UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OCT 25 1994

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#1F03995 (CBTS #14546; Barcode #D208444). Fenbuconazole on Pecans. Evaluation of Method Validation Report dated 10/12/94.

FROM: Nancy Dodd, Chemist
Tolerance Petition Section II
Chemistry Branch I- Tolerance Support
Health Effects Division (7509C)

THROUGH: Richard Loranger, Ph.D., Acting Chief
Chemistry Branch I- Tolerance Support
Health Effects Division (7509C)

TO: Cynthia Giles-Parker, PM#22
Herbicide-Fungicide Branch
Registration Division (7505C)

and

Albin Kocialski, Section Head
Registration Section
Chemical Coordination Branch
Health Effects Division (7509C)

The Analytical Chemistry Branch has informed CBTS of the results of their method validation for fenbuconazole (RH-7592) on pecans (Paul Golden, 10/12/94). The method which was tested was "Residue Analytical Method for Parent RH-7592 and its Metabolites RH-9129, RH-9130, and RH-6467 in Pecans", Rohm and Haas Technical Report No. 34-91-14, 3/5/91 (MRID #418925-03).

Pecans were fortified at levels of 0.05 and 0.10 ppm with RH-7592 and its metabolites RH-9130 and RH-9129. The recoveries are repeated below:

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| Commodity | Chemical Added | PPM Added | PPM Found | Percent Recovery |
|---|---|---|---|---|
| pecans | RH-7592 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.054 | 108 |
| | | 0.05 | 0.067 | 134 |
| | | 0.10 | 0.100 | 100 |
| | | 0.10 | 0.109 | 109 |
| pecans | RH-9130 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.053 | 106 |
| | | 0.05 | 0.065 | 130 |
| | | 0.10 | 0.101 | 101 |
| | | 0.10 | 0.106 | 106 |
| pecans | RH-9129 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.051 | 102 |
| | | 0.05 | 0.064 | 128 |
| | | 0.10 | 0.099 | 99.0 |
| | | 0.10 | 0.102 | 102 |

1. N.D.= <0.001 ppm for RH-7592; <0.002 ppm for RH-9130 and RH-9129

Instead of using calibration curves to determine sample concentrations, ACL determined sample concentrations from a ratio of sample response to the average of bracketing standard response.

EPA's Analytical Chemistry Laboratory (ACL) offered the following comment regarding the adequacy of the method:

> "The method provided acceptable recovery on pecans at the requested spiking levels for RH-7592, RH-9130, and RH-9129. There was evidence, however, of significant signal enhancement caused by the matrix. The data reported below are therefore believed to be artificially higher than actual. The data submitted by the registrant includes several high recovery values which may also be caused by a problem of signal enhancement. Additional clean-up procedures should be investigated to correct the problem of signal enhancement found at ACL."

EPA's Analytical Chemistry Laboratory indicated that "one chemist can have a set of six (6) samples ready for GC analysis within 8 hours, exclusive of the Soxhlet extraction time."

2

EPA's Analytical Chemistry Laboratory indicated that the method would be adequate as a data collection method but may not be acceptable as an enforcement method unless the following modifications are made:

1. Additional clean-up procedures are needed to correct the signal enhancement caused by the matrix.

2. EPA's Analytical Chemistry Laboratory safety policy precludes overnight Soxhlet extractions. The petitioner should be asked if the soxhlet extraction can be shortened.

3. The section on preparation of standard curves states that the response of the analytes "are usually quadratic in nature." If instrument response is not linear, standard and sample response must be carefully matched if a standard curve is not used for quantitation."

4. Average recovery values are used for calculating residue levels. This practice should not be incorporated in a tolerance enforcement method.

Conclusion/Recommendation

A satisfactory method trial has been conducted by EPA's Analytical Chemistry Laboratory for the method in Rohm and Haas Technical Report No. 34-91-14 for fenbuconazole, RH-9129, and RH-9130 on pecans. Although signal enhancement caused by the matrix may have occurred, recoveries are not excessively high and, in our judgement, are adequate for enforcement purposes. CBTS will not require that additional clean-up procedures be added to the method. However, the method should be revised to include the minor modifications (#'s 2 through 4 above). The revised method should be submitted so that it can be sent to FDA for publication in the Pesticide Analytical Manual (PAM) upon our recommendation for a permanent tolerance. This deficiency will remain outstanding pending receipt of the revised analytical method.

Attachment 1: 10/12/94 MTO report

cc with Attachment 1: SF, N. Dodd (CBTS), MTO File, PP#1F3995, PM#22, Albin Kocialski (CCB)

cc without Attachment 1: RF, Circu., E. Haeberer (CBTS), M. Bradley (CBTS), D. Marlow (ACB/BEAD), H. Hundley (ACB/BEAD)

RDI:E. Haeberer:10/24/94:M. Flood:10/24/94
7509C:CM#2:Rm804F:305-5681:N. Dodd:nd:10/24/94

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

ATTACHMENT 1

Analytical Chemistry Section
Building 306, BARC-East
Beltsville, Maryland 20705

OCT 12 1994

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

**MEMORANDUM**

SUBJECT: PP#1F3995. Method Validation of three chemicals on Pecans, RH-7592(Fenbuconazole), RH-9129, and RH-9130.

FROM: Paul E. Golden, Chemist
Analytical Chemistry Section

THRU: Harvey R. Hundley, Head
Analytical Chemistry Section

THRU: Donald A. Marlow, Chief
Analytical Chemistry Branch

TO: Elizabeth Haeberer, Head
Tolerance Petition Section II
Chemistry Branch I-Tolerance Support
Health Effects Division (H7509C)

**INTRODUCTION**

The Analytical Chemistry Laboratory was requested by the Chemistry Branch-I Tolerance Support to conduct a method validation for the fungicide Fenbuconazole (RH-7592) and two metabolites RH-9129 and RH-9130 on pecans. Analyses were requested to be run in duplicate using unfortified control samples and samples fortified at 0.05 ppm and 0.10 ppm.

The analytical method submitted for validation from Rohm and Haas Company was titled: "Residue Analytical Method for Parent RH-7592 and Its Metabolites RH-9129, RH-9130, and RH-6467 in Pecans"; Rohm and Haas Technical Report No. 34-91-14; dated March 5, 1991 (MRID 3418925-03).

**METHOD SUMMARY**

RH-7592 and its metabolites RH-9129 and RH-9130 are extracted from pecans by Soxhlet extraction with hexane/2-propanol. The extract is concentrated to an oily residue. The residue is dissolved in toluene/acetone and cleaned up on Silica gel and then Florisil. The samples are further cleaned up with a C-18 solid phase extraction cartridge. Residues are determined by gas chromatography using a 30 meter, 0.53mm ID RTx-35 capillary column and a nitrogen phosphorus detector.

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COMMENTS

1. All standards used for this method validation were supplied from RTP.

2. Column profiles were performed on the silica gel and Florisil cleanup media using a multi-component standard to verify proper eluent fraction collection. Pre-wash and post-wash fractions were collected before and after the prescribed collection fraction. Fenbuconazole and its metabolites eluted only in the fraction described by the method. The elution scheme did not require modification.

3. ACL utilized a Hewlett-Packard HP5890 Gas Chromatograph equipped with a nitrogen-phosphorus detector. Rohm and Haas used a Varian GC. Carrier and detector gas flows were adjusted to meet specifications for the HP 5890 NP detector.

4. The NP detector was operated at 290° C instead of 300 ° C and without the use of the detector make-up gas as cited in the method. The lower temperature was selected to minimize column bleed which shortens the lifetime of the detector.

5. The method prescribes the use of calibration curves to determine sample concentrations. ACL determined sample concentrations from a ratio of sample response to the average of bracketing standard response.

6. The method provided acceptable recovery on pecans at the requested spiking levels for RH-7592, RH-9130, and RH-9129. There was evidence, however, of significant signal enhancement caused by the matrix. The data reported below are therefore believed to be artificially higher than actual. The data submitted by the registrant includes several high recovery values which may also be caused by a problem of signal enhancement. Additional clean-up procedures should be investigated to correct the problem of signal enhancement found at ACL.

7. One chemist can have a set of six (6) samples ready for GC analysis within 8 hours, exclusive of the Soxhlet extraction time.

8. The sensitivity of the method based on the optimum parameters used, was estimated for this method validation. The estimates in pecans are as follows:

| Analyte | Estimated Limit of Detection |
|---|---|
| RH-7592 | 0.001 ppm |
| RH-9130 | 0.002 ppm |
| RH-9129 | 0.002 ppm |

9. A copy of ACL's method validation pre-review is attached as additional information for this study and should be referenced for additional minor modifications that should be made to the proposed enforcement method.

10. This method generally meets the requirements in the Pesticide Assessment Guidelines, Subdivision O, Section 171-4(b), but may not be acceptable as an enforcement method unless the above comments are addressed and incorporated into the procedure. (particularly note Comment 6)

**METHOD VALIDATION RESULTS:**

| Commodity | Chemical Added | PPM Added | PPM Found | Percent Recovery |
|---|---|---|---|---|
| Pecans | RH-7592 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.054 | 108 |
| | | 0.05 | 0.067 | 134 |
| | | 0.10 | 0.100 | 100 |
| | | 0.10 | 0.109 | 109 |
| Pecans | RH-9130 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.053 | 106 |
| | | 0.05 | 0.065 | 130 |
| | | 0.10 | 0.101 | 101 |
| | | 0.10 | 0.106 | 106 |
| Pecans | RH-9129 | 0.00 | N.D.[1] | - |
| | | 0.00 | N.D.[1] | - |
| | | 0.05 | 0.051 | 102 |
| | | 0.05 | 0.064 | 128 |
| | | 0.10 | 0.099 | 99.0 |
| | | 0.10 | 0.102 | 102 |

1. N.D. = Less than 0.001 ppm for RH-7592, less than 0.002 ppm for RH-9130, less than 0.002 ppm for RH-9129.

**Modifications to Method (major or minor):**

Refer to Comments 3 through 5.

**Special precautions to be taken:**

None

**Source of analytical standards:**

US EPA Pesticides Repository, RTP

**If derivatized standard is used, give source:**

N/A

**Instrumentation for quantitation:**

HP 5890 GLC/NP detection

**Instrumentation for confirmation:**

N/A

**If instrument parameters differ from those given in the method, list parameters used:**

Refer to Comments 3 & 4.

**Commercial sources for any special chemicals or apparatus:**

N/A

**Additional comments:**

See comment section of report.

**Chromatograms:**

Copies of standard curve, sample calculations, pecan controls, low and high level fortifications, and standards are included.

394-40,41

TMV Pre-review of Febuconazole (RH-7592)

Reviewed by: Everett S. Greer, Jr. ESG

Date: 4-8-94

Laboratory assignment number: B94-40,41

Analytes: RE-7592, RH-9129 and RH-9130

Commodities: Pecans

Method: Residue Analytical Method for Parent RH-7592 and its Metabolites RH-9129, RH-9310, and RH-6467 in Pecans

Commodity: Pecans

E. Analytical procedure

2. Extraction

ACL safety policy precludes overnight Soxhlet extractions.

4. Gas chromatography

a. Preparation of standard curves

This section states that the response of the analytes "are usually quadratic in nature." If instrument response is not linear, standard and sample response must be carefully matched if a standard curve is not used for quantitation.

5. Method of calculation

b. Component residue concentration

Average recovery values are used for calculating residue levels. This practice should not be incorporated in a tolerance enforcement method.

Additional reviewer's comments

1. The registrant should be asked if the soxhlet extraction can be shortened.

2. Recovery data and chromatograms are provided for samples fortified at less than the requested levels.

3. ILV recovery data and chromatograms are included for pecans fortified at 1/2X and 1X the requested tolerance.

ANALYTICAL CHEMISTRY BRANCH
SCREEN FOR RESIDUE METHODS FOR TMV

A. LABORATORY ASSIGNMENT NUMBER: B94-90,91

B. PP#: 11-3995

C. TECHNICAL REVIEWER: _____

D. DATE: 4-8-94

E. ANALYTES/LEVEL: RE-7592, RH-9129, RH-9130 / 0.05, 0.10 ppm

F. COMMODITIES: Pecans

G. METHOD: Residue Analytical Method for Parent RH-7592 and its Metabolites RH-9129, RH-9130, and RH-6467 in Pecans

The Analytical Chemistry Section has been asked to screen the residue chemistry methods submitted by the registrant in order to determine if they contain the essential requirements identified in the Residue Chemistry Guidelines. Full scientific review and laboratory evaluation of those methods will take place after the initial screen. The following items need to be resolved before the analytical method can be evaluated.

| | YES | NO |
|---|---|---|
| 1. Does the method use exotic equipment and/or supplies that are not commercially available in the U.S.? | ☐ | ☒ |
| 2. Does the method require any new equipment before the laboratory work begins? | ☐ | ☒ |
| 3. Are chromatograms included? | ☒ | ☐ |
| a. Is (are) peak(s) of interest sufficiently resolved from other peaks? | ☒ | ☐ |
| b. Has registrant included chromatograms of analyses at or below tolerance on all crop types for which tolerance is requested by HED? | ☒ | ☐ |
| c. Do the control samples have reasonably low levels of the analyte in relation to the proposed tolerance? | ☒ | ☐ |
| d. Is the method sufficiently sensitive and specific to measure and identify the residues at levels specified by HED in the TMV request? | ☒ | ☐ |

FIGURE 1: RH-7592 Standard Curve and Regression Data.

## B94-40,41 FENBUCONAZOLE TMV

Compound: RH-7592
Instrument: GC/NPD
Date: 7/29/94

**Linearity on Rtx-35 Column**

| ug/mL | pk ht. 1 | pk ht. 2 | pk ht. 3 | ave pk ht. | calc pk ht. |
|---|---|---|---|---|---|
| 0.126 | 1125 | 1191 | 1068 | 1128 | |
| 0.251 | 2506 | 2671 | 2014 | 2397 | |
| 0.502 | 6258 | 5819 | 5917 | 5998 | |
| 0.753 | 9006 | 8188 | 9005 | 8733 | |
| 1.00 | 12122 | 14006 | 14034 | 13387 | |
| 0.00 | | | | | -925 |
| 1.20 | | | | | 15611 |

**Regression Output:**

| | | |
|---|---|---|
| Constant | | -925.09044 |
| Std Err of Y Est | | 553.1986 |
| R Squared | | 0.9906523 |
| No. of Observations | | 5 |
| Degrees of Freedom | | 3 |
| X Coefficient(s) | 13779.8071 | |
| Std Err of Coef. | 772.812802 | |

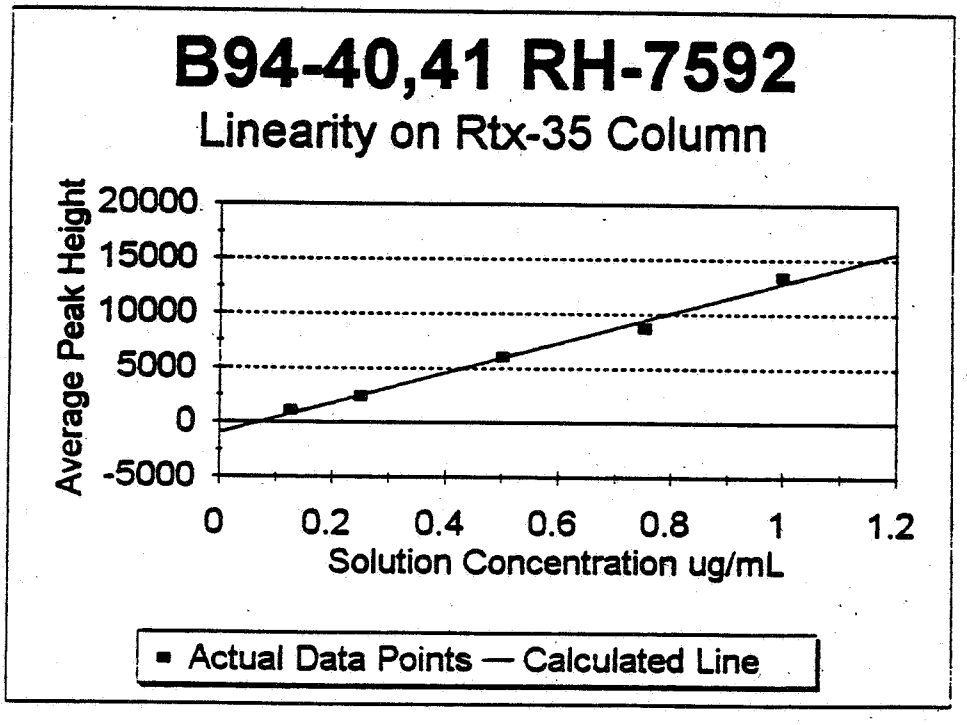


FIGURE 2: RH-9130 Standard Curve and Regression Data.

## B94-40,41 FENBUCONAZOLE TMV

Compound: RH-9130
Instrument: GC/NPD
Date: 7/29/94

**Linearity on Rtx-35 Column**

| ug/mL | pk ht. 1 | pk ht. 2 | pk ht. 3 | ave pk ht. | calc pk ht. |
|---|---|---|---|---|---|
| 0.126 | 593 | 630 | 550 | 591 | |
| 0.251 | 1253 | 1398 | 1004 | 1218 | |
| 0.502 | 3212 | 3025 | 3038 | 3092 | |
| 0.753 | 4546 | 4159 | 4514 | 4406 | |
| 1.00 | 5903 | 7113 | 7023 | 6680 | |
| 0.00 | | | | | -413 |
| 1.20 | | | | | 7818 |

**Regression Output:**

| | | |
|---|---|---|
| Constant | | -413.11795 |
| Std Err of Y Est | | 261.988353 |
| R Squared | | 0.99153025 |
| No. of Observations | | 5 |
| Degrees of Freedom | | 3 |
| X Coefficient(s) | 6858.886678 | |
| Std Err of Coef. | 365.9950569 | |

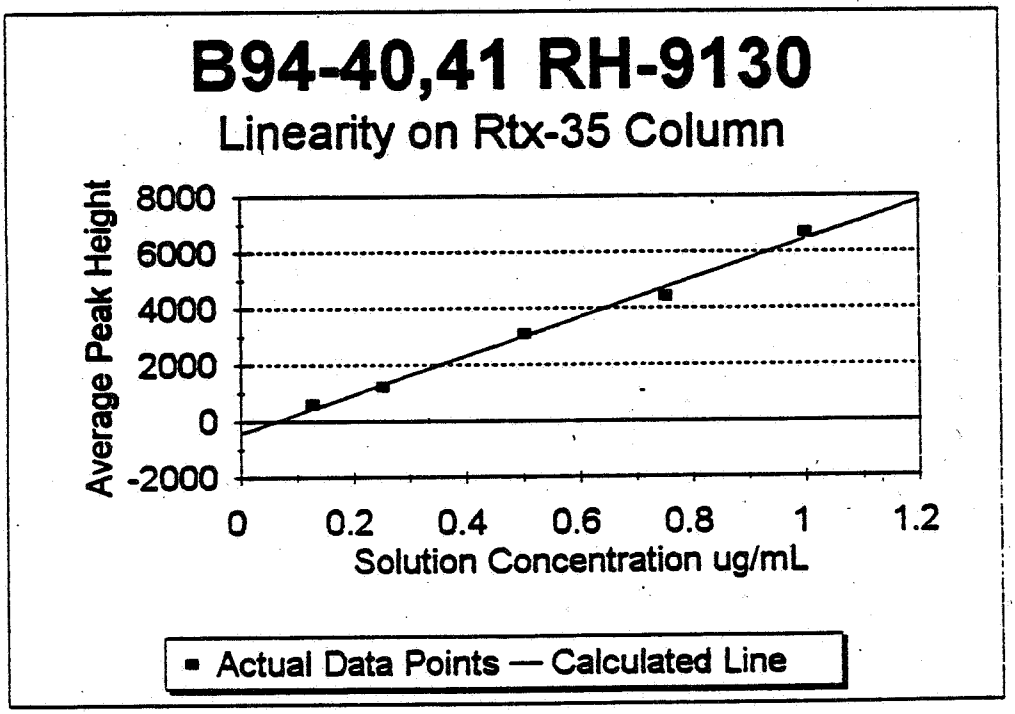


FIGURE 3: RH-9129 Standard Curve and Regression Data.

## B94-40,41 FENBUCONAZOLE TMV

Compound: RH-9129
Instrument: GC/NPD
Date: 7/29/94

**Linearity on Rtx-35 Column**

| ug/mL | pk ht. 1 | pk ht. 2 | pk ht. 3 | ave pk ht. | calc pk ht. |
|---|---|---|---|---|---|
| 0.126 | 545 | 570 | 519 | 545 | |
| 0.251 | 1179 | 1271 | 919 | 1123 | |
| 0.502 | 2955 | 2724 | 2773 | 2817 | |
| 0.753 | 4119 | 3742 | 4097 | 3986 | |
| 1.00 | 5425 | 6447 | 6407 | 6093 | |
| 0.00 | | | | | -367 |
| 1.20 | | | | | 7110 |

**Regression Output:**

| | |
|---|---|
| Constant | -367.0429 |
| Std Err of Y Est | 253.00767 |
| R Squared | 0.9904385 |
| No. of Observations | 5 |
| Degrees of Freedom | 3 |
| X Coefficient(s) | 6230.7045 |
| Std Err of Coef. | 353.44914 |

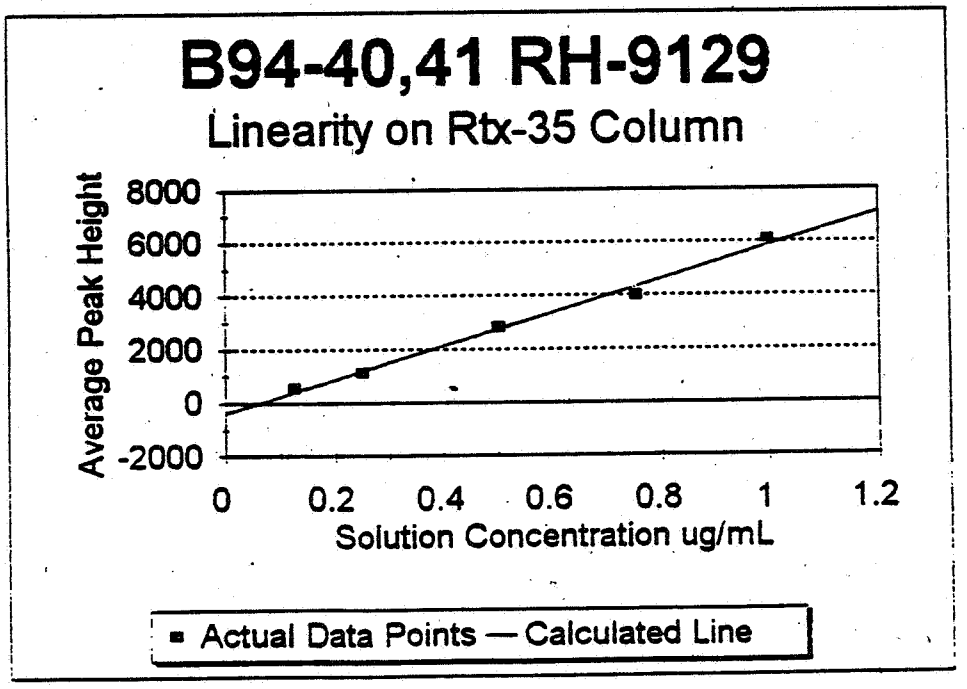


FIGURE 4: Sample Spreadsheet with Calculation Equations.

## B94-40,41 FENBUCONAZOLE TMV

Instrument: GC/NPD
Date: 8/31/94 injection of 8/10/94 samples

**RH-7592**

| Sample ID | Peak Height | Peak Height | Average Peak Ht | Std. Conc (ug/mL) | ppm Found | ppm Added | % Recovery |
|---|---|---|---|---|---|---|---|
| Std | 4902 | 4670 | 4569 | 0.252 | | | |
| C-1(10) | | | 0 | | 0.000 | 0.00 | |
| C-2(10) | | | 0 | | 0.000 | 0.00 | |
| Std | 4654 | 4049 | 4454 | 0.252 | | | |
| .05-1(10) | 4302 | 5275 | 4789 | | 0.054 | 0.05 | 108 |
| .05-2(10) | 6384 | 5490 | 5937 | | 0.067 | 0.05 | 134 |
| Std | 4600 | 4511 | | 0.252 | | | |
| Std | 9974 | 9612 | 10323 | 0.502 | | | |
| 0.1-1(10) | 11018 | 9522 | 10270 | | 0.100 | 0.10 | 100 |
| 0.1-2(10) | 11862 | 10616 | 11239 | | 0.109 | 0.10 | 109 |
| Std | 11509 | 10196 | | 0.502 | | | |

**RH-9130**

| Sample ID | Peak Height | Peak Height | Average Peak Ht | Std. Conc (ug/mL) | ppm Found | ppm Added | % Recovery |
|---|---|---|---|---|---|---|---|
| Std | 2900 | 2749 | 2713 | 0.252 | | | |
| C-1(10) | 25 | | 25 | | 0.000 | 0.00 | |
| C-2(10) | | | 0 | | 0.000 | 0.00 | |
| Std | 2749 | 2452 | 2663 | 0.252 | | | |
| .05-1(10) | 2527 | 3115 | 2821 | | 0.053 | 0.05 | 106 |
| .05-2(10) | 3749 | 3158 | 3454 | | 0.065 | 0.05 | 130 |
| Std | 2738 | 2713 | | 0.252 | | | |
| Std | 5842 | 5534 | 5946 | 0.502 | | | |
| 0.1-1(10) | 6488 | 5524 | 6006 | | 0.101 | 0.10 | 101 |
| 0.1-2(10) | 6613 | 5951 | 6282 | | 0.106 | 0.10 | 106 |
| Std | 6510 | 5897 | | 0.502 | | | |

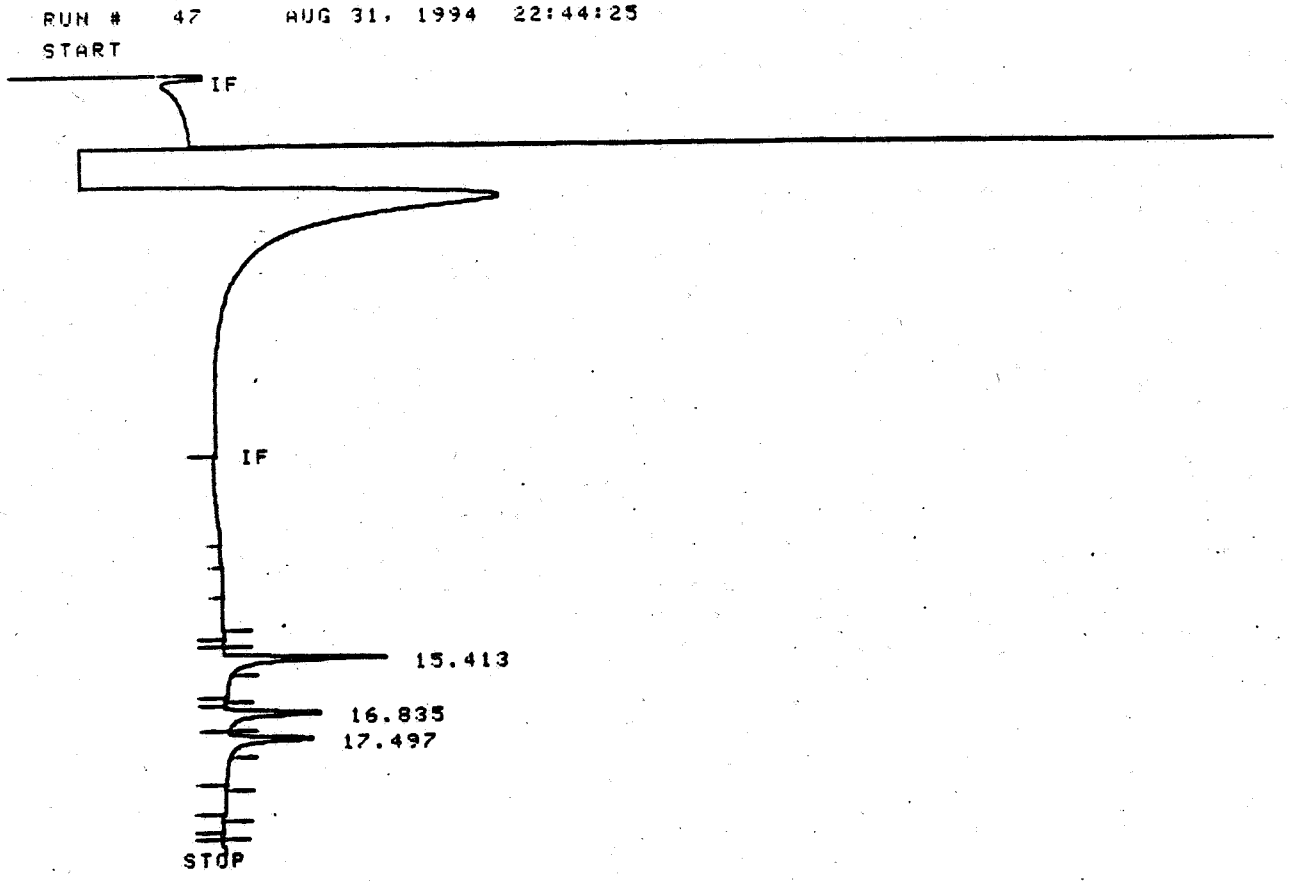
**RH-9129**

| Sample ID | Peak Height | Peak Height | Average Peak Ht | Std. Conc (ug/mL) | ppm Found | ppm Added | % Recovery |
|---|---|---|---|---|---|---|---|
| Std | 2576 | 2438 | 2405 | 0.252 | | | |
| C-1(10) | | | 0 | | 0.000 | 0.00 | |
| C-2(10) | | | 0 | | 0.000 | 0.00 | |
| Std | 2428 | 2176 | 2361 | 0.252 | | | |
| .05-1(10) | 2172 | 2587 | 2380 | | 0.051 | 0.05 | 102 |
| .05-2(10) | 3230 | 2736 | 2983 | | 0.064 | 0.05 | 128 |
| Std | 2437 | 2402 | | 0.252 | | | |
| Std | 5133 | 4835 | 5186 | 0.502 | | | |
| 0.1-1(10) | 5565 | 4644 | 5105 | | 0.099 | 0.10 | 99 |
| 0.1-2(10) | 5533 | 4977 | 5255 | | 0.102 | 0.10 | 102 |
| Std | 5651 | 5124 | | 0.502 | | | |

$$\text{ppm found} = \frac{(\text{ug/mL std}) \times (\text{Average Height Sample}) \times (\text{Final Volume } \{5\text{mL}\})}{(\text{Average Height Std}) \times (\text{Sample Weight } \{25\text{g}\})}$$

$$\%\ \text{Recovery} = \frac{\text{ppm Found} \times 100}{\text{ppm Added}}$$

**CHROMATOGRAM 1: Mixed Fenbuconazole Standard** - 0.252 ug/mL each RH-7592, RH-9130, RH-9129.(0.05 ppm equivalents for a 5 mL final volume of a 25 g pecan sample).




Closing signal file B:Q8F09A4A.BNC

RUN# 47 AUG 31, 1994 22:44:25

SAMPLE NAME: CALSTDA SAMPLE# 66
3uL INJ, 0.252ug/mL MIXED FENBUCONAZOLE STD IN 100/3

SIGNAL FILE: B:Q8F09A4A.BNC

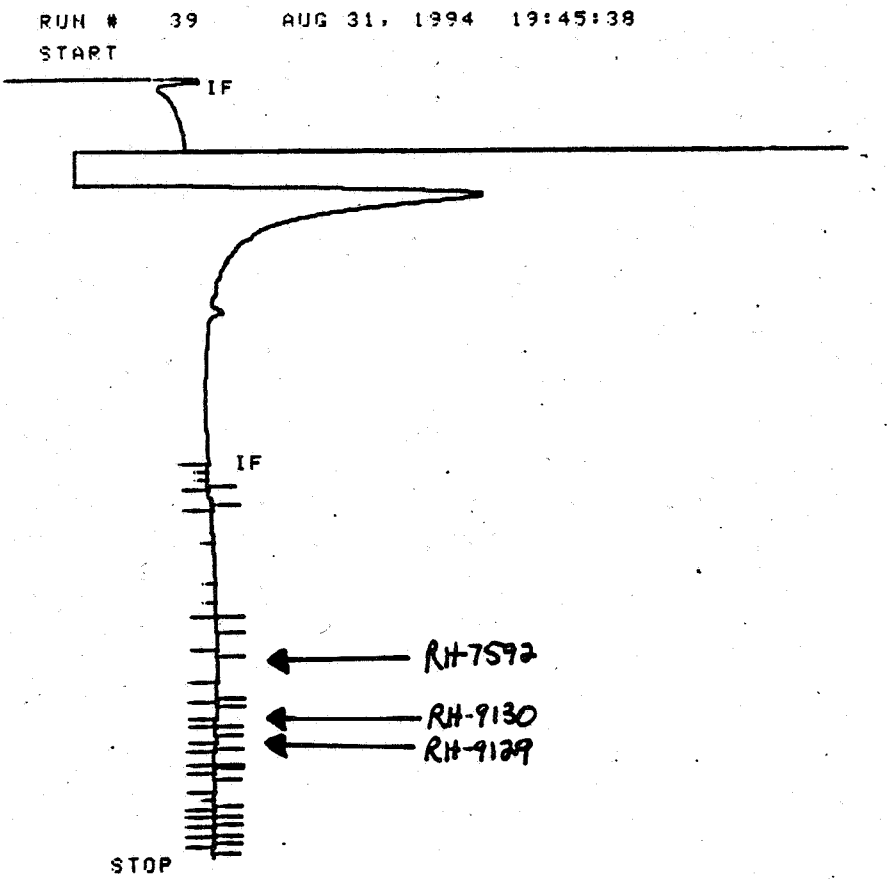
894-40,41 FENBUCONAZOLE TMV

HEIGHT%

| RT | HEIGHT | TYPE | WIDTH | HEIGHT% | |
|---|---|---|---|---|---|
| 15.413 | 4511 | PB | .132 | 46.86266 | RH-7592 |
| 16.835 | 2713 | PB | .147 | 28.18488 | RH-9130 |
| 17.497 | 2402 | BB | .156 | 24.95326 | RH-9129 |

TOTAL HEIGHT= 9626
MUL FACTOR=1.0000E+00

**CHROMATOGRAM 2: Pecan Control - 25g/5mL final volume.**




Closing signal file B:Q8F07063.8NC

RUN# 39 AUG 31, 1994 19:45:38

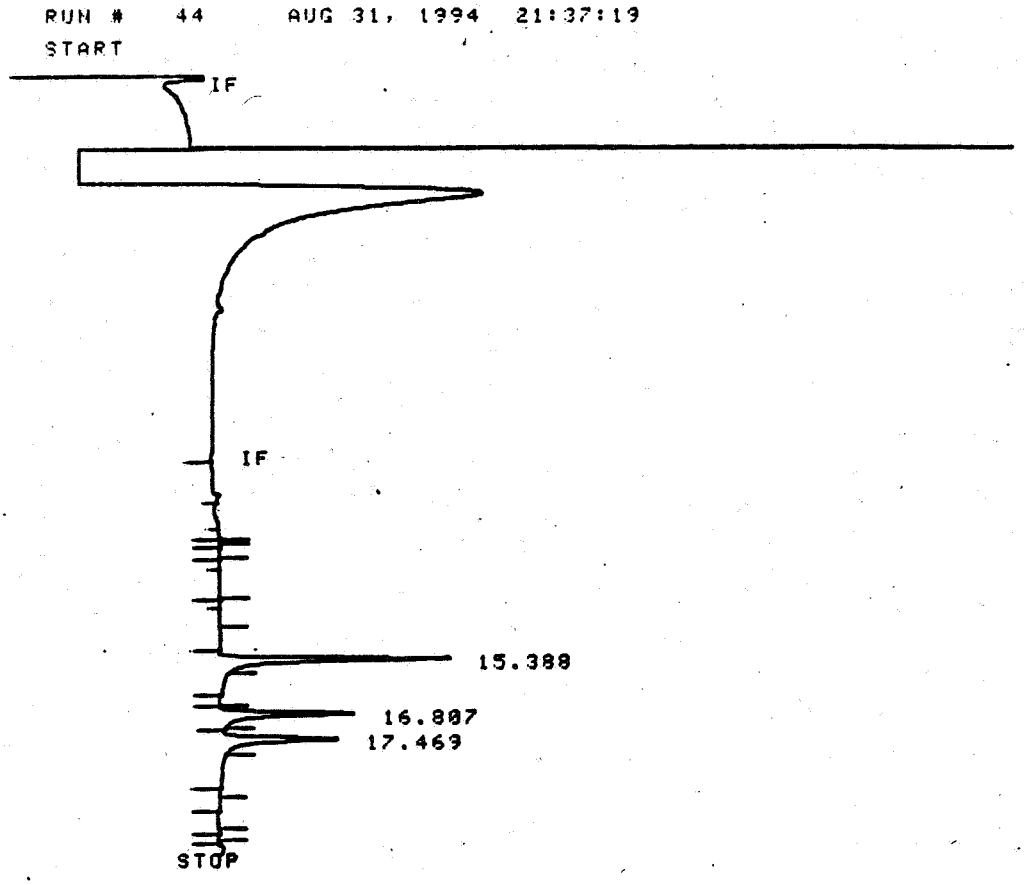
SAMPLE NAME: C-2(10) SAMPLE# 62
3uL INJ, PECAN CONTROL, 25g/5mL FINAL VOL IN 100/3

SIGNAL FILE: B:Q8F07063.8NC

894-40,41 FENBUCONAZOLE TMV

NO RUN PEAKS STORED

**CHROMATOGRAM 3: Pecan Fortification, 0.05 ppm each RH-7592, RH-9130, RH-9129** - 25g/5mL final volume.




Closing signal file B:Q8F08A90.BNC

RUN# 44 AUG 31, 1994 21:37:19

SAMPLE NAME: .05-2(10) SAMPLE# 65
3uL INJ, PECAN SPIKE, 25g + 1.26ug MIXED STD/5mL FINAL VOL IN 100/3

SIGNAL FILE: B:Q8F08A90.BNC

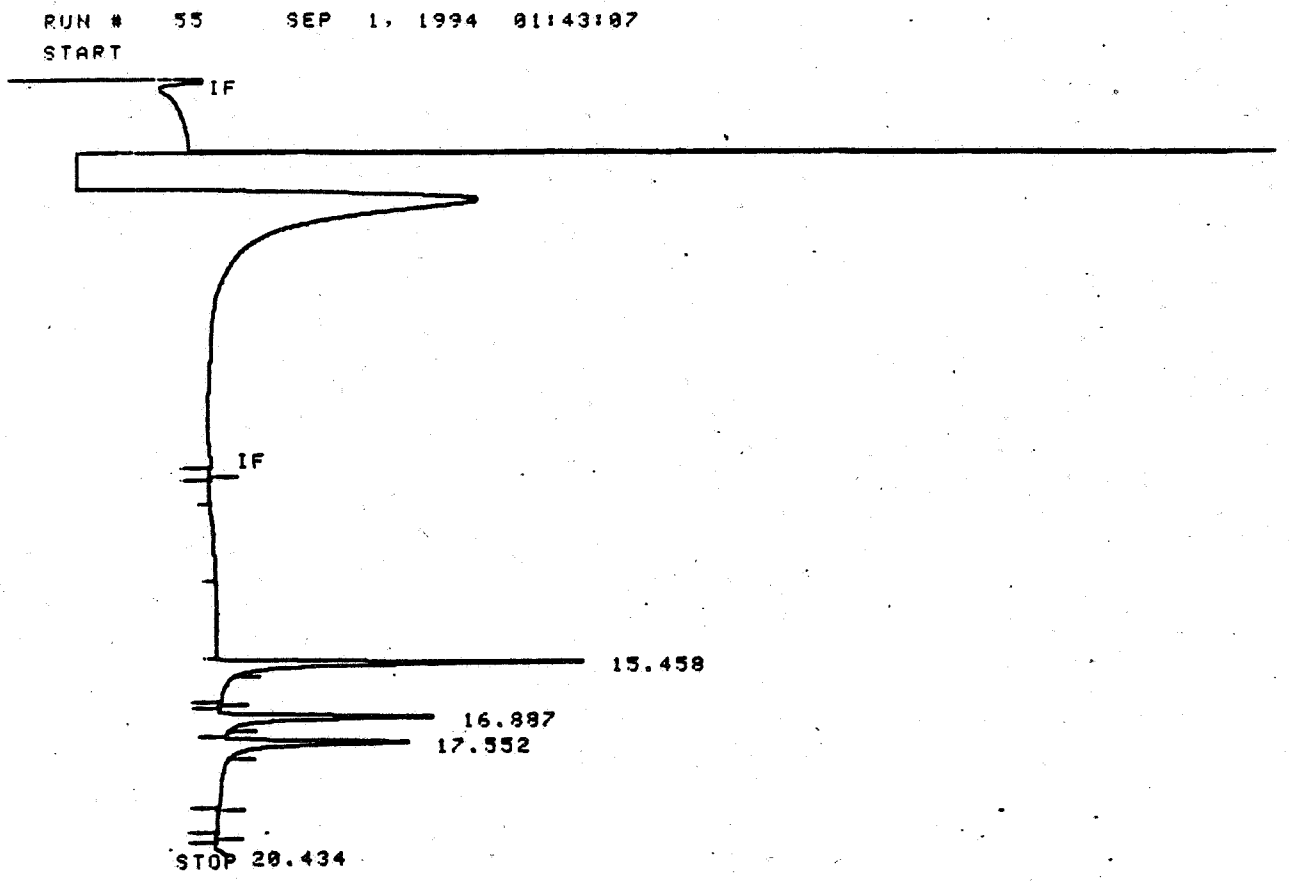
B94-40,41 FENBUCONAZOLE TMV

HEIGHT%

| RT | HEIGHT | TYPE | WIDTH | HEIGHT% | |
|---|---|---|---|---|---|
| 15.388 | 6384 | PB | .121 | 47.77370 | RH-7592 |
| 16.807 | 3749 | BB | .133 | 28.05509 | RH-9130 |
| 17.469 | 3230 | PB | .146 | 24.17122 | RH-9129 |

TOTAL HEIGHT= 13363
MUL FACTOR=1.0000E+00

**CHROMATOGRAM 4: Mixed Fenbuconazole Standard** - 0.502 ug/mL each RH-7592, RH-9130, RH-9129.(0.1 ppm equivalents for a 5 mL final volume of a 25 g pecan sample).




Closing signal file B:Q8F0C42C.BNC

RUN# 55 SEP 1, 1994 01:43:07

SAMPLE NAME: CALSTDB SAMPLE# 70
3uL INJ. 0.502ug/mL MIXED FENBUCONAZOLE STD IN 100/3

SIGNAL FILE: B:Q8F0C42C.BNC

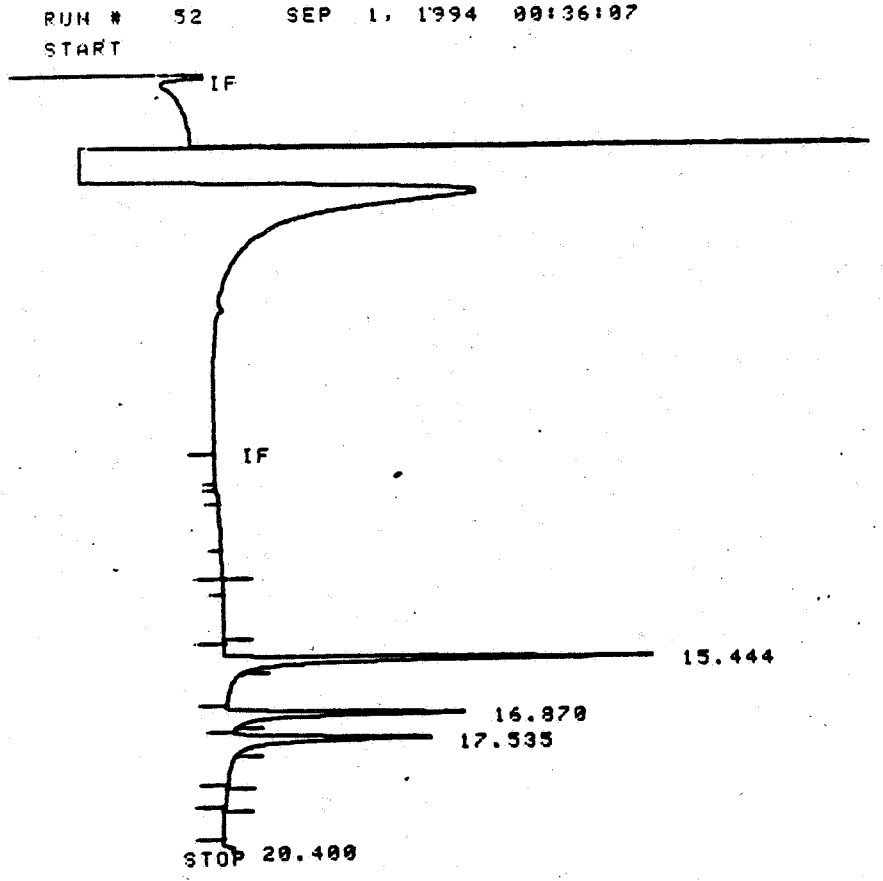
894-40,41 FENBUCONAZOLE TMV

HEIGHT%

| RT | HEIGHT | TYPE | WIDTH | HEIGHT% | |
|---|---|---|---|---|---|
| 15.458 | 10196 | PB | .122 | 47.44974 | RH-7592 |
| 16.887 | 5897 | PB | .135 | 27.44322 | RH-9130 |
| 17.552 | 5124 | BB | .147 | 23.84586 | RH-9129 |
| 20.434 | 271 | I PH | .127 | 1.26117 | |

TOTAL HEIGHT= 21488
MUL FACTOR=1.0000E+00

**CHROMATOGRAM 5: Pecan Fortification, 0.1 ppm each RH-7592, RH-9130, RH-9129** - 25g/5mL final volume.




Closing signal file B:Q8F08478.BNC

RUN# 52 SEP 1, 1994 00:36:07

SAMPLE NAME: 0.1-2(10) SAMPLE# 69
3uL INJ, PECAN SPIKE, 25G + 2.5ug MIXED STD/5mL FINAL VOL IN 100/3

SIGNAL FILE: B:Q8F08478.BNC

894-40.41 FENBUCONAZOLE TMV

HEIGHT%

| RT | HEIGHT | TYPE | WIDTH | HEIGHT% | |
|---|---|---|---|---|---|
| 15.444 | 11862 | PB | .118 | 48.68859 | RH-7592 |
| 16.870 | 6613 | BB | .138 | 27.13916 | RH-9130 |
| 17.535 | 5533 | BB | .149 | 22.70694 | RH-9129 |
| 20.400 | 359 | I PH | .144 | 1.47330 | |

TOTAL HEIGHT= 24367
MUL FACTOR=1.0000E+00