US EPA ARCHIVE DOCUMENT

SR

434924-01

STUDY TITLE

Revised Residue Analytical Method for Parent RH-7592 and Its Metabolites RH-9129, RH-9130, and RH-6467 in Pecans

Replaces Rohm and Haas Co. Report Numbers
34-91-14, (MRID 41892503) Submitted: 5/15/91
34-94-161, (MRID 43450001) Submitted: 11/14/93

DATA REQUIREMENT

Guideline 171-4

AUTHOR

John J. Martin

STUDY COMPLETED ON

December 19, 1994

PERFORMING LABORATORY

Rohm and Haas Company
727 Norristown Rd.
Spring House, PA 19477

LABORATORY PROJECT ID

34-94-175

REPORT NO 34-94-175

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA 10(d)(1)(A), (B), or (C).

COMPANY: Rohm and Haas Company

COMPANY AGENT: Richard D. Costlow DATE: December 20, 1994

Richard D. Costlow Product Registration Manager

SIGNATURE

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TR 34-94-175

Technical Report No.: 34-94-161

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ACCURATE COPY OF THE ORIGINAL DATA
SIGNATURE [illegible] DATE 12/20/94

GLP Compliance Statement

As per 40 CFR 160.3 method development is not required to be conducted in compliance with GLP. However the work was conducted in the spirit of Good Laboratory Practices as defined by the United States Environmental Protection Agency.

11/9/94

S. Wu — Date
Revisionist/Study Director
Rohm & Haas Company

Nov 9/94

S. S. Stavinski — Date
Applicant
Rohm & Haas Company

Nov 9/94

S. S. Stavinski — Date
Sponsor
Rohm & Haas Company

Quality Assurance Statement

This report and the original raw date have been reviewed by the Quality Assurance Unit of the Rohm and Haas Company Agricultural Research Division and has been validated as a true and accurate representation of the data collected.

11/9/94

R. S. Krick — Date
QA Analyst
Rohm & Haas Company

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Technical Report
No. 34-94-161

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REPORT REVISION

This method is a revision of residue analytical method No. TR-34-91-14 (MRID#41892503) which has recently passed EPA validation. The revision is in response to EPA's recommendations for our previous petition for permanent tolerance for Fenbuconazole on pecans (Petion No. 1F3995). The following changes have been made according to their suggestions:

1. The Soxhlet extraction time has been shortened to 6-8 hr according to EPA's Analytical Chemistry Laboratory safety policy which precludes overnight soxhlet extraction.

2. A statement on preparation of standard curves that the response of the analytes "are usually quadratic in nature" has been rewritten as " If instrument response is not linear, standard and sample response must be carefully matched if a standard curve is not used for quantitation".

3. The average recovery values have been removed from calculating residue level according to their recommendation.

REPORT CERTIFICATION

This revision to the original report, TR-34-91-12 (MRID#41892503), is a true and accurate representation of the study as it was conducted.

11/9/94

S. Wu Date
Revisionist/Study Director
Rohm and Haas Company

Quality Assurance Statement

This revised report has been reviewed by the Qulity Assurance Unit of the Rohm and Haas Agricultural Products Development Research Department.

11/9/94

Rhonda Krick Date
Rohm and Haas Company

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REPORT REVISION II

In addition to the first revision (TR-34-94-161), a factor of 100 in equation (2) on page 15 was mistakenly retained when the equation was modified by removing the Average Recovery (%) from the denominator. This mistake has been corrected.

REPORT CERTIFICATION

This revision to the revised report, TR-34-94-161 (MRID No. 43450001), is a true and accurate representation of the study as it was conducted.

12/19/94

S. Wu — Date
Revisionist/Study Director
Rohm and Haas Company

Quality Assurance Statement

This revised report has been reviewed by the Quality Assurance Unit of the Rohm and Haas Agricultural Products Development Research Department.

Rhonda S. Krick 12/20/94

Rhonda Krick — Date
Rohm and Haas Company

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I. Summary

The RH-7592 residues (parent and the three metabolites) are extracted from finely chopped pecans by Soxhlet extraction with hexane/2-propanol. The extract is concentrated to an oily residue. The residue is dissolved in toluene/acetone and c aned up on silica gel and then Florisil. The samp'es are further cleaned up with a C-18 solid phase extraction cartridge.

RH-7592 and the metabolites are determined by gas chromatography using a 30 meter, 0.53 mm ID capillary column (RTx-35) and a Capillary Thermionic Specific Detector optimized for nitrogen selectivity. A flow diagram of the method is shown on page 7.

The sensitivity of the method is 0.010 ppm for all analytes. The average recovery for RH-7592 is 91%, for RH-9130 94%, for RH-9129 86% and for RH-6467 99%

II. Introduction

RH-7592 is an experimental triazole fungicide being developed for use on stonefruit, wheat, pecans, almonds and other crops. In order to obtain commercial registration an analytical method is needed to determine the magnitude of the total toxic residues from the proposed uses of RH-7592 and to provide a means of obtaining data for the setting and enforcement of tolerances for residues in food and feed.

The extraction efficiency is very good. Samples fortified with radiolabeled RH-7592 and metabolites show greater than 90% recovery. Field studies conducted with radiolabeled RH-7592 on peaches and wheat have shown that the residues of concern consist mainly of parent RH-7592 and lesser amounts of the lactone metabolite RH-9129 and the benzylic-ketone metabolite RH-6467. Because the lactone metabolite can exist in two diasteriomeric forms, the second diasteriomer RH-9130 was also included in the development of a residue analytical method for pecans. The structures of these compounds are shown below.

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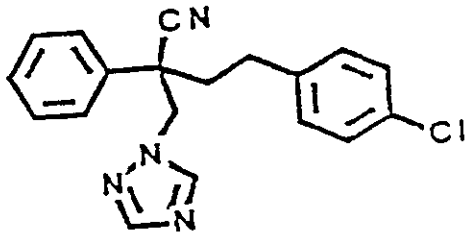
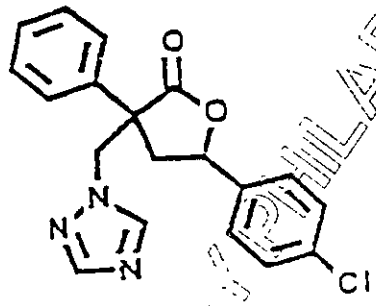
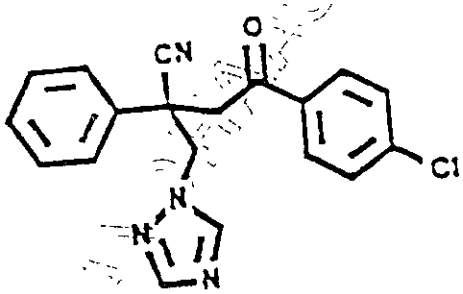


RH-7592

α-(2-[4-chlorophenyl]ethyl)-α-phenyl-3-(1H-1,2,4-triazole)-1-propanenitrile



Lactones A & B
(RH-9129 and RH-9130)
5-(4-chlorophenyl)dihydro-3-phenyl-3-(methyl-1-H-1,2,4-triazole-1-yl)-2-(3H)-furanone



Ketone (RH-6467)

4-(4-chlorophenyl)-2-(methyl-1H-1,2,4-triazole)-4-oxo-2-phenyl butanenitrile

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FLOW DIAGRAM

Soxhlet Extraction with hexanehexane/isopropyl Alcohol

⇓

Silica Gel Column chromatography

⇓

Florisil Column Chromatopraphy

⇓

C-18 SPE Cartridge Cleanup

⇓

Gas Liquid Chromatgraphy

⇓

Quantitation

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III. Method

A. Chemicals/Supplies

Acetone, Pesticide Grade	Fisher
Silica gel,40-140 mesh	Baker
(Activate at 200°C for 24 hours.	
Bottle and cap immediately.	
Store in a desiccator cabinet	
Standardize before use, Section IV).	
Boileezers	Fisher
Cotton, Sterile, Absorbent	Johnson & Johnson
Florisil 60-100 Mesh	U S. Silica Co.
(Activate at 200°C for 24 hours.	
Bottle and cap immediately.	
Store in a desiccator cabinet	
Standardize before use, Section IV).	
Hexane	Baker
LC-18 Supelclean Tubes, 6 ml, 1 g	Supelco
Methanol, Pesticide Grade	Baker
2-Propanol HPLC Grade	Baker
Sodium Sulfate, Anhydrous, Granular AR	Mallinckrodt
Thimbles, Soxhlet, Cellulose, 43 x 123 mm	Whatman
Toluene, Pesticide Grade	Baker
RH-7592 Analytical Standard	Rohm and Haas Co.
RH-9129 Lactone Analytical Standard	Rohm and Haas Co
RH-9130 Lactone Analytical Standard	Rohm and Haas Co.
RH-6467 Analytical Standard	Rohm and Haas Co.
Water Milli-Q	Millipore

B. Preparation of Solutions

1 Prepare 100/30, 100/10 and 100/5 (v/v) toluene/acetone solutions by adding 300, 100 and 50 ml of acetone to 1000 ml of toluene

2. Prepare 100/3 (v/v) toluene/methanol solution by adding 30 ml of methanol to 1000 ml of toluene

3. Prepare 2/1 and 4/5 methanol/water solutions by adding 200 ml of methanol to 100 ml of Milli-Q water and 40 ml of methanol to 50 ml of Milli-Q water.

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4. Prepare 3/1 (v/v) hexane/2-propanol solution by adding 500 ml of 2-propanol to 1500 ml of hexane.

5 Prepare GLC standard and fortification solutions by carefully weighing on an analytical balance 0.100 g, corrected for purity, of each of the analytical standards (RH-7592, RH-9129, RH-9130 and RH-6467) into individual 100 ml volumetric flasks. Add approximately 80 ml of the toluene/methanol (100/3) solution and sonicate until dissolution occurs. Bring to volume with toluene/methanol (100/3). These are the primary standards of 1000 µg/ml. Into a 100 ml volumetric flask carefully pipet 10 ml of each of the primary standards and bring to volume with toluene/methanol (100/3) This is the primary multi-component standard of 100 µg/ml. Make serial dilutions of the multi-component standard to 10, 5, 1, 0.5, 0.1 and 0.05 µg/ml for working standards.

C. Equipment

Chromatographic Columns, 19 mm ID x 30 cm length with 250 ml integral reservoir at the top, teflon stopcock (Code 5907-T-15)	Ace Glass, Inc.
Food Processor, Commercial	Waring
Linear Polyethylene LSC vials	DuPont
Rotary Evaporator, Buchi Rotovap R with dry ice trap	Brinkman
Round bottom flasks, 500, 300, 200 ml	Kimax
Solid Phase Extraction Vacuum Manifold with 20 ml reservoirs and adaptors (Visiprep)	Supelco
Soxhlet Extraction Apparatus, 55/50 joint, size 23	Kontes
Standard laboratory equipment, balances, beakers, etc.	
Ultrasonic Cleaner	Branson
Volumetric flasks, 100 ml	Kimble

D. Instrumentation

Varian 3500 Capillary Gas Chromatograph equipped with a Varian Model 8100 Autosampler, a 1040 Megabore Injector and a Capillary Thermionic Specific Detector Data are obtained with an HP 300 Data Acquisition and Processing Station with Hewlett-Packard Extrachrom Software Data are processed by Nelson Analytical Software

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Column Fused silica capillary, RTx-35 0 53 mm ID, 30 meters 0.25 um df – Restek Corporation.

Temperatures:	Column	-	5 min at 220°C then to 285°C at 10°C/min, hold for 9 min.
	Injector	-	275°C
	Detector	-	300°C

Flows: Air (zero grade) - 175 ml/min
Hydrogen (UPC) - 4.5 ml/min
Helium (UPC) - 2 ml/min, 10 ml/min purge

Bead Current: 3 15 amps (varies)

Under these conditions, the retention times are as follows:

RH-7592 - 15.8 minutes
RH-9129 - 17 3 minutes
RH-9130 - 18.0 minutes
RH-6467 - 19.1 minutes

E. Analytical Procedure

1. Sample Processing

Separate the nuts from the shell with a nut cracker and discard the shells. Add a small amount of dry ice and chop the nuts into small pieces in the Waring food processor. Thoroughly mix the finely chopped pecans and allow the dry ice to sublime in a freezer. Store the samples in the freezer (-10°C) until analysis

2. Extraction

Weigh 25 g of the finely chopped pecan nuts into Soxhlet thimbles. Add 275 ml of hexane/2-propanol(3/1[v/v]) solution and a boileezer granule to the 500 ml round bottom flask. Set up the Soxhlet extraction apparatus for 6-8 hr reflux. After the completion of extraction, cool the extracts and remove the boileezer granule with a pair of forceps. Concentrate the extract to an oily residue on the rotary evaporator at 65-70°C under gentle vacuum Be sure to avoid excessive foaming of the extracts out of the flask. Add 10 ml of hexane to the flask and concentrate the extract on the rotary evaporator again . Add 25 ml of the toluene/acetone (100/10) to the flask and swirl to dissolve the residue. Proceed to the Silica gel column cleanup step, Section 3.a.

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3. Cleanup

a. Silica Gel Column Chromatography

Insert a small cotton plug into a 19 mm ID chromatographic column and dry pack the column with 15 cc of the activated silica gel (measure in a 25 cc graduated cylinder). Top the column with about 1 inch of anhydrous sodium sulfate. Add the 25 ml of toluene/acetone (100/10) solution in the 500 ml round bottom flask from the extraction step to the column and collect the eluent in a 250 ml Erlenmeyer flask. Rinse the 500 ml round bottom flask with 10 ml of toluene/acetone (100/10) and add to the column when the previous addition just enters the top of the column. Add 25 ml of toluene/acetone (100/10) to the column when the previous addition just enters the top of the column and collect the eluent in the 250 ml Erlenmeyer flask. Discard the combined eluent.

Elute the column with 150 ml of toluene/acetone (100/30) and collect the eluent in a 500 ml round bottom flask. Concentrate the eluent to dryness on the rotary evaporator at 70°C under reduced pressure. Add 25 ml of toluene/acetone (100/5) to the flask and swirl to dissolve the residue and proceed to the Florisil column cleanup step. The elution pattern for each batch of silica gel should be checked before use (see Section IV). Proceed to the Florisil column chromatography step, Section 3 b

b. Florisil Column Chromatography

Insert a small cotton plug into a 19 mm ID chromatographic column and dry pack the column with 15 cc of the activated Florisil (measure in a 25 cc graduated cylinder). Top the column with 1 inch of anhydrous sodium sulfate. Add the 25 ml of the toluene/acetone (100/5) solution in the 500 ml round bottom flask to the column and collect the eluent in a 250 ml Erlenmeyer flask. Rinse the 500 ml round bottom flask with 10 ml of toluene/acetone (100/5) and add to the column when the previous addition just enters the top of the column. Add 25 ml of toluene/acetone (100/5) to the column in like manner. Discard the eluent.

Elute the column with 150 ml of toluene/acetone (100/30) and collect the eluent in a 300 ml round bottom flask and concentrate the eluent to dryness on the rotary evaporator at 70°C under reduced pressure. Be sure the samples are free of residual toluene. Use a gentle stream of nitrogen to remove traces of solvent. Add 4 ml of methanol to the flasks and swirl to dissolve the residue (sonicate briefly if necessary). The elution pattern for each batch of Florisil should be checked before use (see Section IV). Proceed to the C-18 solid phase extraction cleanup, Section 3 c.

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c. C-18 Solid Phase Extraction Cleanup

Insert the appropriate number of Supelclean LC-18 Solid Phase Extraction tubes (1 g) into the Solid Phase Extraction Manifold. Following the manufacturers instructions on using the manifold condition the tubes by passing 5 ml of methanol, followed by 5 ml of Milli-Q water through the tubes. Do not allow the tubes to dry. Insert the adaptors and 20 ml reservoirs in the top of the extraction tubes. To the 4 ml methanol samples in the 300 ml round bottom flask add 5 ml of Milli-Q water and swirl to mix well. Pour the methanol/water solution into the 20 ml reservoir. Wash the 300 ml round bottom flask with 5 ml of methanol/water (4/5) by swirling and add to the reservoir. Elute the sample to waste.

Place the collection rack containing 20 ml scintillation vials in the vacuum manifold chamber and elute the samples with 15 ml of methanol/water (2/1). Transfer the eluent from the 20 ml PE vials to 200 ml round bottom flasks. Wash the PE vials with a small volume of methanol and add it to the 200 ml flask. Add 50 ml of 2-propanol. Concentrate the samples to dryness on the rotary evaporator at 70°C under gentle vacuum. Add the appropriate volume, usually 5.0 ml, of toluene/methanol (100/3) and proceed to the gas chromatography step, Section 4.

4. Gas Chromatography

a. Preparation of Standard Curves

At least duplicate 3 ul aliquots of each multi-component standard in the range of 1.0 to 0.05 µg/ml is injected into the gas chromatograph. The resulting peak heights are measured and plotted vs. concentration (µg/ml) of RH-7592 and its metabolites to obtain 4 standard calibration curves, one for each analyte. Standard curves are prepared for each analysis day. If instrument response is not linear, standard and sample response must be carefully matched if a standard curve is not used for quantitation

b. Sample Analysis

A 3 µl aliquot of the final GLC sample is injected into the gas chromatograph. If necessary, the sample is diluted with toluene/methanol (100/3) to give a response within the standard curve range. The peak heights are measured and the concentration of each component is determined from the standard curves. The limit of quantitation was established at 0.01 ppm by analysis of fortifications at that level. By definition, under Rohm and Haas SOP, the limit of detection is 30% of the limit of quantitation, 0.003 ppm.

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5. Method of Calculation

The RH-7592, RH-9129, RH-9130 and RH-6467 residue concentration are determined as follows.

a Fortification Recovery

For samples fortified with known amounts of RH-7592, RH-9129, RH-9130 and RH-6467 prior to extraction, measure the peak heights, determine the concentration (µg/ml) from the standard curves and calculate the percent recovery from the equation 1.

Eq.1

$$\frac{(\mu g/ml\ Found) \times Final\ Sample\ Vol.(ml) - Ctl\ corr.}{Fortification\ (\mu g)} \times 100 = Recovery$$

b. Component Residue Concentration

The component residue concentration is determined as follows:

Eq.2

$$\frac{Final\ Sample\ Vol.\ (ml) \times Component\ Conc.\ (\mu g/ml)}{Sample\ Weight\ (g)} = ppm$$

IV. Results and Discussion

Typical chromatograms of standards, calibration curves, pecan control and fortified control samples are shown in Figures 1 to 8.

It is important that each lot of silica gel and Florisil be evaluated for consistency by running a multi-component standard through the elution scheme, concentrating the 150 ml of toluene/acetone (100/30) cut to dryness and determining the recovery by GLC. This should be greater than 85%.

The RTx-35 capillary column should be conditioned according to the manufacturers instructions before connecting it to the detector. It was also necessary to prime the column before analysis by making several injections of substrate or 1 or 2 ug/ml standard solution followed by two injections of toluene/methanol (100/3) solvent. If this is not

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done, standards injected at the beginning of analytical run give a lower detector response than standards injected at the end of a run.

The TSD bead should be conditioned and the bead current set following the manufacturers instructions. The bead current should then be adjusted to a setting that will give a detector response sufficient to reliably measure a standard of 0.05 ug/ml concentration. The bead current will need to be adjusted as the bead ages.

The extraction efficiency is very good. Samples fortified with radiolabeled RH-7592 and metabolites show greater than 90% recovery. The recovery data are summarized in Table I. The detailed recovery data are presented in Table II.

The 2-propanol is added to the eluent from the C-18 solid phase extraction cleanup to form an azeotropic solution with the water in the eluent. This helps to prevent bumping during the concentration step prior to analysis by gas chromatography.

A Material Safety Data Sheet for RH-7592 is included in Appendix A.

V. Project Information and Study Certificate Statement

Title: Residue Analytical Method for Parent RH-7592 and Its Metabolites RH-9129, RH-9130 and RH-6467 in Pecans.

Sponsor and Testing Facility:
Rohm and Haas Company
Research Laboratories
727 Norristown Road
Spring House, PA 19477

Dates:
Work Initiated: May, 1990
Work Completed: March, 1991

Study Personnel:
Study Director: John Martin
Scientist: Ni Ding
Technical Assistant Theodore F. Burnett

Notebook References:
JM-39 Rohm and Haas No. 053047
JM-40 Rohm and Haas No. 056720
ND-1 Rohm and Haas No. 056670
TB-6 Rohm and Haas No 054730

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Table I

Summary of Pecan Recovery Data

	Average(%)	Max	Min
RH-7592	91 +/- 17	122	49
RH-9130	94 +/- 19	123	55
RH-9129	86 +/- 20	122	51
RH-6467	99 +/- 24	153	48

n=21

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Table II

Recovery Data for RH-7592 and Metabolites in Pecans

SAMPLE No	RH7592	RH9130	RH9129	RH6467	FORTIFICATION LEVEL(PPM)
90-0175-003	89	90	77	95	0.04
90-0175-012	70	67	57	51	0 04
90-0229-004	72	73	64	85	0.04
90-0229-001	80	79	75	87	0 04
90-0229-007	102	100	93	111	0.04
90-0229-010	111	109	89	104	0.04
90-0250-016	86	93	79	101	0.04
90-0251-003	122	114	99	97	0.04
AVG (0.04PPM)	9 2	91	7 9	9 3	
STD DEV	1 9	17	1 4	1 5	
90-0251-003	84	75	67	71	0.02
90-0251-016	100	96	92	108	0.02
90-0234-001	100	109	108	127	0.02
90-0234-001	103	113	112	134	0.02
90-0175-006	108	106	76	108	0.02
90-0175-009	94	106	88	79	0.02
AVG (0.02PPM)	9 8	101	91	105	
STD DEV	8	14	1 8	2 5	
90-0229-01 0	95	92	82	90	0 01
90-0250-006	64	60	62	84	0.01
90-0250-016	100	116	120	125	0.01
90-0251-016	49	55	51	48	0.01
90-0251-003	104	93	86	94	0.01
90-0234-001	84	97	97	109	0.01
90 0234-001	99	123	122	153	0 01
AVG (0.01 PPM)	8 5	91	8 9	10 0	
STD DEV	21	26	27	33	
METHOD AVG	91	94	8 6	9 9	
STD DEV	1 7	1 9	2 0	2 4	

Data file: J131913 Type: STANDARD

Sample Name: PECAN Cal. Curve: 1/31/91

Date: 31 Jan 1991 13:56 Method: M7592m Operator: VD

Interface: 713 Cycle#: 3 Channel#: A

Instrument: GC-25-89

Column: RTX-35 Column Length: 30 Meters

Start Temp-Time (deg-min): 220 Ramp Hold (deg-min):

Program Rate (deg/min): 10 End Time-Temp (deg-min): 285

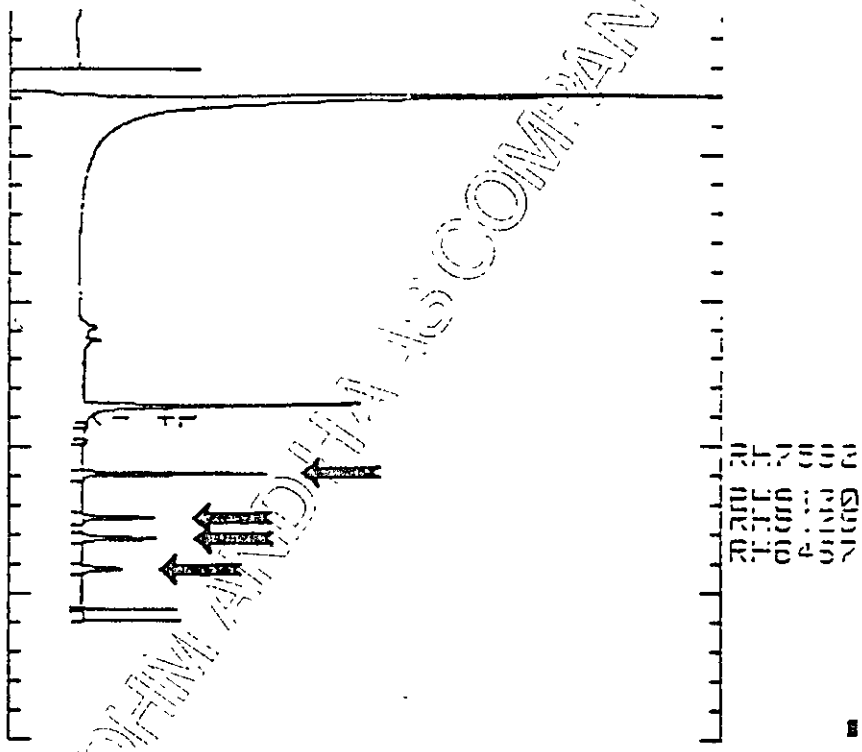
Prog Slope (# or Linear): Inj Port Temp: 275

Flowrate/Gas: 10 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 25 minutes

Plot range: 20 millivolts (-3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
15.91	RH7592	0.500	5.400E+00	4.940E+02
17.42	RH9130	0.500	2.520E+00	1.940E+02
18.12	RH9129	0.500	2.710E+00	1.990E+02
19.16	RH6467	0.500	1.670E+00	1.090E+02

Figure 1
0.5 ug/ml Standard Solution

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Data file: J131914 Type: STANDARD

Sample Name: PECAN Cal. Curve: 1/31/91

Date: 31 Jan 1991 14:20 Method: M75920m Operator: MD

Interface: 713 Cycle#: 4 Channel#: A

Instrument: GC-25-89

Column: RTX-35 Column Length: 30 Meters

Start Temp-Time (deg-min): 220 Ramp Hold (deg-min):

Program Rate (deg/min): 10 End Time-Temp (deg-min): 285

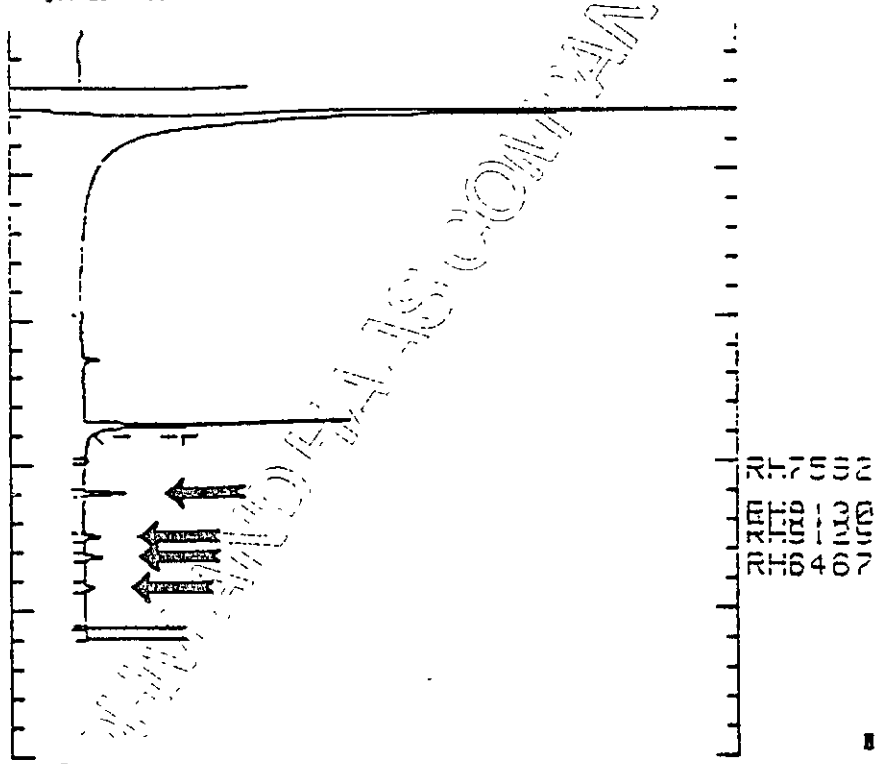
Prog Slope (# or Linear): Inj Port Temp: 275

Flowrate/Gas: 10 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 25 minutes

Plot range: 20 millivolts (-3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
15.92	RH7592	0.100	1.250E-00	1.130E+02
17.43	RH9130	0.100	5.790E-01	4.410E+01
18.13	RH9129	0.100	6.460E-01	4.670E+01
19.18	RH6467	0.100	3.780E-01	2.400E+01

Figure 2
0.1 ug/ml Standard Solution

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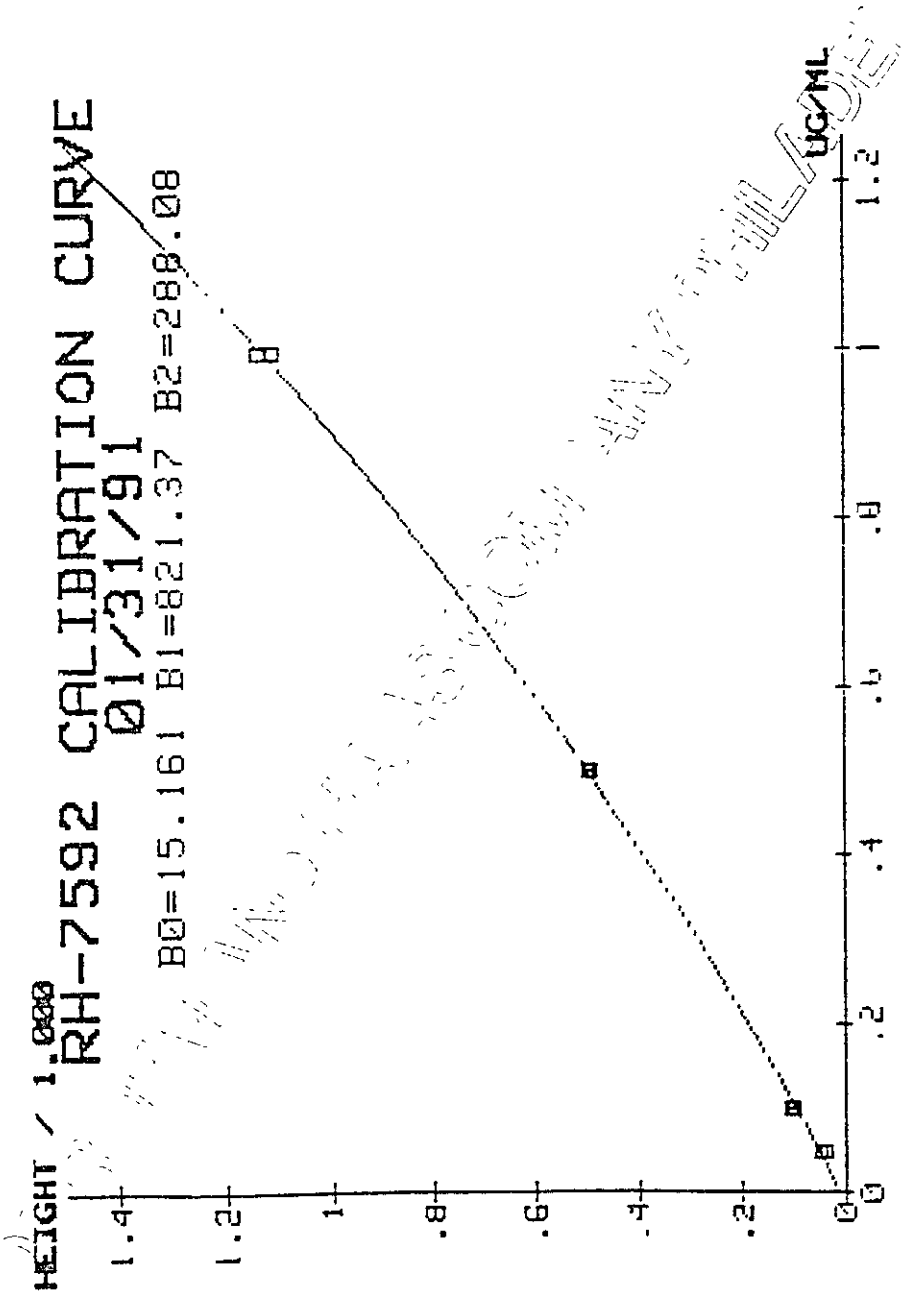

Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data

FIGURE 3
TYPICAL RH-7592 CALIBRATION CURVE

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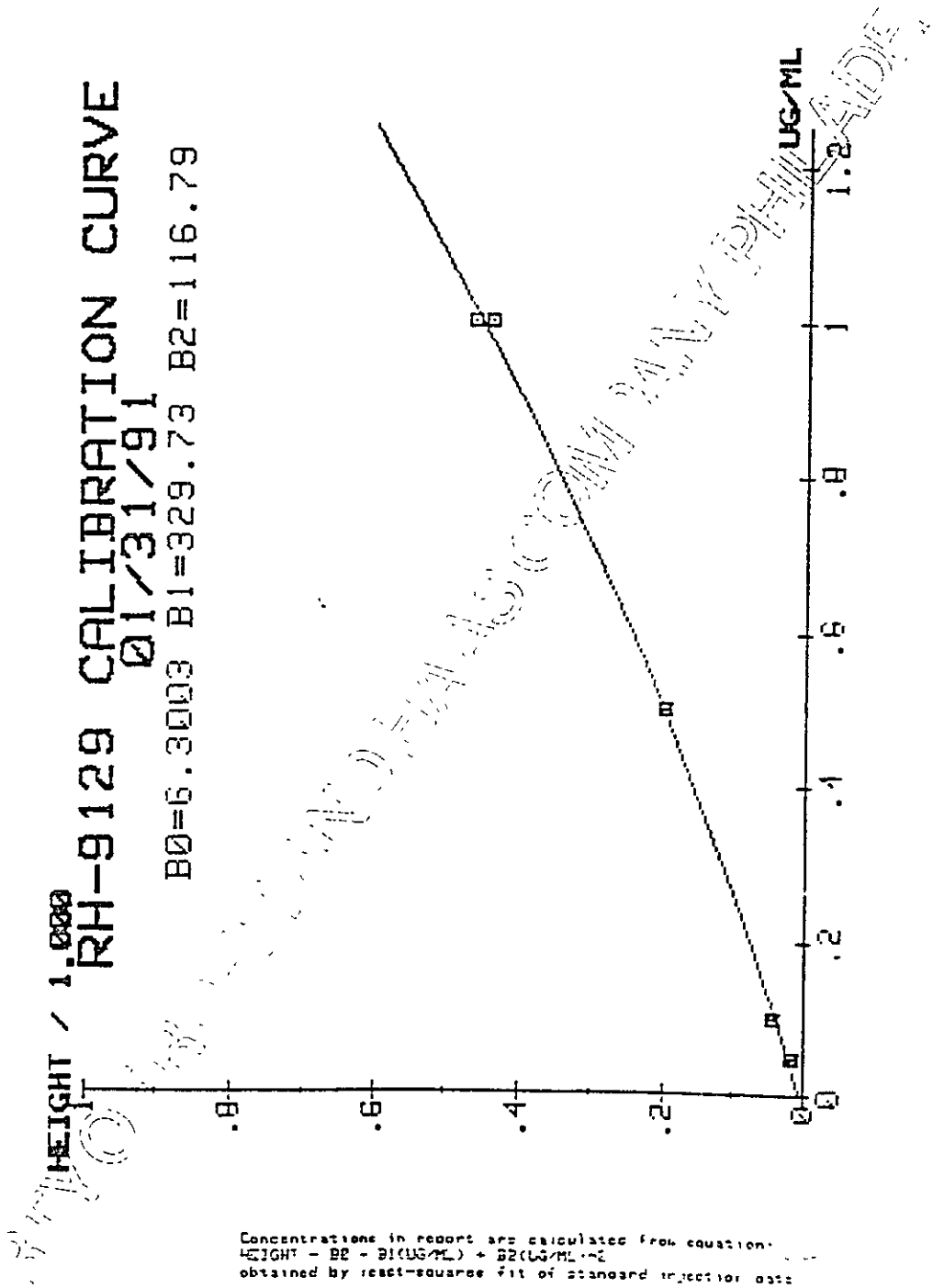
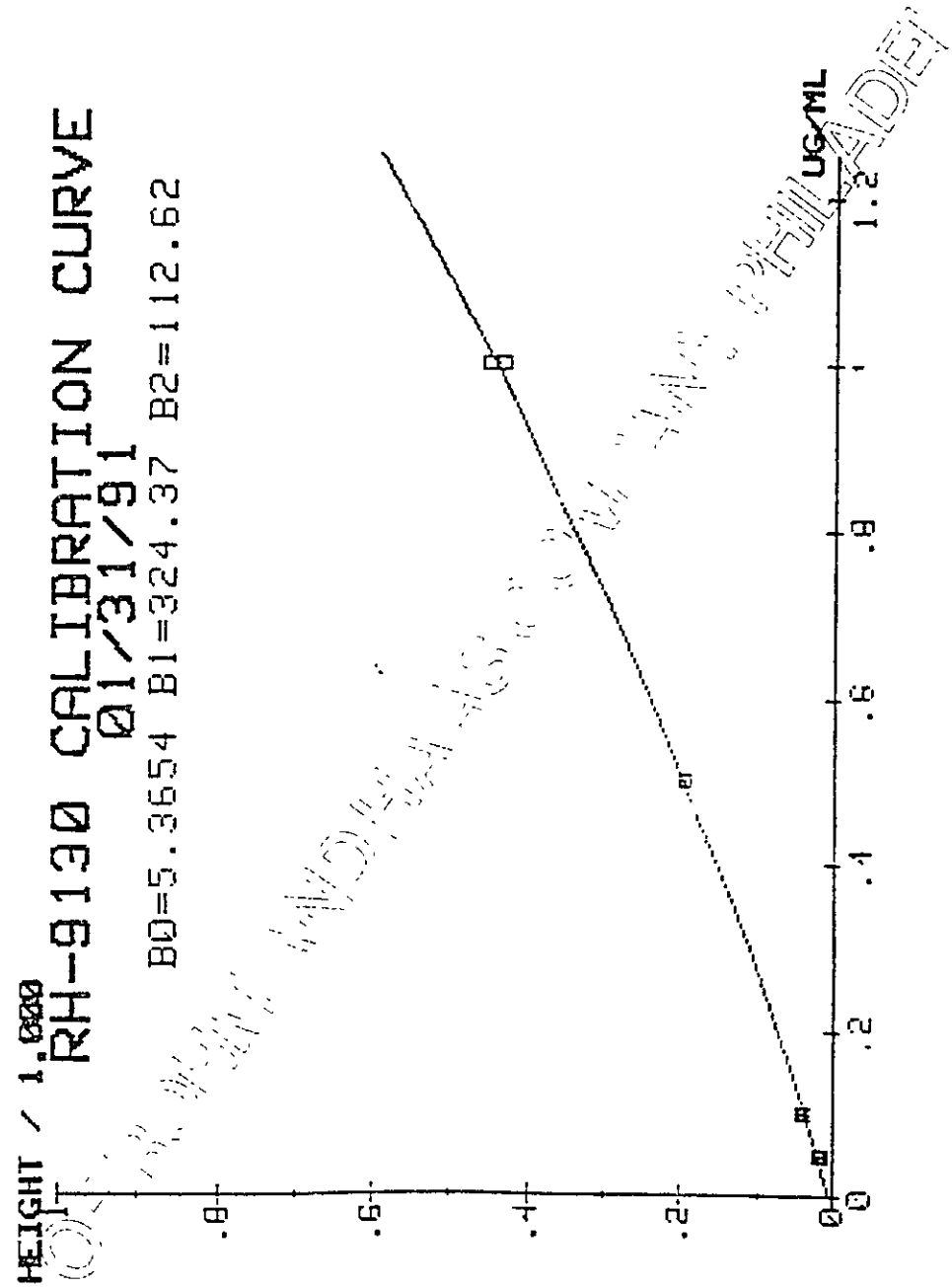


Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data

FIGURE 4

TYPICAL RH-9129 CALIBRATION CURVE



Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data

FIGURE 5
TYPICAL RH-9130 UG/ML CALIBRATION CURVE

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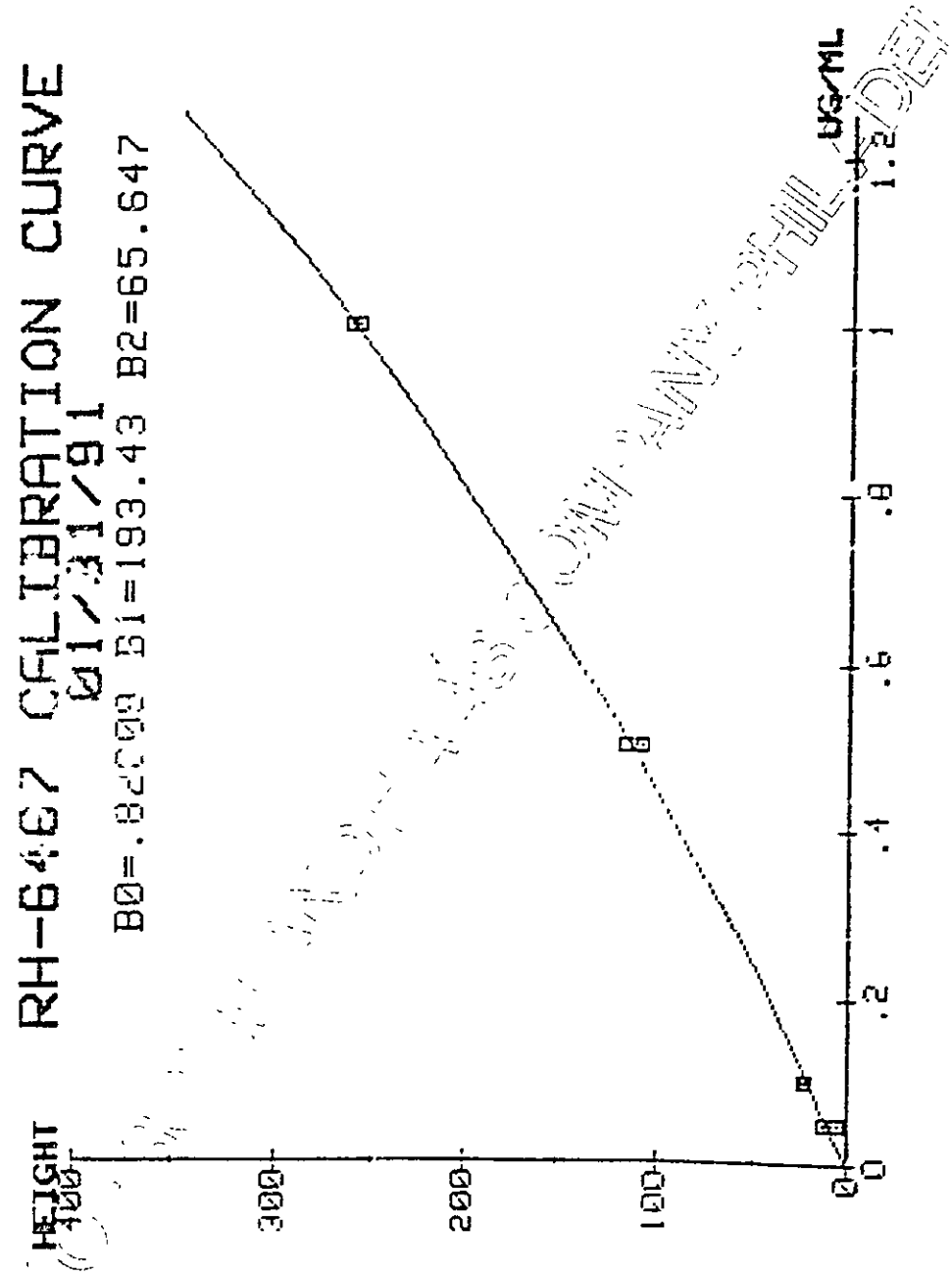

Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data

FIGURE 6
TYPICAL Rh-6467 CALIBRATION CURVE

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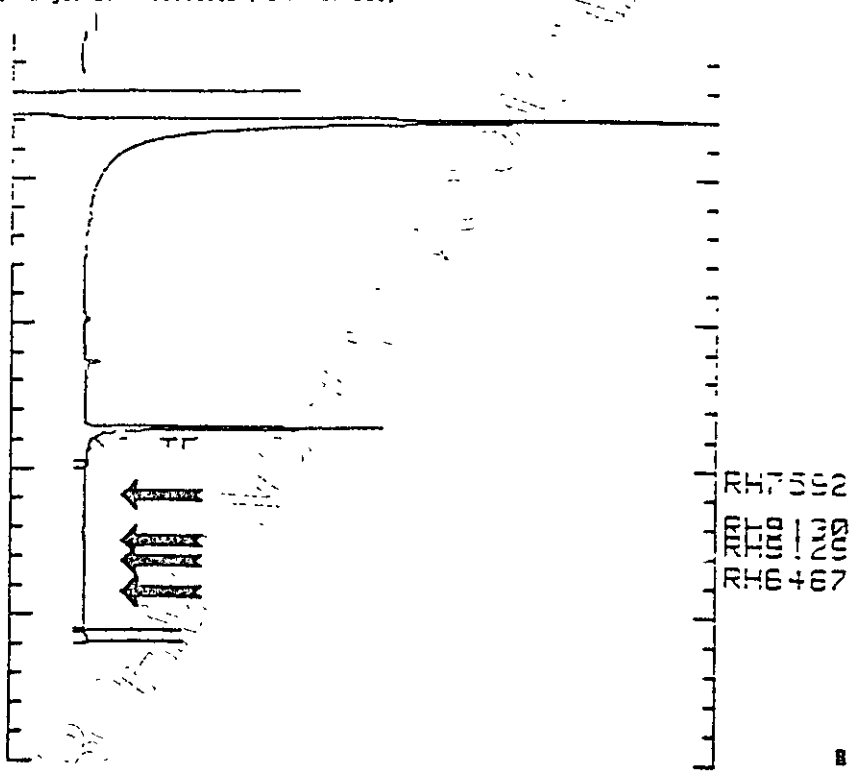
TR 34-94-175

Data file: J1319117 RAR number: 90-0229
Method file: M7592Dm Sample No: 007
Type: SAMPLE Component: NUT

Sample Name: PECAN Cal. Curve: 1/31/91
Date: 31 Jan 1991 19:25 Method: M7592Dm Operator: MD
Interface: /13 Cycle#: 17 Channel#: A

Instrument: 00-25-89
Column: RTX-35 Column Length: 30 Meters
Start Temp-Time (deg-min): 220 Ramp Hold (deg-min):
Program Rate (deg/min): 10 End Time-Temp (deg-min): 285
Prog Slope (# or Linear): Inj Port Temp: 275
Flowrate/Gas: 10 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 25 minutes
Plot range: 20 millivolts (-5 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
15.94	RH7592	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
17.46	RH9130	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
18.16	RH9129	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
19.21	RH6467	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00

Figure 7
Control Pecan Sample (90-0229-007)

FORM 1685 REV. 10/78

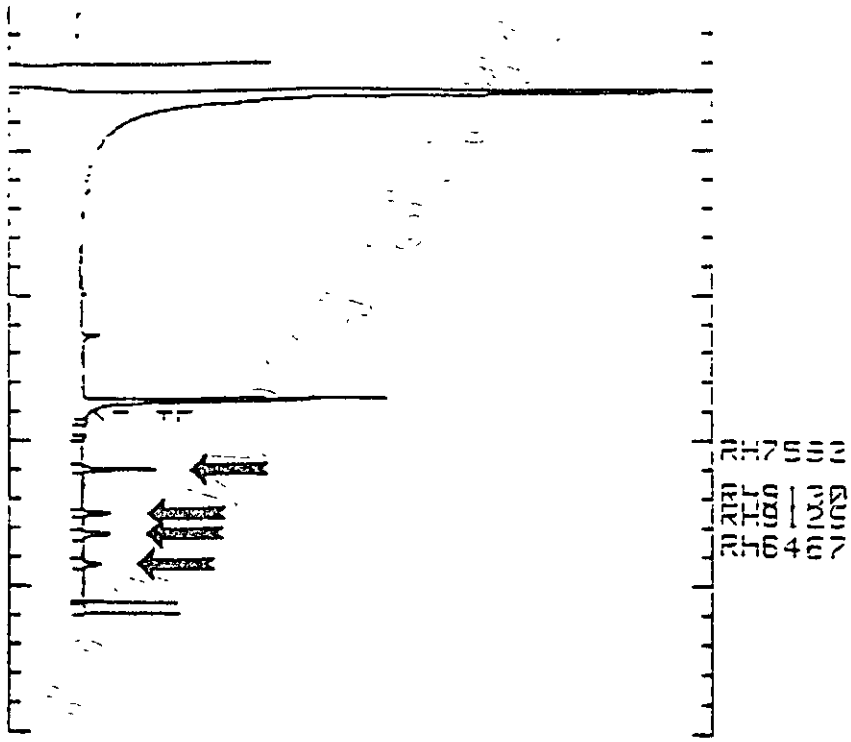
TR 34-94-175

Data File: J1319118 RAR Number: 90-0229
Method File: M7592Dm Sample No: 007
Type: FORTIFICATION Component: WUT

Sample Name: PECAN Cal. Curve: 1/31/91
Date: 31 Jan 1991 19:49 Method: M7592Dm Operator: WD
Interface: 7*3 Cycle#: 18 Channel#: A

Instrument: GC-25-89
Column: TTX-35 Column Length: 30 Meters
Start Temp-Time (deg-min): 220 Ramp Hold (deg-min):
Program Rate (deg/min): 10 End Time-Temp (deg-min): 285
Prog Slope (# or Linear): Inj Port Temp: 275
Flowrate/Gas: 10 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 25 minutes
Plot range: 20 millivolts (-3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Ctl Corr.	ug Added	Pct Recov
15.96	RH7592	2.090E+00	.195E+03	0.204	5.00	0.000	1.00	102.0
17.48	RH9130	9.290E-01	.745E+02	0.199	5.00	0.000	1.00	99.5
18.19	RH9129	9.760E-01	.715E+02	0.186	5.00	0.000	1.00	93.0
19.24	RH6467	7.130E-01	.467E+02	0.221	5.00	0.000	1.00	111.0

Figure 8
Control Pecan Sample (90-0229-007)
Fortified with 0.04 ppm

APPENDIX A

Material Safety Data Sheet

TR 34-94-175

ROHM AND HAAS COMPANY

CORPORATE PRODUCT INTEGRITY DEPARTMENT
INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19105

EMERGENCY TELEPHONE
215-592-3000 (ROHM AND HAAS)
800-424-9300 (CHEMTREC)

HAZARD RATING
4=EXTREME
3=HIGH
2=MODERATE
1=SLIGHT
0=INSIGNIFICANT
*=SEE SECTION IV

JL08
LIST 5

MATERIAL SAFETY DATA SHEET

NOT OSHA HAZARDOUS

MATERIAL	CODE	KEY	DOT HAZARD CLASS
RH-7592 TECHNICAL MATERIAL	NONE	892353-3	NONREGULATED
	DATE ISSUED 02/02/88		

FORMULA	CHEMICAL NAME OR SYNONYMS
NA	Proprietary triazole fungicide

I - COMPOSITIONAL INFORMATION

	CAS Reg. No.	APPROX WT %	TWA/TLV R&H	OSHA	ACGIH
Triazole fungicide	TR SECRET #1	95-100	NE	NE	NE
Related reaction products	NE	0-5	NE	NE	NE

NE=none established

II - PHYSICAL PROPERTY INFORMATION

APPEARANCE - ODOR - pH	VISCOSITY
White solid; slight	NA

MELTING OR FREEZING POINT	BOILING POINT	VAPOR PRESSURE mm Hg	VAPOR DENSITY (AIR=1)
116-118C /241-244F	No data	<1@200C/392F	NA
SOLUBILITY IN WATER	**PERCENT VOLATILE (BY WEIGHT)**	**SPECIFIC GRAVITY (WATER=1)**	**EVAPORATION RATE (BUTYL ACETATE=1)**
[illegible]	0	1.20	NA

III - FIRE AND EXPLOSION HAZARD INFORMATION

FLASH POINT	AUTO IGNITION TEMPERATURE	LOWER EXPLOSION LIMIT (%)	UPPER EXPLOSION LIMIT (%)
NA	NA	NA	NA

EXTINGUISHING MEDIA
☐ FOAM ☐ "ALCOHOL" FOAM ☒ CO_2 ☒ DRY CHEMICAL ☒ WATER SPRAY ☐ OTHER

SPECIAL FIRE FIGHTING PROCEDURES
Wear respirator (pressure-demand, self-contained breathing apparatus, MSHA/NIOSH-approved or equivalent) and full protective gear. Run-off water should be contained.

UNUSUAL FIRE AND EXPLOSION HAZARDS
Toxic fumes are evolved when material is exposed to fire. Personnel should remain upwind and avoid exposure to smoke, because pesticides particulates may become airborne.

IV - HEALTH HAZARD INFORMATION

ROHM AND HAAS RECOMMENDED WORK PLACE EXPOSURE LIMITS
TWA--See Section I.

EFFECTS OF OVEREXPOSURE
Skin Contact: Irritating to skin upon repeated or prolonged exposure.

Eye Contact: Possibly irritating to eyes.

Ingestion: Possibly harmful if swallowed.

EMERGENCY AND FIRST AID PROCEDURES
Inhalation: Move subject to fresh air.

Eye and Skin Contact: Flush eyes with large amounts of water for at least 15 minutes. Consult a physician if irritation persists. Wash affected skin areas with soap and water.

Ingestion: If swallowed dilute by giving 2 glasses of water to drink and call a physician. Never give anything by mouth to an unconscious person.

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V – REACTIVITY INFORMATION

STABILITY	CONDITIONS TO AVOID
[X] STABLE [ ] UNSTABLE	NA

HAZARDOUS DECOMPOSITION PRODUCTS
None known

HAZARDOUS POLYMERIZATION	CONDITIONS TO AVOID
[ ] MAY OCCUR [X] WILL NOT OCCUR	NA

INCOMPATIBILITY (MATERIALS TO AVOID)
[ ] WATER [X] OTHER Oxidizing materials, acids.

VI – SPILL OR LEAK PROCEDURE INFORMATION

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Scoop or shovel into containers for disposal or recovery. Keep dusting to a minimum. Flush contaminated area with a large amount of water to a chemical sewer. Remove contaminated clothing and wash affected skin areas with soap and water. Wash clothing before reuse. Keep spills and cleanning run-offs out of the municipal sewers and open bodies of water.

WASTE DISPOSAL METHODS Pesticide, spray mixture, and rinsate that cannot be used according to label directions must be disposed of by incineration at a permitted facility according to state and local regulations.

VII – SPECIAL PROTECTION INFORMATION

VENTILATION TYPE Mechanical local exhaust at point of contaminant release.

RESPIRATORY PROTECTION
None required if good ventilation is maintained. Wear suitable MSHA/NIOSH approved respirator or equivalent where high mist concentrations are encountered.

PROTECTIVE GLOVES	EYE PROTECTION
Impervious	Chemical splash goggles (ANSI Z87.1)

OTHER PROTECTIVE EQUIPMENT
Eyewash facility, safety shower, protective clothing

VIII – STORAGE AND HANDLING INFORMATION

STORAGE TEMPERATURE MAX / MIN	INDOOR	HEATED	REFRIGERATED	OUTDOOR
	YES	NO	NO	NO

Indoor storage should be in a cool, dry, well ventilated area. Store away from food or feed.

IX – TOXICITY INFORMATION

Acute oral LD50 (rat): >2 g/kg (male)
Acute dermal LD50 (rat) >5 g/kg
Skin rabbit: practically non-irritating, Eye rabbit: inconsequentially irritating.

X – MISCELLANEOUS INFORMATION

CONTAINER DISPOSAL Triple rinse (or equivalent) or puncture and dispose of in a sanitary landfill or by other state or federally approved procedures, such as, incineration.

NA = NOT APPLICABLE C = CEILING VALUE	KEY	DATE OF ISSUE	SUPERSEDES
	892353-3	02/02/88	04/11/85

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF

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FORM 4685 REV 12/78