

CORNELL ANALYTICAL LABORATORIES—RESIDUE METHOD SERIES  
CORNELL UNIVERSITY  
GENEVA, NEW YORK 14456

I. TITLE: PR#1727

Residue Analysis of Permethrin (Parent). Version: #2A

Prepared by: A. M. Roloson Date: 2/15/95

Verified by: George Helfman Date: 5/31/96

Commodity(s) in this version: Avocado

II. REFERENCES

“Determination of FMC 33297 Residues in Plant, Animal and Soil Matrices by Gas Chromatography.” Glenn H. Fujie and Oliver H. Fullmar. J. Agric. Food Chem., Vol. 26:395, 1978.

III. STANDARDS

A. Primary Standards:

Permethrin #262, 99.4%, Exp. 9/98, Source: Zeneca, Storage refrigerator #2; 0°-10°C

B. Stock Solutions:

Permethrin #262, 100g/mL = 0.020g/200 mL acetone. Prep. 9/6/94; Exp. 9/6/95; Storage refrigerator #3; 0°-10°C

C. Working Standards:

Spiking standard of Permethrin, Prep. 11/28/94,

1.0 µg/mL = 1.0 mL of 100 µg/mL/100 mL hexane

Working standards of Permethrin, Prep. 1/24/94 in hexane

Working standards of Permethrin, 0.2 µg/mL = 20 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.1 µg/mL = 10 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.04 µg/mL = 4 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.02 µg/mL = 2 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.01 µg/mL = 1 mL of 1.0 µg/mL/100 mL

IV. REAGENTS

Hexane, Fisher Scientific, HPLC grade

Acetone-Redistilled, in-house

Sodium sulfate-Anhydrous

V. MATERIALS AND EQUIPMENT

Blenders, glass fritted funnels, etc.

Blow down apparatus (nitrogen)

VI. INSTRUMENTATION AND PARAMETERS

Room 235

Tracor 565 Gas Chromatograph

Column RTX-50, 0.32 mm x 30 m x 0.25 µm #1098B

Detector: Ni63 Electron Capture @ 300°C  
Inlet: 275°  
Oven: 150°C initial temperature 1 min hold  
15°/min.—rate  
260°C—final temperature, hold 5 min.  
Purge time (2.5 min) 15 mL/min Helium (Split/Splitless)  
Detector Makeup: 75 mL/min. Nitrogen  
Carrier: 25 psi Helium  
Strip Chart Recorder: 1 cm/min  
Attenuation: 2

## VIII. PROCEDURE FOR METHOD USED/SOPs

### A. Sample preparation and storage:

Avocados are macerated in a Hobart food chopper and the samples are stored frozen.

### B. Extraction:

- 1) Take 25g of sample and blend w/125 mL Hexane. Add 40g NaSO<sub>4</sub> and blend for ~2 min.
- 2) Filter through a Whatman #1 filter.
- 3) Add another 125 mL. Blend for ~3 min, rinse w/~20 mL Hexane and filter.
- 4) Bring up to a total of 250 mL w/hexane.
- 5) Take 20.0 mL (2.0g sample) blow down under nitrogen to 8-10 mL.
- 6) NOTE: This matrix needs more cleanup than peppers.

### C. Sep-Paks Clean-up

Prewash Sep-Pak with 5 mL 10% (ethyl) ether/hexane and then with 5 mL hexane.  
Load sample at 1 mL/min.  
Elute with 5 mL 10% ether/hexane at 1 mL/min.  
Collect both load and elutions.  
Dilute with hexane. Ready for GC,

### D. Quantitation:

2 µL of both samples and standard are injected and the two peak heights are added up. The data are then entered into the Cricket Graph program which generates a standard curve. The regression equation becomes the basis for the final calculation.

Sample Calculation:

2/6/95

#4, 40288C, 2.0 ppm recovery spike, 40 mL final volume, 2.0g aliquot wt.

Rt<sub>1</sub> Peak ht = 7.5 cm

Rt<sub>2</sub> Peak ht. = 8.2 cm  
15.7 cm

From standard curve of 2/6/95

$$y = 135.313x + 0.17$$

$$x = 0.1159 \mu\text{g/mL}$$

$$0.1159 \mu\text{g/mL} \times 40 \text{ mL} = 4.64 \mu\text{g} \approx 4.6 \mu\text{g}$$

$$4.6 \mu\text{g} \div 2.0\text{g} = 2.3 \text{ ppm}$$

$$2.3 \text{ ppm} \div 2.0 \text{ ppm} = 1.15 \times 100\% = 115\%$$

VIII. SENSITIVITY/DETECTION

A. Points on Standard Curve:

0.0, 0.01, 0.02, 0.04, 0.1, 0.2  $\mu\text{g/mL}$

IX. RECOVERY SPIKES

A. Spikes: in %

0.040 ppm	117 $\pm$ 4(4)
0.20 ppm	114 $\pm$ 5(4)
2.0 ppm	110 $\pm$ 5(3)

B. Estimated Detection Level: 0.025 ppm

C. Practical Detection Level: 0.040 ppm

CORNELL ANALYTICAL LABORATORIES—RESIDUE METHOD SERIES  
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I. TITLE PR#1727

Residue Analysis of Permethrin (Parent). Version: #2B

Prepared by: A. M. Roloson Date: 4/15/96

Verified by: George Helfman Date: 5/31/96

Commodity(s) in this version: Avocado

II. REFERENCES

“Determination of FMC 33297 Residues in Plant, Animal and Soil Matrices by Gas Chromatography.” Glenn H. Fujie and Oliver H. Fullmar. J. Agric. Food Chem., Vol. 26:395, 1978.

III. STANDARDS

A. Primary Standards:

Permethrin #262, 99.4%, Exp. 9/98, Source: Zeneca, Storage refrigerator #2; 0°-10°C

B. Stock Solutions:

Permethrin #262, 100 µg/mL = 0.0200g/200 mL acetone; Prep. 2/19/96; Exp. 2/19/97; Storage refrigerator #3; 0°-10°C

C. Working Standards:

Spiking standard of Permethrin, Prep. 3/13/96

10.0 µg/mL = 10 mL of 100 µg/mL/100 mL hexane

1.0 µg/mL = 1.0 mL of 100 µg/mL/100 mL hexane

Working standards of Permethrin, Prep. 2/20/96 in hexane

Working standards of Permethrin, 0.2 µg/mL = 20 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.1 µg/mL = 10 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.04 µg/mL = 4 mL of 1.0 µg/mL/100 mL

Working standards of Permethrin, 0.02 µg/mL = 2 mL of 1.0 µg/mL/100 mL

IV. REAGENTS

Hexane, Fisher Scientific, HPLC grade

Acetone-Redistilled, in-house

V. MATERIALS AND EQUIPMENT

Blenders, glass fritted funnels, etc.

Blow down apparatus (nitrogen)

## VI. INSTRUMENTATION AND PARAMETERS

Room 235

Tracor 565 Gas Chromatograph

Column RTx-50, 0.32 mm x 30 M x 0.5  $\mu$ m df

Detector: Ni63 Electron Capture @ 350°C

Inlet: 275° splitt/splittless time (1 min)

Oven: 200°C initial temperature 1 min hold

15°/min.—rate

300°C—final temperature, hold 6 min.

Purge time (2.5 min.) 15 mL/min Helium (Split/Splitless)

Total Detector Makeup: 87 mL/min. Column, Purge, Makeup

Carrier: 22 psi Helium

Strip Chart Recorder: 1 cm/min

Atten: 2

## VIII. PROCEDURE

### A. Sample preparation and storage:

Avocados are macerated in a Hobart food chopper and the samples are stored frozen.

### B. Extraction:

- 1) Take 25g of sample and blend w/125 mL 10% acetone; 90% hexane and blend for 2 min.
- 2) Filter through a Whatman #1.
- 3) Add another 125 mL and blend for 3 min, rinse w/~20 mL Hexane and filter.
- 4) Bring up to a total of 250 mL w/Hexane.
- 5) Take 20 mL or 2.0g, Blow down under nitrogen to 8-10 mL.
- 6) NOTE: This matrix needs more cleanup than peppers.

### Sep-Pak Clean-up Procedure

Prewash Sep-Pak with 5 mL 10% (Ethyl) Ether/Hexane and then with 5 mL hexane.

Load sample at 1 mL/min.

Elute with 5 mL 10% Ether/Hexane at 1 mL/min.

Collect both Load and Elutions

Dilute with Hexane. Ready for GC.

### D. Quantitation:

2  $\mu$ L of both samples and standard are injected and the two peak heights are added up. The data are then entered into the Cricket Graph program which generates a standard curve. The regression equation becomes the basis for the final calculation.

Sample Calculation:

4/2/96

#6, 60059C, 0.50 ppm recovery spike, 10 mL finale volume, 2.0g aliquot wt.

RT<sub>1</sub>, Peak ht. 3.5 cm

RT<sub>2</sub>, Peak ht. 2.8 cm

6.3 cm

From standard curve of 4/2/96, use 6.3 cm for y and solve for x

$$y = 74.748x + 0.289$$

$$x = 0.0881 \mu\text{g/mL} \approx 0.088 \mu\text{g/mL}$$

$$0.088 \mu\text{g/mL} \times 10 \text{ mL} = 0.88 \mu\text{g}$$

$$0.88 \mu\text{g} \div 2.0\text{g} = 0.44 \text{ ppm}$$

$$0.44 \text{ ppm} \div 0.50 \text{ ppm} = 0.88 \times 100\% = 88\%$$

### VIII. SENSITIVITY/DETECTION

A. Points on Standard Curve:

0.0, 0.02, 0.04, 0.1, 0.2  $\mu\text{g/mL}$

### IX. RECOVERIES

A. Spikes: %

0.10 ppm      82 $\pm$ 22(4)

0.50 ppm      84 $\pm$ 14(7)

1.0 ppm        82 $\pm$ 17(6)

B. Estimated Detection Level: 0.050 ppm

C. Practical Detection Level: 0.10 ppm

### X. COMMENTS

Due to caking of sample, sodium sulfate was deleted. Due to a higher moisture content or some other variables, the method was modified and changes verified from Version #2A.

CORNELL ANALYTICAL LABORATORIES—RESIDUE METHOD SERIES  
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GENEVA, NEW YORK 14456

I. TITLE: #1727

Residue Analysis of Permethrin Metabolites. Version: #2C

Prepared by: A. M. Roloson Date: 10/5/95

Verified by: George Helfman Date: 6/5/96

Commodity(s) in this version: Avocado

II. REFERENCE METHOD

Methodology for the Determination of Dichlorovinyl Acid and m-Phenoxybenzyl Alcohol Residues in/on Grapes and Grape Processing Products, (FMC). J. W. Stearns. (FMC) RAN-0201M. 1987.

III. ANALYTICAL STANDARDS

A. Primary Standard:

Dichlorovinyl Acid (cis, trans) #263, Source: Zeneca, 99.5%, Storage: 0°-4°C, Refrigerator #2, exp.: 6/99.

B. Stock Solution:

#263-Acid 0.020g/200.0 mL = 100 µg/mL in acetone; prep. 11/22/94, exp. 11/22/95

C. Working and Spiking Standards:

#263 Acid 1.0 µg/mL = 1.0 mL of 100 µg/mL/100.0 mL in hexane; prep. 8/16/95; exp. 11/22/95, used only for spiking.

#263 Acid 1.0 µg/mL = 1.0 mL of 100 µg/mL/100 mL in MeCl<sub>2</sub>; prep. 8/16/95; exp. 11/22/95, used only for preparing working standard.

D. Working Standards for Derivatization: (Prepared together with each analytical set)

1 mL of 1.0 µg/mL #263 in MeCl<sub>2</sub> → 50.0 mL hexane = 0.020 µg/mL

From the derivatized working standards above, simple dilutions with hexane are done as follows:

5.0 mL of 0.020 µg/mL/10 mL = 0.010 µg/mL

5.0 mL of 0.020 µg/mL/20 mL = 0.0050 µg/mL

5.0 mL of 0.020 µg/mL/40 mL = 0.0025 µg/mL

IV. REAGENTS AND MATERIALS

Hexane - HPLC, Fisher

Tetrabutylammonium phosphate - 0.5 M, ACROS

Pentafluorobenzyl Bromide 99+%, Aldrich

Florisil - 60-80 mesh, Fisher

Sodium Sulfate-anhydrous, Fisher

NaOH - ACS, Fisher  
Ethyl Ether - ACS, Fisher  
HCl - ACS, Fisher  
H<sub>2</sub>O - HPLC, Fisher  
MeCl<sub>2</sub> - Potter - Redistilled in house

1N NaOH = 10g NaOH/250 mL in H<sub>2</sub>O  
0.1 N NaOH = 1g NaOH/250 mL in H<sub>2</sub>O  
0.2 M NaOH = 2g NaOH ÷ 250 mL in H<sub>2</sub>O  
0.1M tetrabutylammonium phosphate/0.2M NaOH from 10.0 mL of 0.5M (TBP)/50 mL in 0.2 M NaOH

## V. EQUIPMENT

Basic Lab Equipment and Glassware

## VI. INSTRUMENTATION AND PARAMETERS

Room 235

**Tracor 565 Gas Chromatograph with E.C. Detector (instead of GC/MS)**

**Column RTX-5, 0.53 mm x 30 m x 1 μm #10255**

**Detector: 350°C**

**Spittless injection - 27 sec. injection time**

**Inlet: 275°C**

**Oven: 150°C initial temperature, 10 min hold**

**6°/min.—rate**

**300°C—final temperature, hold 3 min.**

**Detector flow: 37 mL/min. Nitrogen (Makeup and Purge)**

**Carrier 6 psi Helium (17 mL/min)**

**Strip Chart Recorder 1 cm/min**

**Atten: 5**

## VII. PROCEDURE FOR METHOD USED/SOPs

### A. Sample preparation and storage:

The avocados are cut in halves and the pits removed then chopped in a Hobart food chopper and subsampled. Samples are stored frozen.

### B. Extraction and Partition of DCVA

Note: Sample fortification is done at this point.

- 1) 25g sample is blended for ~5 min. with 200 mL **hexane** (HPLC). **Two mL of HCl** is added for efficient extraction prior to blending.
- 2) The extract is poured off and the chopped sample is rinsed with ~ 40 mL of hexane and combined with the original extract.
- 3) The extract is then brought up to a total of 250 mL with hexane.
- 4) A 50 mL aliquot (5g sample) is then placed in a separatory funnel.
- 5) The sample is partitioned three times with 3 mL of 1N NaOH + 25 mL H<sub>2</sub>O. Collect the lower layers and let the little bit of hexane separate out in another separatory funnel during the partitions.
- 6) Split the final sample in half (2.5g aliquot). Add 2 mL HCl.
- 7) Reflux for 1 hr., cool. (NOTE: Use cold-cool water)

- 8) Rinse condenser with 10-20 mL MeCl<sub>2</sub> and 5 mL of 0.1N NaOH.
- 9) Place extracts in separatory funnel and add 5g NaCl.
- 10) Shake out with another 10 mL MeCl<sub>2</sub>.

Derivatization:

- 1) Add 1 mL 0.1M Tetrabutylammonium phosphate/.2N NaOH and 30 μL pentafluorobenzyl bromide to the sample.
- 2) Shake for 1-1/2 hr.
- 3) Add 10 mL hexane to stop the reaction.
- 4) Lightly blow down to <10 mL under N<sub>2</sub>.

Acid Cleanup:

- 1) Prepare Florisil column by using 2g of activated Florisil.
- 2) Then add 2g anhydrous sodium sulfate.
- 3) Wash with 10 mL hexane.
- 4) Load sample from blow (<10 mL).
- 5) Elute with 20 mL of 20% ethyl ether/hexane.
- 6) Ready for G.C.

NOTE: Both load and eluate are collected.

D. Quantitation:

The cis and trans acids are injected together but reported individually.

Sample calculation: 9/12/95

C'gram #13, 50 mL volume, 2.5g aliquot wt.

40290D spike 0.4 ppm

RT<sub>1</sub> = cis ≈ 8.9 cm

from the standard curve of 9/12/95, use 8.9 cm for y and solve for x

$$y = 1949.006 x^{1.245}$$

$$r^2 = 0.996$$

$$y \div 1949.006 = x^{1.245}$$

$$8.9 \div 1949.006 = 0.0045664 = x^{1.245}$$

as done on TI 30 calculator to solve for x

$$0.0045664(\text{inv})(y^x)(1.245) = 0.0131868 \approx 0.0132 \mu\text{g/mL}$$

$$0.0132 \mu\text{g/mL} \times 50 \text{ mL} = 0.659 \mu\text{g} \approx 0.66 \mu\text{g}$$

$$0.66 \mu\text{g} \div 2.5 \text{ g} = 0.26 \text{ ppm}$$

$$0.26 \text{ ppm} \div 0.40 \text{ ppm} = 0.65 \times 100\% = 65\%$$

VIII. SENSITIVITY/DETECTION

A. Points on Standard Curve:

DCVA

0.0 μg/mL

0.0025 μg/mL

0.0050 μg/mL

0.010 μg/mL

0.020 μg/mL

IX. RECOVERIES

A. Spikes: (in %)

	<u>cis Acid</u>	<u>Trans Acid</u>
0.10 ppm	99±19(10)	83±6(7)
0.20 ppm	88±8(5)	79±6(5)
0.40 ppm	57±7(9)	68±5(9)

B. Estimated Detection Level: 0.050 ppm

C. Practical Detection Level: 0.10 ppm (cis, trans)

X. COMMENTS

In this part of the study, power equation was used for the daily standard curve in order to get the best curve fit  $r^2 \geq 0.98$ . The sample calculation on p.3 shows how to use this power equation.

CORNELL ANALYTICAL LABORATORIES—RESIDUE METHOD SERIES  
CORNELL UNIVERSITY  
GENEVA, NEW YORK 14456

I. TITLE: #1727

Residue Analysis of Permethrin Metabolites. Version: #2D

Prepared by: A. M. Roloson Date: 5/4/96

Verified by: George Helfman Date:

Commodity(s) in this version: Avocado

II. REFERENCE METHOD

Methodology for the Determination of Dichlorovinyl Acid and m-Phenoxybenzyl Alcohol Residues in/on Grapes and Grape Processing Products, (FMC). J. W. Stearns. (FMC) RAN-0201M. 1987.

III. ANALYTICAL STANDARDS

A. Primary Standard:

Dichlorovinyl Acid (cis, trans) #263, Source: Zeneca, 99.5%, Storage: 0°-4°C, Refrigerator #2, exp.: 6/99.

B. Stock Solution:

#263-Acid 0.020g/200.0 mL = 100 µg/mL in acetone; prep. 4/23/96, exp. 4/23/97

C. Working and Spiking Standards:

#263 Acid 1.0 µg/mL = 1.0 mL of 100 µg/mL/100.0 mL in hexane; prep. 4/23/96; exp. 4/23/97, used only for spiking.

#263 Acid 1.0 µg/mL = 1.0 mL of 100 µg/mL/100 mL in MeCl<sub>2</sub>; prep. 4/23/96; exp. 4/23/97, used only for preparing working standard.

D. Working Standards for Derivatization: (Prepared at time of sample preparation)

1 mL of 1.0 µg/mL #263 in MeCl<sub>2</sub> Æ 50.0 mL hexane = 0.020 µg/mL

From the derivatized working standards above, simple dilutions with hexane are done as follows:

5.0 mL of 0.020 µg/mL/10 mL = 0.010 µg/mL

5.0 mL of 0.020 µg/mL/20 mL = 0.0050 µg/mL

5.0 mL of 0.020 µg/mL/40 mL = 0.0025 µg/mL

IV. REAGENTS AND MATERIALS

Hexane - HPLC, Fisher

Tetrabutylammonium phosphate - 0.5 M, ACROS

Pentafluorobenzyl Bromide 99+%, Aldrich

Florisil - 60-80 mesh, Fisher

Sodium Sulfate-anhydrous, Fisher

NaOH - ACS, Fisher

Ethyl Ether - ACS, Fisher  
HCl - ACS, Fisher  
H<sub>2</sub>O - HPLC, Fisher  
MeCl<sub>2</sub> - Potter - Redistilled in house

1N NaOH = 10g NaOH/250 mL in H<sub>2</sub>O  
0.1 N NaOH = 1g NaOH/250 mL in H<sub>2</sub>O  
0.2 M NaOH = 2g NaOH  $\square$  250 mL in H<sub>2</sub>O  
0.1M tetrabutylammonium phosphate/0.2M NaOH from 10.0 mL of 0.5M (TBP)/50 mL in 0.2 M NaOH

V. EQUIPMENT

Basic Lab Equipment and Glassware

VI. INSTRUMENTATION AND PARAMETERS

Room 235  
**Tracor 565 Gas Chromatograph with E.C. Detector (instead of GC/MS)**  
**Column RTX-5, 0.53 mm x 30 m x 1  $\mu$ m #10255**  
**Detector: 350°C**  
**Spittless injection - 27 sec. injection time**  
**Inlet: 275°C**  
**Oven: 150°C initial temperature, 5 min hold**  
**6°/min.—rate**  
**300°C—final temperature, hold 3 min.**  
**Detector flow: 37 mL/min. Nitrogen (Makeup and Purge)**  
**Carrier 6 psi Helium (17 mL/min)**  
**Strip Chart Recorder 1 cm/min**  
**Atten: 5**

VII. PROCEDURE FOR METHOD USED/SOPs

A. Sample preparation and storage:

The avocados are cut in halves and the pits removed then chopped in a Hobart food chopper and subsampled. Samples are stored frozen.

B. Extraction and Partition of DCVA

Note: Sample fortification is done at this point.

- 1) 25g sample is blended for ~5 min. with 200 mL **hexane** (HPLC). **Two mL of HCl** is added for efficient extraction prior to blending.
- 2) The extract is poured off and the chopped sample is rinsed with ~ 40 mL of hexane and combined with the original extract.
- 3) The extract is then brought up to a total of 250 mL with hexane.
- 4) A 50 mL aliquot (5g sample) is then placed in a separatory funnel.
- 5) The sample is partitioned three times with 3 mL of 1N NaOH + 25 mL H<sub>2</sub>O. Collect the lower layers and let the little bit of hexane separate out in another separatory funnel during the partitions.
- 6) Split the final sample in half (2.5g aliquot). Add 2 mL HCl.
- 7) Reflux for 1 hr., cool. (NOTE: Use cold-cool water)
- 8) Rinse condenser with 10-20 mL MeCl<sub>2</sub> and 5 mL of 0.1N NaOH.
- 9) Place extracts in separatory funnel and add 5g NaCl.

10) Shake out with another 10 mL MeCl<sub>2</sub>.

Derivatization:

- 1) Add 1 mL 0.1M Tetrabutylammonium phosphate/.2N NaOH and 30 µL pentafluorobenzyl bromide to the sample.
- 2) Shake for 1-1/2 hr.
- 3) Add 10 mL hexane to stop the reaction.
- 4) Lightly blow down to <10 mL under N<sub>2</sub>. Discard water fraction of bottom

Acid Cleanup:

- 1) Prepare Florisil column by using 2g of activated Florisil.
- 2) Then add 2g anhydrous sodium sulfate.
- 3) Wash with 10 mL hexane.
- 4) Load sample from blow down (<10 mL).
- 5) Elute with 20 mL of 20% ethyl ether/hexane.
- 6) Ready for G.C.

NOTE: Both load and eluate are collected.

D. Quantitation:

The cis and trans acids are injected together but reported individually.

Sample calculation: 6/6/96 using standard curve

$$y = 855.652x - 1.255$$

$$y = 1.8 \text{ cm}$$

solve for x

C'gram #308, 2 µL injection, 100 mL volume, 2.5g aliquot wt.

600091 spike 0.20 ppm

$$RT_1 = \text{cis}^a 1.8 \text{ cm}$$

$$x = 0.0035704 \text{ µg/mL}$$

$$0.0035704 \text{ µg/mL} \times 100 \text{ mL} = 0.3570 \text{ µg}$$

$$0.3570 \text{ µg} \div 2.5\text{g} = 0.14 \text{ ppm}$$

$$0.14 \text{ ppm} \div 0.2 = 0.70 \times 100\% = 70\%$$

VIII. SENSITIVITY/DETECTION

A. Points on Standard Curve:

DCVA

0.0 µg/mL

0.0025 µg/mL

0.005 µg/mL

0.010 µg/mL

0.020 µg/mL

IX. RECOVERIES

A. Spikes: (in %)

	<u>cis acid</u>	<u>trans acid</u>
0.10 ppm	88±13(6)	103±6(6)
0.20 ppm	71±9(6)	77±7(6)
0.40 ppm	57±4(3)	65±5(3)

B. Estimated Detection Level: 0.050 ppm

C. Practical Detection Level: 0.10 ppm