average, % | | 0.046 | 0.084 |
| Standard Deviation, % | | 0.002 | 0.003 |
| Relative Standard Deviation, % | | 4.8 | 3.9 |

# VI. TABLES

## Table XXVI

Precision Data for 1,2,2-Trichloropropane in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.032 | 0.15 |
| | 2 | 0.031 | 0.15 |
| | 3 | 0.031 | 0.15 |
| | 4 | 0.026 | 0.15 |
| | 5 | 0.025 | 0.15 |
| Day 2 | 1 | 0.028 | 0.15 |
| | 2 | 0.030 | 0.14 |
| | 3 | 0.034 | 0.15 |
| | 4 | 0.036 | 0.15 |
| | 5 | 0.037 | 0.15 |
| | 6 | 0.031 | 0.15 |
| Average, % | | 0.031 | 0.15 |
| Standard Deviation, % | | 0.004 | 0.003 |
| Relative Standard Deviation, % | | 12.0 | 2.0 |

## VI. TABLES

### Table XXVII

Precision Data for cis-1,3,3-Trichloropropene in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.039 | 0.26 |
| | 2 | 0.038 | 0.27 |
| | 3 | 0.038 | 0.26 |
| | 4 | 0.038 | 0.26 |
| | 5 | 0.038 | 0.26 |
| Day 2 | 1 | 0.037 | 0.26 |
| | 2 | 0.039 | 0.26 |
| | 3 | 0.039 | 0.26 |
| | 4 | 0.042 | 0.26 |
| | 5 | 0.042 | 0.26 |
| | 6 | 0.041 | 0.26 |
| Average, % | | 0.039 | 0.26 |
| Standard Deviation, % | | 0.002 | 0.003 |
| Relative Standard Deviation, % | | 3.9 | 1.1 |

## VI. TABLES

### Table XXVIII

Precision Data for trans-1,3,3-Trichloropropene in Telone Samples TSN101516 and TSN101496

| | Run # | TSN101516 Weight % | TSN101496 Weight % |
|---|---|---|---|
| Day 1 | 1 | 0.0* | 0.049 |
| | 2 | 0.0* | 0.043 |
| | 3 | 0.0* | 0.046 |
| | 4 | 0.0* | 0.049 |
| | 5 | 0.0* | 0.055 |
| Day 2 | 1 | 0.0* | 0.051 |
| | 2 | 0.0* | 0.047 |
| | 3 | 0.0* | 0.046 |
| | 4 | 0.0* | 0.048 |
| | 5 | 0.006 | 0.049 |
| | 6 | 0.013 | 0.051 |
| Average, % | | 0.002 | 0.049 |
| Standard Deviation, % | | 0.004 | 0.003 |
| Relative Standard Deviation, % | | N/A | 6.8 |

*Not detected in these samples

## VI. TABLES

### Table XXIX

Summary of Precision Data for Impurities in Telone Samples TSN101516 and TSN101496

| | | TSN101516 | | | TSN101496 | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Impurity | Standard | Average Wt % | Standard Deviation (%) | Relative Standard Deviation (%) | Average Wt % | Standard Deviation (%) | Relative Standard Deviation (%) | LOD[2] | LOQ[3] |
| 2-Chloro-2-methylpentane | AGR238091 TSN101695 | 0.37 | 0.005 | 1.5 | 0.12 | 0.005 | 4.3 | 0.015 | 0.05 |
| 2-Chloro-4-methylpentane | TSN101687 | 0.15 | 0.003 | 2.0 | 0.072 | 0.005 | 7.5 | 0.009 | 0.03 |
| 2-Chloro-2,3-dimethylbutane | AGR238087 | 0.13 | 0.004 | 3.4 | 0.054 | 0.01 | 17.8 | 0.012 | 0.04 |
| 3-Chloro-2-methylpentane | TSN101695 | 0.089 | 0.003 | 3.0 | 0.035 | 0.002 | 6.0 | 0.009 | 0.03 |
| 1,3-Dichloropropane | AGR238090 | 0.046 | 0.002 | 4.8 | 0.084 | 0.003 | 3.9 | 0.006 | 0.02 |
| 1,2,2-Trichloropropane | AGR238089 | 0.031 | 0.004 | 12.0 | 0.15 | 0.003 | 2.0 | 0.012 | 0.04 |
| cis-1,3,3-Trichloropropene | AGR238088 | 0.039 | 0.002 | 3.9 | 0.26 | 0.003 | 1.1 | 0.006 | 0.02 |
| trans-1,3,3-Trichloropropene | AGR238086 | 0.002 | 0.004 | N/A[1] | 0.049 | 0.003 | 6.75 | 0.012 | 0.04 |

[1]RSD is not calculated; impurity was detected in only two of the 11 samples analyzed.
[2]LOD is Limit of Detection for this instrument in this study, based on 3 times the standard deviation for batch TSN101516.
[3]LOQ is the Limit of Quantitation for this instrument in this study, based on 10 times the standard deviation for batch TSN101516.

# VI. TABLES

## Table XXX

### System Precision Data for Instrument used in Method Validation

| Injection # | Detector Response Area | Detector Response Area | Detector Response Area |
|---|---|---|---|
| 1 | 8068 | 1151753 | 989093 |
| 2 | 8212 | 1161089 | 995976 |
| 3 | 8243 | 1164977 | 999086 |
| 4 | 8346 | 1163344 | 997145 |
| 5 | 8104 | 1163751 | 998493 |
| 6 | 8128 | 1162344 | 996896 |
| Average: | 8183.5 | 1161209.7 | 996114.8 |
| Std. Dev.: | 103.4 | 4815.3 | 3618.9 |
| RSD, %: | 1.3 | 0.4 | 0.4 |

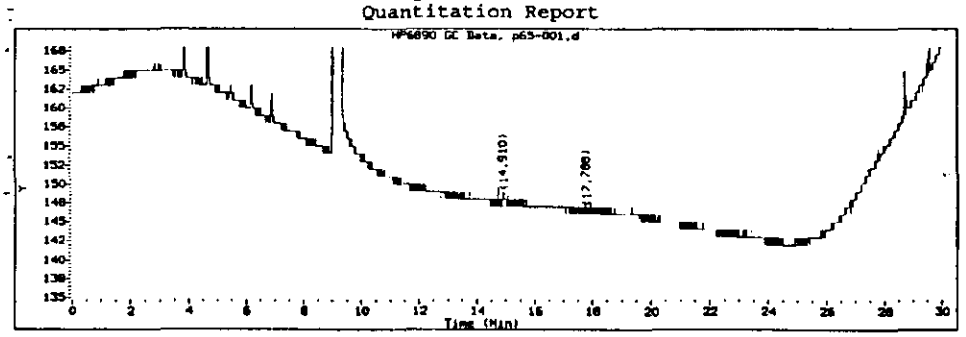
# VII. FIGURES

## Figure 1

## Representative Chromatogram of Epoxidized Soybean Oil: Ethyl Acetate Solvent Blank

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-001.d Page 1
Report Date 02/26/1999 08:21

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

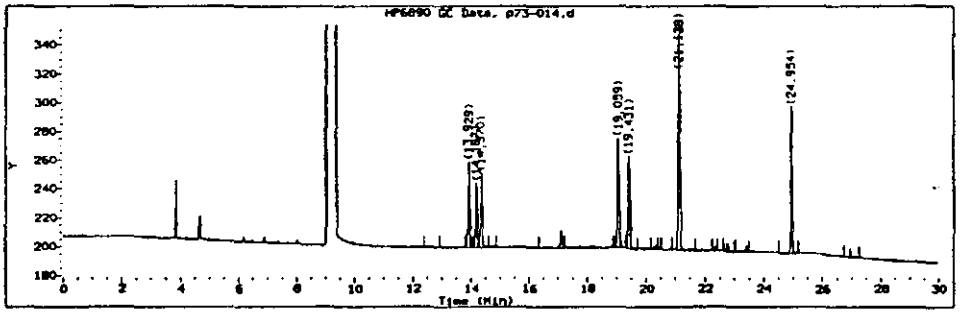
# VII. FIGURES

## Figure 2

## Representative Chromatogram of Impurity Standards

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p73-014.d Page 1
Report Date 02/22/1999 11:56

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

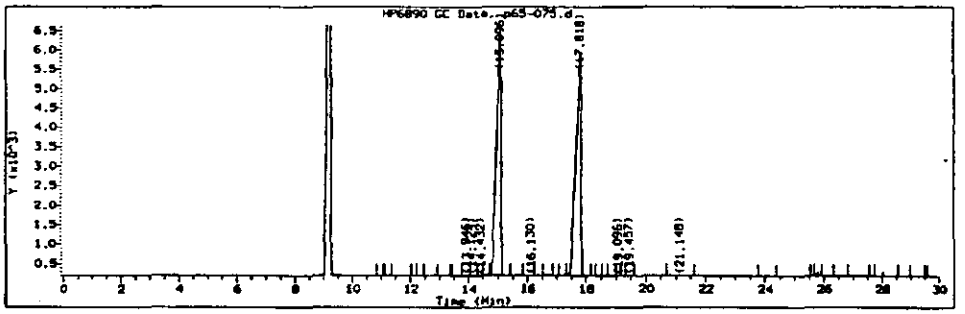
# VII. FIGURES

## Figure 3

Representative Chromatogram of cis-1,3-Dichloropropene and trans-1,3-Dichloropropene Analytical Standard

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-075.d Page 1
Report Date 02/22/1999 11:48

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

# VII. FIGURES

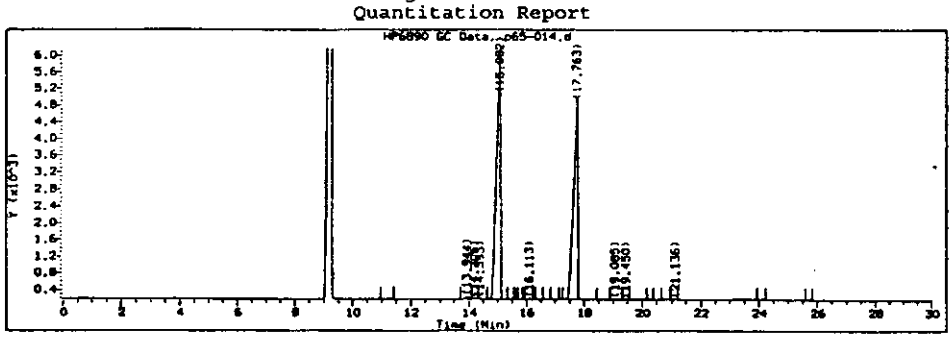
## Figure 4

Representative Chromatogram of Impurities in Telone Sample (TSN101516)

a) Chromatogram showing cis-1,3-D and trans-1,3-D

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-014.d Page 1
Report Date 02/26/1999 08:24

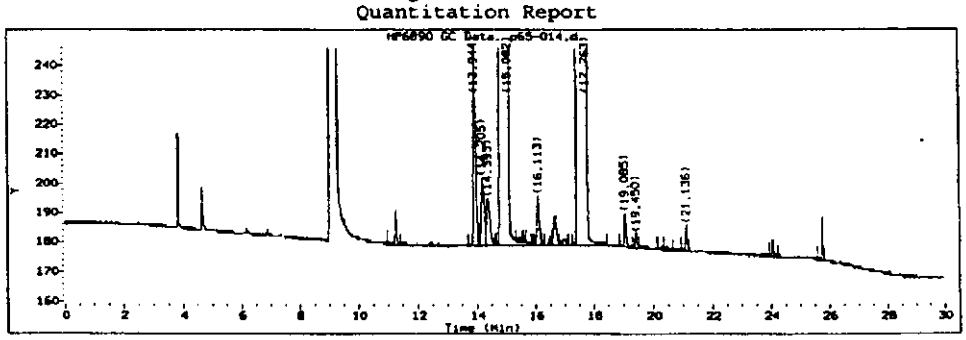
Dow AgroSciences LLC
Quantitation Report




b) Expanded scale to show impurities

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-014.d Page 1
Report Date 08/11/1999 16:12

Dow AgroSciences LLC
Quantitation Report

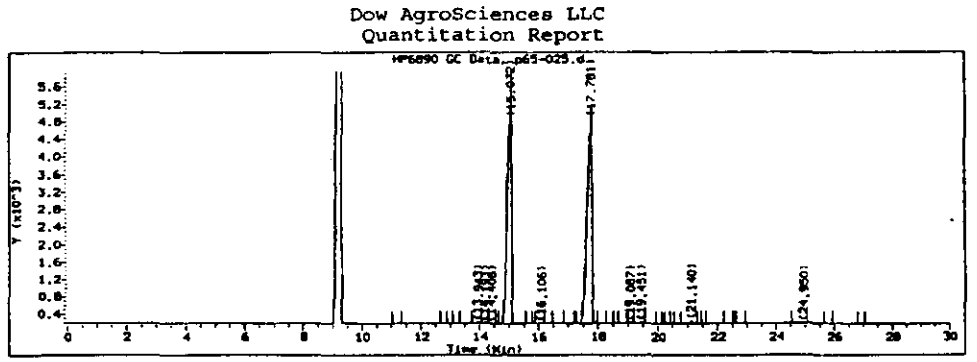



Chromatographic conditions are listed in Section III.D.
Components and approximate retention times are listed in Figure 3.

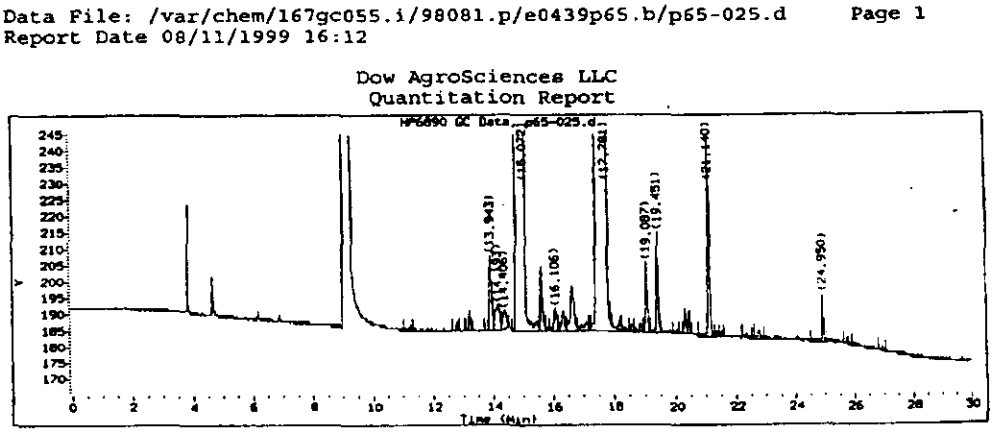
# VII. FIGURES

Figure 5
Representative Chromatogram of Impurities in Telone Sample (TSN101496)

a) Chromatogram showing cis-1,3-D and trans-1,3-D




b) Expanded scale to show impurities




Chromatographic conditions are listed in Section III.D.
Components and approximate retention times are listed in Figure 3.

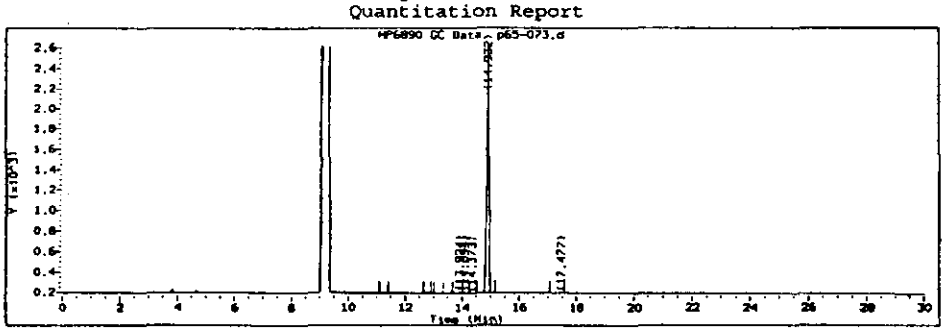
# VII. FIGURES

## Figure 6

### Representative Chromatogram of cis-1,3-Dichloropropene (TSN100275)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-073.d Page 1
Report Date 02/22/1999 11:47

Dow AgroSciences LLC
Quantitation Report



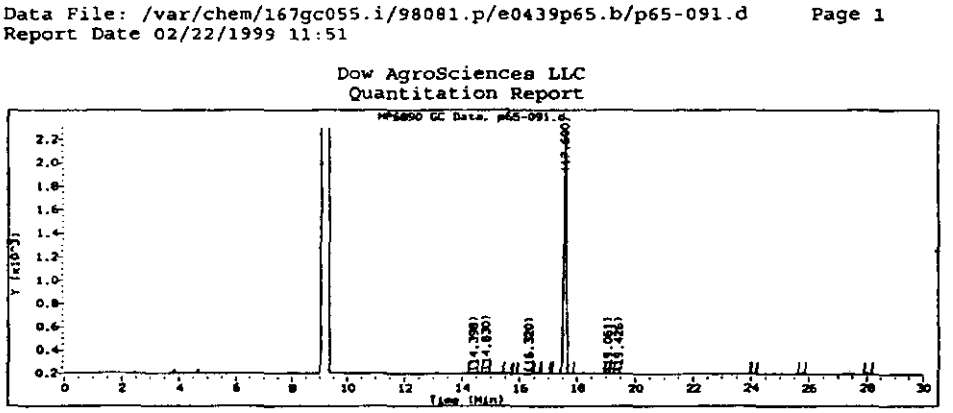

Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

# VII. FIGURES

## Figure 7

Representative Chromatogram of trans-1,3-Dichloropropene (TSN100276)



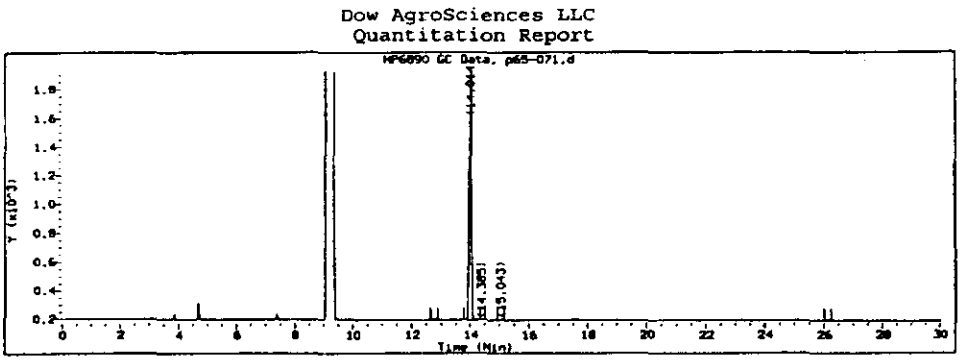

Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

# VII. FIGURES

## Figure 8

Representative Chromatogram of 2-Chloro-2-methylpentane (AGR238091)




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

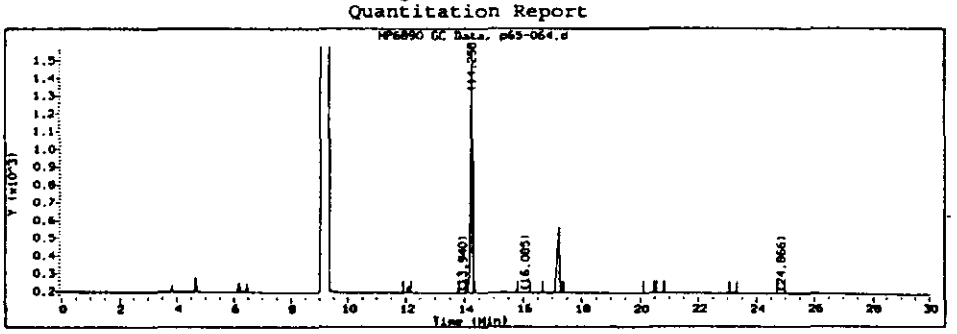
# VII. FIGURES

## Figure 9

### Representative Chromatogram of 2-Chloro-4-methylpentane (TSN101687)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-064.d Page 1
Report Date 02/26/1999 08:31

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

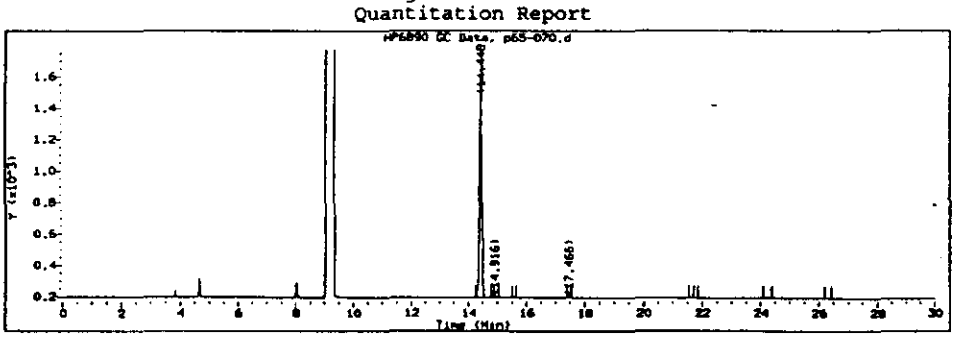
# VII. FIGURES

## Figure 10

## Representative Chromatogram of 2-Chloro-2,3-dimethylbutane (AGR238087)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-070.d Page 1
Report Date 02/22/1999 11:47

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

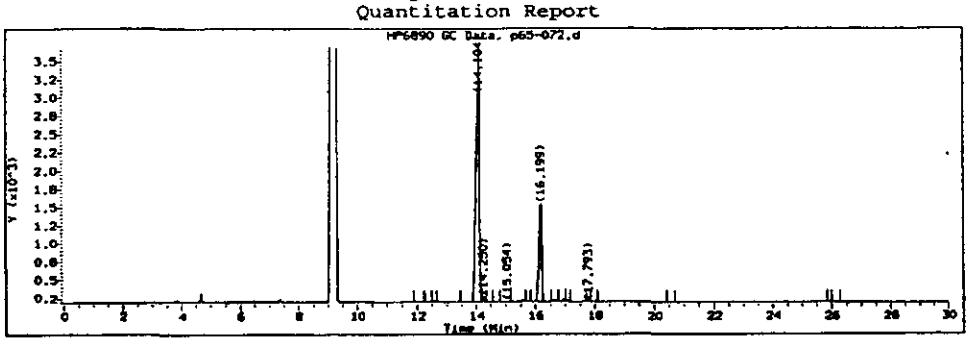
# VII. FIGURES

## Figure 11

Representative Chromatogram of 3-Chloro-2-methylpentane (minor peak) and 2-Chloro-2-methylpentane (major peak) (TSN101695)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-072.d Page 1
Report Date 02/22/1999 11:47

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

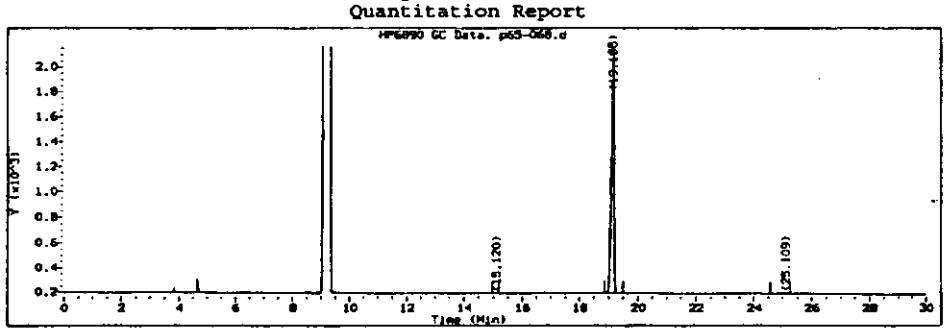
# VII. FIGURES

## Figure 12

## Representative Chromatogram of 1,3-Dichloropropane (AGR238090)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-068.d Page 1
Report Date 02/26/1999 08:32

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

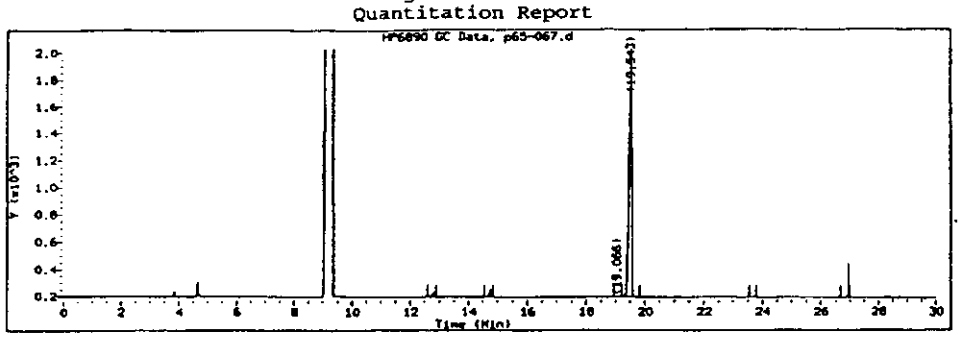
# VII. FIGURES

## Figure 13

## Representative Chromatogram of 1,2,2-Trichloropropane (AGR238089)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-067.d Page 1
Report Date 02/26/1999 08:32

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

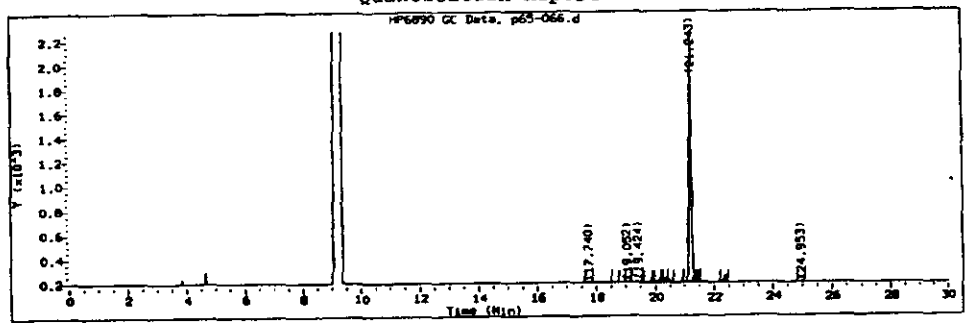
# VII. FIGURES

## Figure 14

## Representative Chromatogram of cis-1,3,3-Trichloropropene (AGR238088)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-066.d Page 1
Report Date 02/26/1999 08:32

Dow AgroSciences LLC
Quantitation Report




Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

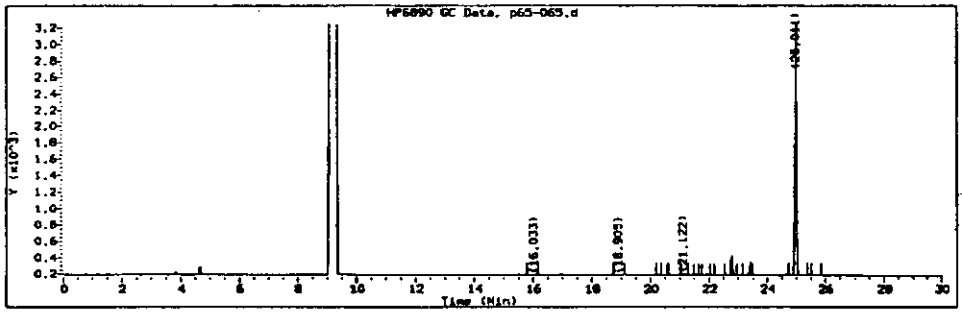
# VII. FIGURES

## Figure 15

## Representative Chromatogram of trans-1,3,3-Trichloropropene (AGR238086)

Data File: /var/chem/167gc055.i/98081.p/e0439p65.b/p65-065.d Page 1
Report Date 02/26/1999 08:32

Dow AgroSciences LLC
Quantitation Report



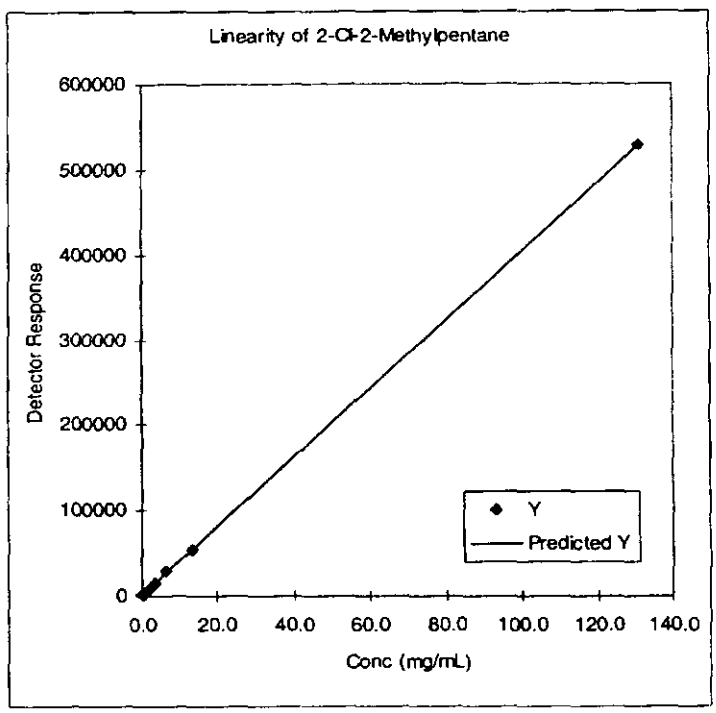

Chromatographic conditions are listed in Section III.D.

| Component | Standard | Approx. Retention Time (min) |
|---|---|---|
| 2-Cl-2-methylpentane | TSN101695, AGR238091 | 14.1 |
| 2-Cl-4-methylpentane | TSN101687 | 14.3 |
| 2-Cl-2,3-dimethylbutane | AGR238087 | 14.45 |
| **cis-1,3-dichloropropene** | TSN100275 | 14.9 |
| 3-Cl-2-methylpentane | TSN101695 | 16.2 |
| **trans-1,3-dichloropropene** | TSN100276 | 17.6 |
| 1,3-Dichloropropane | AGR238090 | 19.2 |
| 1,2,2-Trichloropropane | AGR238089 | 19.5 |
| cis-1,3,3-trichloropropene | AGR238088 | 21.2 |
| trans-1,3,3-trichloropropene | AGR238086 | 25.0 |

# VII. FIGURES

## Figure 16

Linearity of 2-Chloro-2-methylpentane

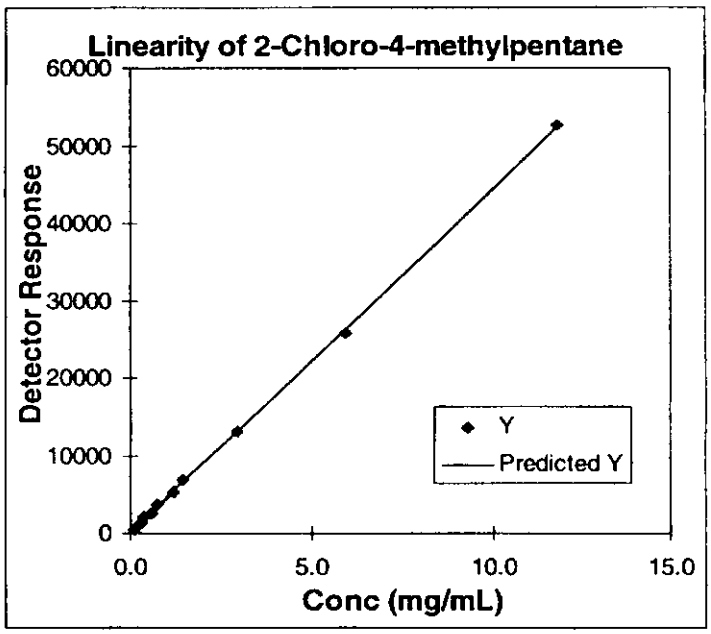



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.164 | 1082 |
| 0.328 | 1528 |
| 0.876 | 3862 |
| 1.751 | 7583 |
| 2.388 | 10371 |
| 3.283 | 14347 |
| 6.566 | 29826 |
| 13.133 | 54204 |
| 131.328 | 528663 |

Slope = 4018.863
y-axis intercept = 1020.277
Regression Coefficient = 0.99997
Observations = 9

# VII. FIGURES
## Figure 17

Linearity of 2-Chloro-4-methylentane



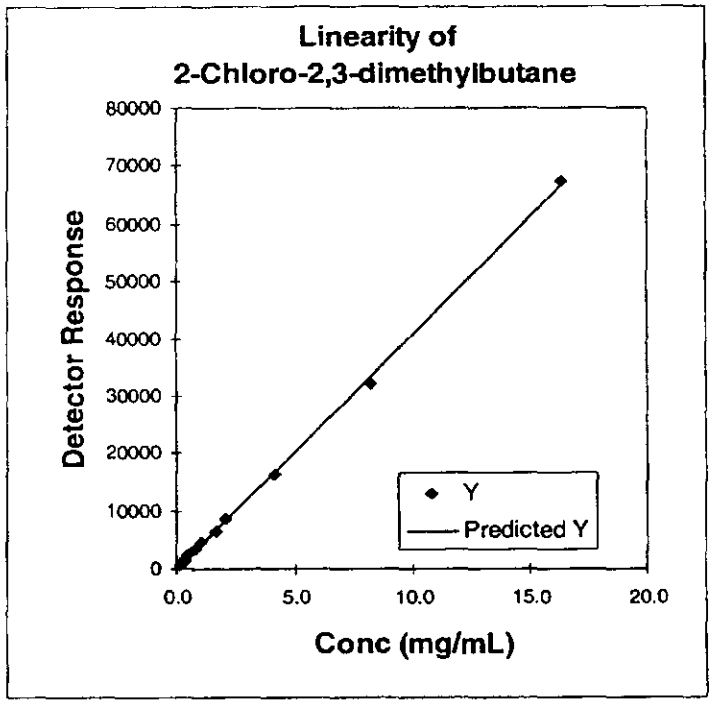

| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.092 | 499 |
| 0.185 | 1024 |
| 0.296 | 1289 |
| 0.370 | 2015 |
| 0.591 | 2615 |
| 0.739 | 3661 |
| 1.183 | 5237 |
| 1.478 | 6980 |
| 2.957 | 13030 |
| 5.913 | 25854 |
| 11.827 | 52660 |

Slope = 4421.988
y-axis intercept = 138.7981
Regression Coefficient = 0.999779
Observations = 11

# VII. FIGURES

## Figure 18

Linearity of 2-Chloro-2,3-dimethylbutane

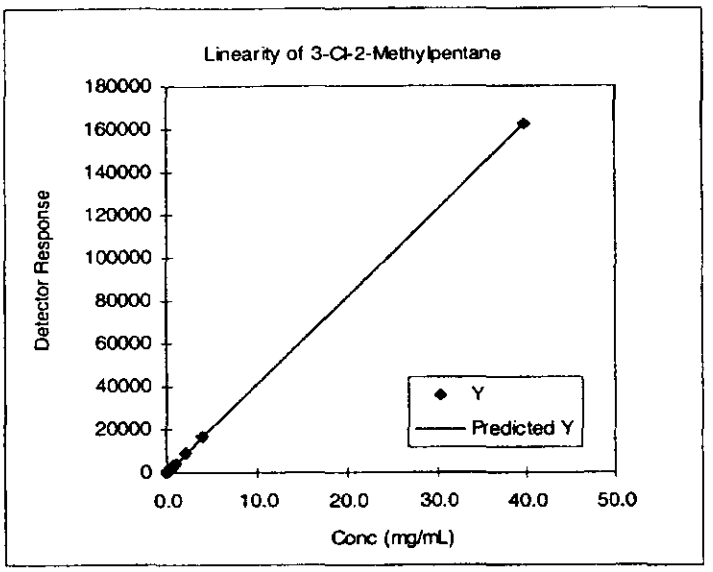



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.128 | 620 |
| 0.256 | 1263 |
| 0.409 | 1606 |
| 0.512 | 2481 |
| 0.818 | 3256 |
| 1.023 | 4560 |
| 1.637 | 6532 |
| 2.046 | 8717 |
| 4.092 | 16257 |
| 8.184 | 32235 |
| 16.369 | 67232 |

Slope = 4068.082
y-axis intercept = 40.65927
Regression Coefficient = 0.999454
Observations = 11

# VII. FIGURES
## Figure 19

Linearity of 3-Chloro-2-methylpentane

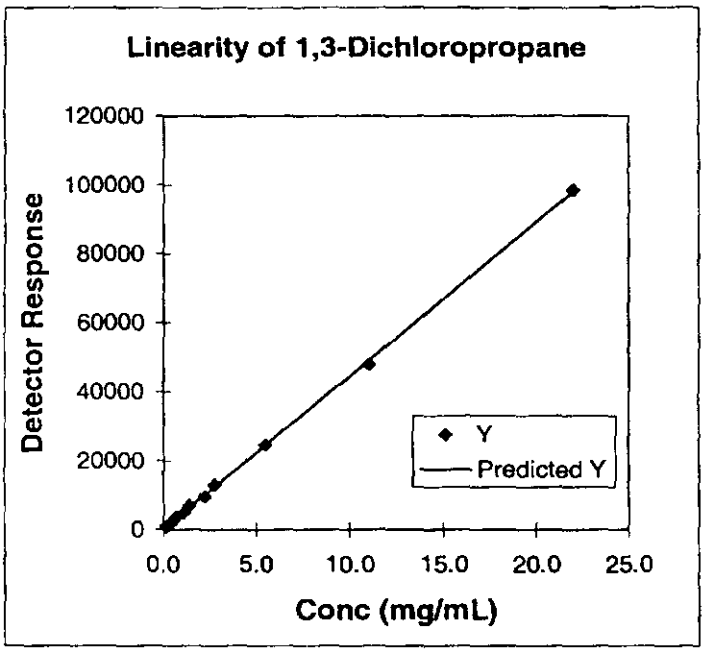



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.0497 | 372 |
| 0.0994 | 514 |
| 0.265 | 1199 |
| 0.530 | 2327 |
| 0.723 | 3179 |
| 0.994 | 4427 |
| 1.987 | 9453 |
| 3.974 | 16536 |
| 39.744 | 162456 |

Slope = 4079.806
y-axis intercept = 348.9076
Regression Coefficient = 0.999947
Observations = 9

## VII. FIGURES

Figure 20
Linearity of 1,3-Dichloropropane

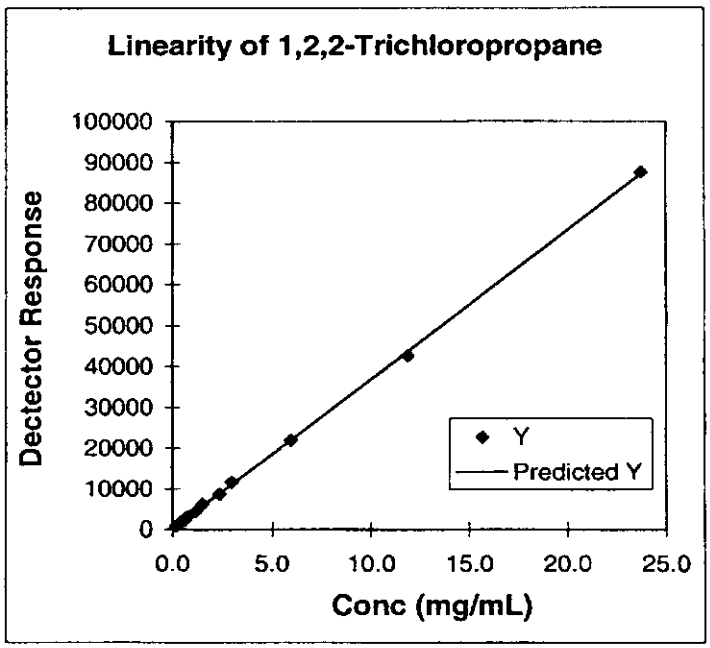



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.172 | 868 |
| 0.344 | 1854 |
| 0.550 | 2510 |
| 0.688 | 3566 |
| 1.101 | 4835 |
| 1.376 | 6954 |
| 2.201 | 9609 |
| 2.752 | 13102 |
| 5.504 | 24594 |
| 11.007 | 47978 |
| 22.014 | 98476 |

Slope = 4439.833
y-axis intercept = 229.5268
Regression Coefficient = 0.999693
Observations = 11

VII. FIGURES

Figure 21
Linearity of 1,2,2-Trichloropropane

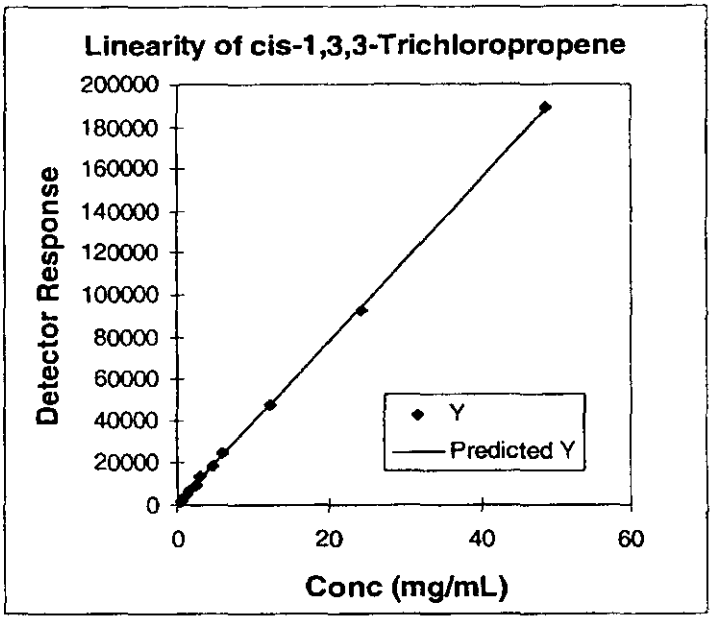



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.185 | 812 |
| 0.371 | 1628 |
| 0.594 | 2278 |
| 0.742 | 3107 |
| 1.187 | 4419 |
| 1.484 | 6125 |
| 2.374 | 8643 |
| 2.968 | 11601 |
| 5.936 | 21917 |
| 11.871 | 42678 |
| 23.742 | 87581 |

Slope = 3660.779
y-axis intercept = 220.875
Regression Coefficient = 0.999736
Observations = 11

## VII. FIGURES

Figure 22
Linearity of cis-1,3,3-Trichloropropene

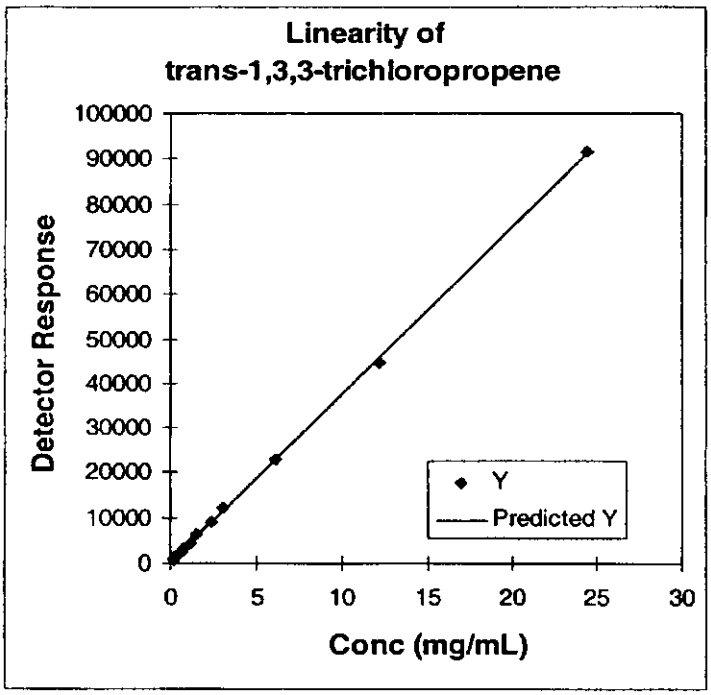



| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.38 | 1701 |
| 0.76 | 3499 |
| 1.21 | 4682 |
| 1.51 | 6810 |
| 2.42 | 9445 |
| 3.03 | 13162 |
| 4.85 | 18485 |
| 6.06 | 24839 |
| 12.11 | 47389 |
| 24.23 | 92162 |
| 48.46 | 188834 |

Slope = 3870.231
y-axis intercept = 416.5894
Regression Coefficient = 0.999753
Observations = 11

## VII. FIGURES

Figure 23
Linearity of trans-1,3,3-Trichloropropene




| Concentration (mg/mL) | Detector Response |
|---|---|
| 0.19 | 810 |
| 0.38 | 1718 |
| 0.61 | 2375 |
| 0.76 | 3338 |
| 1.22 | 4443 |
| 1.52 | 6463 |
| 2.44 | 8940 |
| 3.05 | 12164 |
| 6.10 | 23096 |
| 12.19 | 44670 |
| 24.39 | 91519 |

Slope = 3726.497
y-axis intercept = 236.3419
Regression Coefficient = 0.999712
Observations = 11

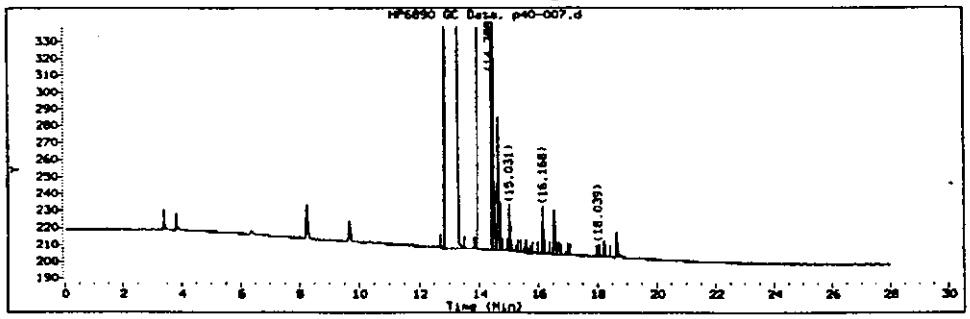
# VII. FIGURES

## Figure 24

### Representative Chromatogram using DB-1 Column for Separation of Telone Impurities

Data File: /var/chem/167gc055.i/98081.p/e0439p39.b/p40-007.d Page 1
Report Date 08/11/1999 16:13

Dow AgroSciences LLC
Quantitation Report




| | | | | |
|---|---|---|---|---|
| Instrument: | Hewlett-Packard 6890 or equivalent | | | |
| Column: | J&W DB-1 60 m x 0.25 mm 1.0 micron film | | | |
| Inlet: | Split | | | |
| Split Ratio: | 38 | | | |
| Purge flow: | 1.2 mL/min | | | |
| Inlet Temperature: | 150°C | | | |
| Oven Temperature: | Initial 50°C | | | |
| Initial Time: | 3.0 min | | | |
| Oven Gradient: | Level | Rate °C/min | Final °C | Final Time (min) |
| | 1 | 3.0 | 110 | 5 |
| Detector: | TCD | | | |
| Detector Temperature: | 155°C | | | |
| Back Inlet: | Constant Flow | | | |
| Carrier Flow: | 72 mL/min | | | |
| Column Flow Rate: | 1.9 mL/min | | | |
| Injection Volume: | 1 µL | | | |
| Run Time: | 28 min. | | | |
| Time to prepare and analyze one sample: | 40 min | | | |

# VIII. APPENDIX

## Analytical Method Summary: Impurities in Telone* II

A. Preparation of Standard Solution for Quantitation

Weigh approximately 100-200 mg of each impurity, recording the weight to the nearest 0.1 mg, into a 10 mL volumetric flask. Dilute to volume with ethyl acetate.
Transfer 1 mL of the solution to a 10 mL volumetric flask and dilute to volume with ethyl acetate, resulting in a standard solution containing each impurity at approximately 1-2 mg/mL.

B. Preparation of Telone Solution for Quantitation

Volumetrically pipet 4 mL of Telone II into a 10 mL volumetric flask, recording the weight to the nearest 0.1 mg. Dilute to volume with ethyl acetate. (Alternatively, the weight of the sample may be calculated using a density of 1.21 g/mL if 4 mL is volumetrically transferred to a 10 mL volumetric flask prior to diluting to volume.

C. Gas Chromatographic Conditions

| | | | | | |
|---|---|---|---|---|---|
| Instrument: | Hewlett-Packard 6890 or equivalent | | | | |
| Column: | J&W DB-1701 60 m x 0.32 mm 1.0 micron film | | | | |
| Inlet: | Split | | | | |
| Split Ratio: | 38:1 | | | | |
| Purge flow: | 1.2 mL/min | | | | |
| Inlet Temperature: | 150°C | | | | |
| Oven Temperature: | Initial 40°C | | | | |
| Initial Time: | 2.0 min | | | | |
| Oven Gradient: | Level | | Rate °C/min | Final °C | Final Time (min) |
| | 1 | 5.0 | 80 | 7.5 | |
| | 2 | 5.0 | 110 | 1.0 | |
| | 3 | 25.0 | 270 | 0 | |
| Detector: | TCD | | | | |
| Detector Temperature: | 280°C | | | | |
| Inlet: | Constant Flow | | | | |
| Column Flow: | 1.9 mL/min | | | | |
| Injection Volume: | 2 µL | | | | |
| Run Time: | 30 min | | | | |

*Trademark of Dow AgroSciences LLC

# VIII. APPENDIX

D. Calculation

Response Factor for Calibration Standard:

$$Rf = \frac{A \times P}{B}$$

Where: Rf = response factor for standard
A = weight (mg), or concentration (mg/mL) of standard
P = purity of standard, expressed as decimal
B = peak area of standard in calibration standard

Concentration of Component in Sample:

$$\% \text{ Component} = \frac{Rf \times D \times E}{F}$$

Where: Rf = response factor for component in standard, calculated above
D = peak area of component in sample
E = multiplication factor (used to adjust for difference in dilution of standard and sample and to express value as weight percent)
F = sample weight, expressed in mg, or concentration (mg/mL)

Determination of Linearity:

The general equation for calculating a least square linear fit for the standard curve is as follows:

$$PR = mC + b$$

where PR = detector response for component
m = slope of the line.
C = concentration of component, in mg/mL.
b = y-axis intercept

For further details, refer to the main body of this report.