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ROHM AND HAAS COMPANY
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STUDY TITLE

A Residue Analytical Method for Parent RH-7592 in Stonefruit

DATA REQUIREMENT

Guideline 171-4

AUTHOR

John J. Martin

ROHM AND HAAS STUDY DIRECTOR

John J. Martin

STUDY COMPLETED ON

November 29, 1988

PERFORMING LABORATORY

Rohm and Haas Company
727 Norristown Road
Spring House, PA 19477

LABORATORY PROJECT ID

Rohm and Haas Technical Report No. 34S-88-20

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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 Agent: Michael A. Morelli Date: December 2, 1988

Product Registration Manager Michael A. Morelli
TITLE SIGNATURE

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GOOD LABORATORY PRACTICE CONFORMANCE STATEMENT

This study was conducted in conformance with standards of Good Laboratory Practice as defined by the United States Environmental Protection Agency.

John J. Martin 11/30/88

John J. Martin — Date
Study Director

QUALITY ASSURANCE STATEMENT

This report and the original raw data from this study have been reviewed by the Quality Assurance Unit of the Rohm and Haas Company Agricultural Products Development Research Department, and have been validated as a true and accurate representation of the data collected.

Rhonda S. Krick 11/29/88

Rhonda S. Krick — Date
Quality Assurance

Table of Contents

	Page
I. Summary	6
II. Introduction	6
III. Method	6
A. Chemicals	6
B. Preparation of Solutions	8
C. Equipment	8
D. Instrumentation	9
E. Analytical Procedure	9
1. Sample Processing	9
2. Extraction	10
3. Partition	10
4. Clean-up	10
a. BioSil A Column Cleanup	10
b. Florisil Column Cleanup	11
5. Gas Chromatography	11
a. Preparation of Standard Cruves	11
b. Sample Analysis	11
6. Method of Calculation	11
a. Fortification Recovery	11
b. RH-7592	12
IV. Results and Discussion	12
V. Project Information and Study Certification Statement	12
Tables	
I. Detailed Recovery Data for RH-7592 in Stonefruit	14
Figures	
1. 1.0 ug/ml RH-7592 Standard	15
2. 0.5 ug/ml RH-7592 Standard	16
3. 0.1 ug/ml RH-7592 Standard	17
4. 0.05 ug/ml RH-7592 Standard	18
5. 0.02 ug/ml RH-7592 Standard	19
6. Calibration Cruve 09/14/88	20
7. Control Peach Sample No. 87-0170-029	21

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Table of Contents (continued)

Page

8. Control Peach Sample No. 87-0170-029 fortified with 0.65 ppm RH-7592 22
9. Treated Peach Sample No. 87-0170-017 23
10. Control Cherry Sample No. 87-0333-025 24
11. Control Cherry Sample No. 87-0333-025 fortified with 0.49 ppm RH-7592 25
12. Treated Cherry Sample No. 87-0333-006 26
13. Control Plum Sample No. 87-0206-009 27
14. Control Plum Sample No. 87-0206-009 fortified with 0.10 ppm RH-7592 28
15. Treated Plum Sample No. 87-0206-012 29

Appendix

A. Material Safety Data Sheet for RH-7592 30

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I. Summary

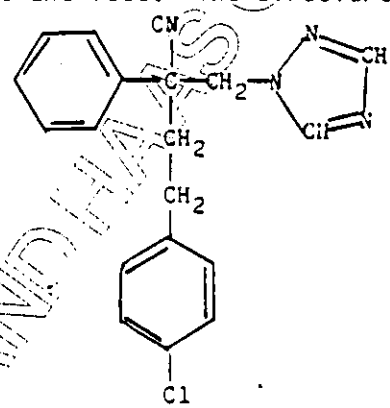
RH-7592 is extracted from stonefruit by blending with methanol. The methanol extract is partitioned with 10% sodium chloride solution and methylene chloride. The methylene chloride is evaporated and the samples are cleaned up on silica gel and Florisil.

RH-7592 is determined by gas chromatography using a 0.53 mm ID capillary column (SPB-608) 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 0.01 and the overall average recovery is 93 ± 12%.

II. Introduction

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



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

III. Method

A. Chemicals/Supplies

Acetone, Pesticide Grade	Fisher
BioSil A, 100-200 Mesh (Activate at 200°C for 24 hours. Bottle and cap immediately. Store in a desiccator cabinet.)	Bio-Rad
Boileezers	Fisher
Celite-545	Johns Mansville

FLOW DIAGRAM OF METHOD

Extract with Methanol

Partition with 10% Sodium Chloride Solution and Methylene Chloride

BioSil A Column Cleanup

Florisil Column Cleanup

GLC on SPB-608 (Nitrogen Specific Detector)

Quantitation (Data System)

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Cotton, Sterile, Absorbent	Johnson & Johnson
Florisil 60-100 Mesh (activate at 200°C for 24 hours. Bottle and cap immediately. Store in a desiccator cabinet.)	Floridin Co.
Filter paper, 7 cm, P4 Qualitative	Fisher
Methanol, Pesticide Grade	Baker
Methylene Chloride, Pesticide Grade	Baker
Sodium Chloride, Certified A.C.S.	Fisher
Sodium Sulfate, Anhydrous, Granular AR	Mallinckrodt
RH-7592 Analytical Standard	Rohm and Haas Co.
Water, Milli-Q	Millipore

B. Preparation of Solutions

1. Prepare a 10% sodium chloride solution by dissolving 400 g of sodium chloride in four liters of Milli-Q water.

2. Prepare 100/25, 100/15 and 100/10(v/v) toluene/acetone solutions by adding 250, 150 and 100 ml of acetone to 1000 ml of toluene.

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

4. Prepare standard GLC and fortification solutions by carefully weighing on an analytical balance 0.010 g of RH-7592 analytical standard into a 100 ml volumetric flask. Add approximately 80 ml of the toluene/methanol (100/3) solution and sonicate until dissolution occurs. Bring to volume with toluene/methanol(100/3). This is the primary standard of 100 ug/ml Make serial dilutions to 10, 5, 1, 0.5, 0.1, 0.05 and 0.02 ug/ml for working standards.

C. Equipment

Blenders Explosion-Resistant, Waring EPI	A. H. Thomas
Buchner Funnels, Porcelain, 83 mm ID	A. H. Thomas
Chromatography columns, 17 mm OD, 14.5 mm ID with 250 ml reservoir and Teflon stopcock	Kontes

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Filter Flasks, 500 ml	Fisher
Food Processor, Commercial	Waring
Hobart Food Chopper	Hobart Mfg. Co.
Rotary Evaporator, Buchi Rotovap R with dry ice trap	Brinkman
Round bottom flasks, 500, 300 ml	Fisher
Separatory funnels, 500 ml	Fisher
Standard laboratory equipment, balances, beakers, etc.	
Utrasonic Cleaner	Branson
Volumetric Flasks, 100 ml	Kimble

D. Instrumentation

Varian 3500 Capillary Gas Chromatograph equipped with a Varian Model 8035 Autosampler 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.

Column: Fused silica capillary, SPB-608, 0.53 mm ID, 15 meters, 0.5 um df - Supelco

Temperatures: Column - 245°C
Injector - 255°C
Detector - 300°C

Flows: Air (zero grade) - 175 ml/min
Hydrogen (UPC) - 4.5 ml/min
Helium (UPC) - 18.7 ml/min, 10 ml/min purge

Bead Current: 3.3 amps (varies)

Under these conditions, the retention time of RH-7592 is 4.20 minutes.

E. Analytical Procedure

1. Sample Processing

All samples are received frozen (dry ice) at the Rohm and Haas Research Laboratories. The samples are slightly thawed and split with a knife. The stones are removed and discarded. The fruit is well chopped in the Hobart Food Chopper with dry ice and is thoroughly mixed. The dry ice is allowed to sublime overnight in a freezer (4°F).

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All samples are kept frozen at -10°F until analysis.

2. Extraction

Allow the samples to thaw sufficiently to remove an aliquot for analysis. Weigh 25 g of the chopped fruit into a 1 pint blender jar. Add 5-10 g of Celite-545, 100 ml of methanol, cap and blend for 5 min. Vacuum suction filter the blended sample through the 7.5 cm filter paper using a porcelain filter funnel and a 500 ml filter flask. Wash the blender jar with 25 ml of methanol and pour over the filter cake into the 500 ml flask. Transfer the filtrate to a 500 ml separatory funnel. Wash the filter flask with 10 ml of methanol and add it to the separatory funnel.

3. Partition

To the 500 ml separatory funnel containing the methanol extract, add 150 ml of methylene chloride and 250 ml of 10% sodium chloride solution. Let the solution stand for one or two minutes to allow the release of pressure. Cap and shake for approximately 10 seconds. Invert the separatory funnel and release the pressure by opening the stopcock. Close the stopcock and shake vigorously for one minute. When the phases separate, draw down the lower methylene chloride phase into a 500 ml round bottom flask. Evaporate the methylene chloride on the rotary evaporator at 45-50°C at atmospheric pressure. Remove the final traces of methylene chloride under vacuum. Add 25 ml of toluene/acetone(100/15) and swirl to dissolve the residue. Proceed to the BioSil cleanup step.

4. Cleanup

a. BioSil A Column Chromatography

Insert a small cotton plug into a 14.5 mm ID chromatographic column and dry pack the column with 10 cc of the activated BioSil A (measure in a 10 cc graduated cylinder). Top the column with 1 inch of anhydrous sodium sulfate. Add the 25 ml of toluene/acetone(100/15) solution in the 500 ml round bottom flask from the partition 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(100/15) and add to the column when the previous addition just enters the top of the column. Add 25 ml of toluene/acetone(100/15) 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. Collect 150 ml of toluene/acetone(100/25) in a 300 ml round bottom flask concentrate the eluent to dryness on the rotary evaporator at 70°C under reduced pressure. Add 25 ml of toluene to the flask and swirl to dissolve the residue. Proceed to the Florisil column cleanup step.

b. Florisil Column Chromatography

Insert a small cotton plug into a 14.5 mm ID chromatographic column and dry pack the column with 7 cc of the activated Florisil (measure in a 10 cc graduated cylinder). Top the column with 1 inch of anhydrous sodium sulfate. Add the 25 ml of the toluene solution in the 300 ml round bottom flask to the column and collect the eluent in a 125 ml Erlenmeyer flask. Rinse the 300 ml round bottom flask with 10 ml of toluene and add to the column when the previous addition just enters the top of the column. Discard the eluent. Collect 150 ml of toluene/acetone(100/10) in a 300 ml round bottom flask and concentrate the eluent to dryness on the rotary evaporator at 70°C under reduced pressure. For all samples, add the appropriate volume of toluene/methanol(100/3) for gas chromatography (5 ml).

5. Gas Chromatography

a. Preparation of Standard Curves

A 2 ul aliquot of each standard in the range of 1.0 to 0.02 ug/ml is injected into the gas chromatograph. The resulting peak heights are measured and plotted vs. concentration (ug/ml) of RH-7592 to obtain a standard curve. Standard curves are prepared for each analysis day.

b. Sample Analysis

A 2 ul 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 height is measured and the concentration is determined from the standard curve. The limit of detection is set by the data system at one-half the value of the peak height of the lowest standard injected (0.5 x lowest peak height of the 0.02 ug/ml standard).

6. Method of Calculation

The RH-7592 residue concentration is determined as follows:

a. Fortification Recovery

For samples fortified with known amounts of RH-7592 prior to extraction, measure the peak height, determine the concentration (ug/ml) from the standard curve and calculate the percent recovery from the equation 1.

Eq. 1 $$\frac{\text{(ug/ml Found)} \times \text{Final Sample Volume (ml)}}{\text{Fortification (ug)}} \times 100 = \%\ \text{Recovery}$$

b. RH-7592 Residue Concentration

The RH-7592 residue concentration is determined as follows:

Eq. 2 $\frac{\text{Final Sample Volume (ml)} \times \text{Concentration (ug/ml)}}{\text{Average Recovery (\%)} \times \text{Sample Weight (g)}} \times 100 = \text{ppm}$

IV. Results and Discussion

Recoveries of fortifications to the fruit averaged 93 ± 12% (n = 38 with a range of 62% minimum to 116% maximum. Detailed recovery data are shown in Table I. Sample chromatograms of standards and substrates are shown in Figures 1 to 15. A typical calibration curve is shown in Figure 6.

Each lot of BioSil A should be evaluated for consistency by running a reagent fortification through the elution scheme. Note that RH-7592 will elute slightly later than when chromatographed in the presence of substrate.

The SPB-608 capillary column should be conditioned overnight at 260°C before connecting it to the detector. It was also necessary to prime the column before analysis by making two injections of the 5 ug/ml standard solution followed by two injections of toluene/methanol(100/3) solvent. If this is not done, standards injected at the beginning of an analytical run give a lower detector response than standards injected at the end of a run. ok

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 0.02 ug/ml concentration. The bead current will need to be adjusted as the bead ages.

The extraction efficiency is very good as metabolism studies with field aged radiolabelled RH-7592 treated peaches have shown that blending with methanol extracts 95% of the total radioactive residue from the fruit.

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

V. Project Information and Study Certification Statement

Title: A Residue Analytical Method for Parent RH-7592 in Stonefruit

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

Dates: Work Initiated: October 27, 1986
Wrok Completed: October 18, 1988

Study Personnel: Study Director: John J. Martin
Theodore F. Burnett

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Notebook References:

JM-35 Rohm and Haas No. 048623
JM-36 Rohm and Haas No. 048674
JM-37 Rohm and Haas No. 51666
JM-38 Rohm and Haas No. 52305
TB-4 Rohm and Haas No. 50808
TB-5 Rohm and Haas No. 52778

Technical Report References:

No. 34A-88-55 RH-7592 Parent Residue Data and Half-Life of Decline for Peaches, RAR 87-0237, 87-0276, 87-0156, 87-0245, 87-0170

No. 34A-88-62 RH-7592 Parent Residue Data for Cherry, RAR 87-0194, 87-0263, 87-0269, 87-0333, 87-0110, 87-0113, 87-0211

No. 34A-88-69 RH-7592 Parent Residue Data for Plums, RAR 87-0206, 87-0258, 87-0204, 87-0163

This report is an accurate and true reflection of the study as it was performed.

John J. Martin 11/30/88
John J. Martin Date

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Table I

Detailed Recovery Data for RH-7592 in Stonefruit

Sample No.	Standard Curve No.	ug Found	*ug Corrected	ug Added	ppm Added	Percent Recovery
Peaches						
87-0276-021	021688	0.857	0.814	1.00	0.04	81
87-0156-029	040488	1.55	1.55	2.00	0.08	78
87-0237-027	04/20/88	2.92	2.85	2.50	0.10	114
87-0245-031	09/28/88	2.04	2.04	2.50	0.10	82
87-0237-026	04/06/88	5.20	5.03	5.00	0.20	101
87-0156-029	040488	4.47	4.47	5.00	0.20	89
87-0276-033	030388	3.78	3.74	5.00	0.20	75
87-0245-031	04/29/88	5.10	5.02	5.00	0.20	100
87-0170-028	9/07/88	6.18	6.18	10.0	0.40	62
87-0245-013	04/27/88	10.8	10.8	10.0	0.40	108
87-0276-046	031088	11.6	11.6	10.0	0.40	116
87-0276-007	031788	8.96	8.90	10.0	0.40	89
87-0245-014	04/27/88	17.2	17.2	15.0	0.60	115
87-0170-029	09/14/88	15.2	15.2	16.2	0.65	94
87-0156-016	040488	19.0	19.0	20.0	0.80	95
87-0245-032	04/29/88	20.7	20.6	20.0	0.80	103
87-0237-007	4/18/88	19.1	19.1	20.0	0.80	96
87-0276-045	031088	23.5	23.4	25.0	1.0	94
87-0276-010	030988	23.4	23.3	25.0	1.0	93
87-0276-034	031788	37.7	37.7	40.0	1.6	94
Cherries						
87-0113-026	09/28/88	2.74	2.74	2.50	0.10	110
87-0110-026	09/22/88	4.59	4.59	4.86	0.19	94
87-0211-019	09/26/88	4.22	4.22	5.00	0.20	84
87-0333-026	09/20/88	7.02	7.02	7.36	0.29	95
87-0194-009	B09/07/88	8.07	8.03	7.36	0.29	109
87-0263-026	09/12/88	7.61	7.55	7.36	0.29	103
87-0211-019	09/26/88	9.19	9.19	10.0	0.40	92
87-0194-009	09/18/88	8.36	8.36	11.2	0.45	75
87-0263-025	09/09/88	10.6	10.5	12.4	0.49	85
87-0333-025	09/16/88	11.4	11.4	12.4	0.49	92
Plums						
87-0204-028	10/06/88	0.232	0.232	0.25	0.01	93
88-0055-013	10/14/88	0.203	0.203	0.25	0.01	81
87-0367-025	10/21/88	0.187	0.187	0.25	0.01	75
87-0258-021	10/10/88	2.33	2.33	2.50	0.10	93
87-0206-009	10/03/88	2.41	2.41	2.50	0.10	96
87-0204-009	10/04/88	2.42	2.42	2.50	0.10	97
87-0163-026	09/28/88	4.69	4.69	5.00	0.20	94
87-0163-025	09/26/88	11.3	11.3	12.4	0.49	91

Minimum Percent Recovery:	62
Maximum Percent Recovery:	116
Number:	38
Average Percent Recovery:	93
Standard Deviation Percent Recovery:	12

*This is the ug found corrected by substracting the ug found in the corresponsing unfortified control sample.

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TR 34S-88-20

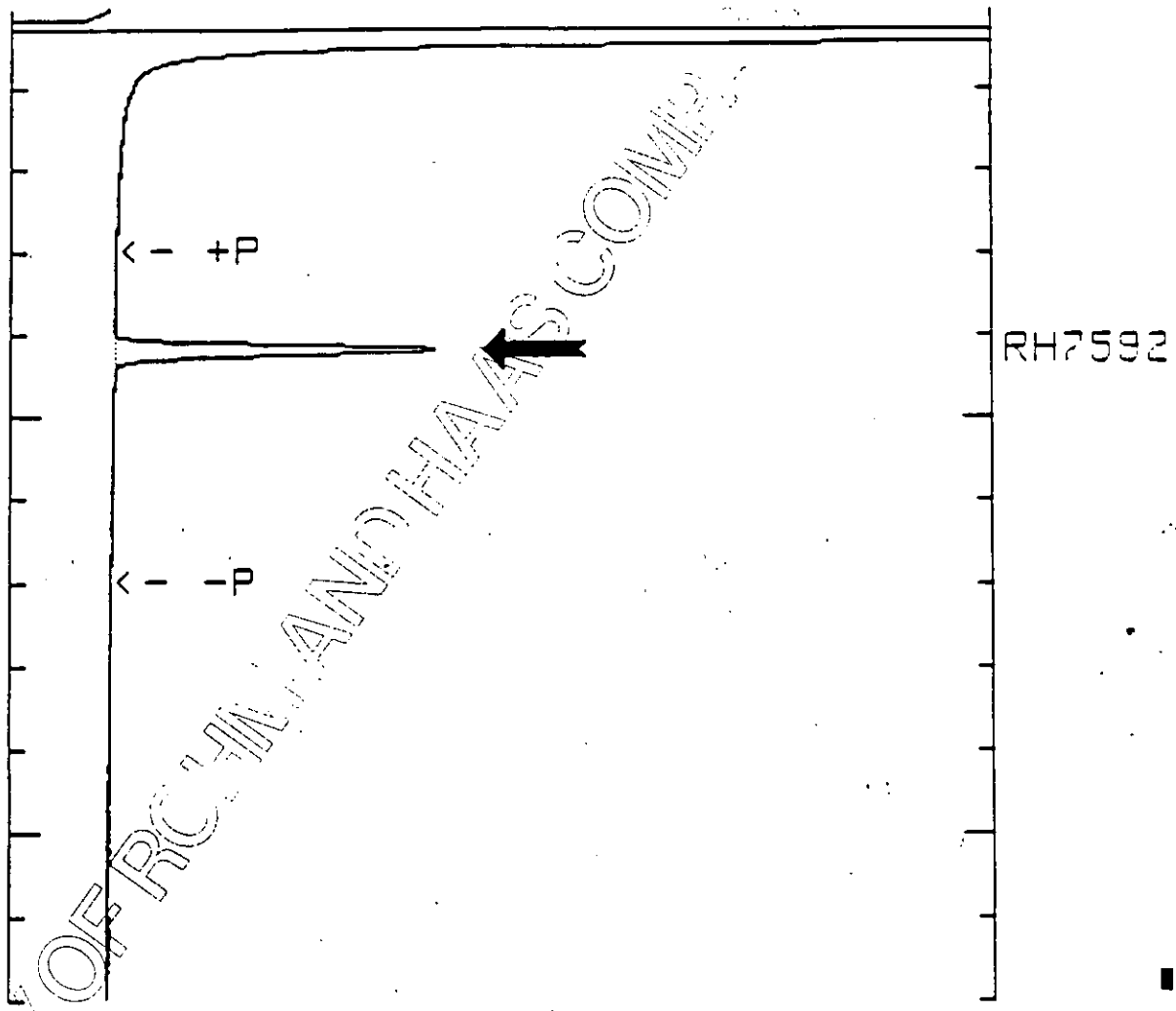
Data file: B914881 Type: STANDARD

000015

Sample Name: PEACHES Cal. Curve: 09/14/88
Date: 14 Sep 1988 18:40 Method: M7592m Operator: TFB
Interface: 711 Cycle#: 1 Channel#: A Vial#: 1

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.18	RH7592	1.00	54.80	644.00

Figure 1

1.0 ug/ml RH-7592 Standard

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TR 34S-88-20

Data file: B9148821 Type: STANDARD

Sample Name: PEACHES Cal. Curve: 09/14/88

Date: 14 Sep 1988 22:50 Method: M7592m Operator: TFB

Interface: 711 Cycle#: 21 Channel#: A Vial#: 21

000016

Instrument: VARIAN 3500(18-87)

Column: SP8-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

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

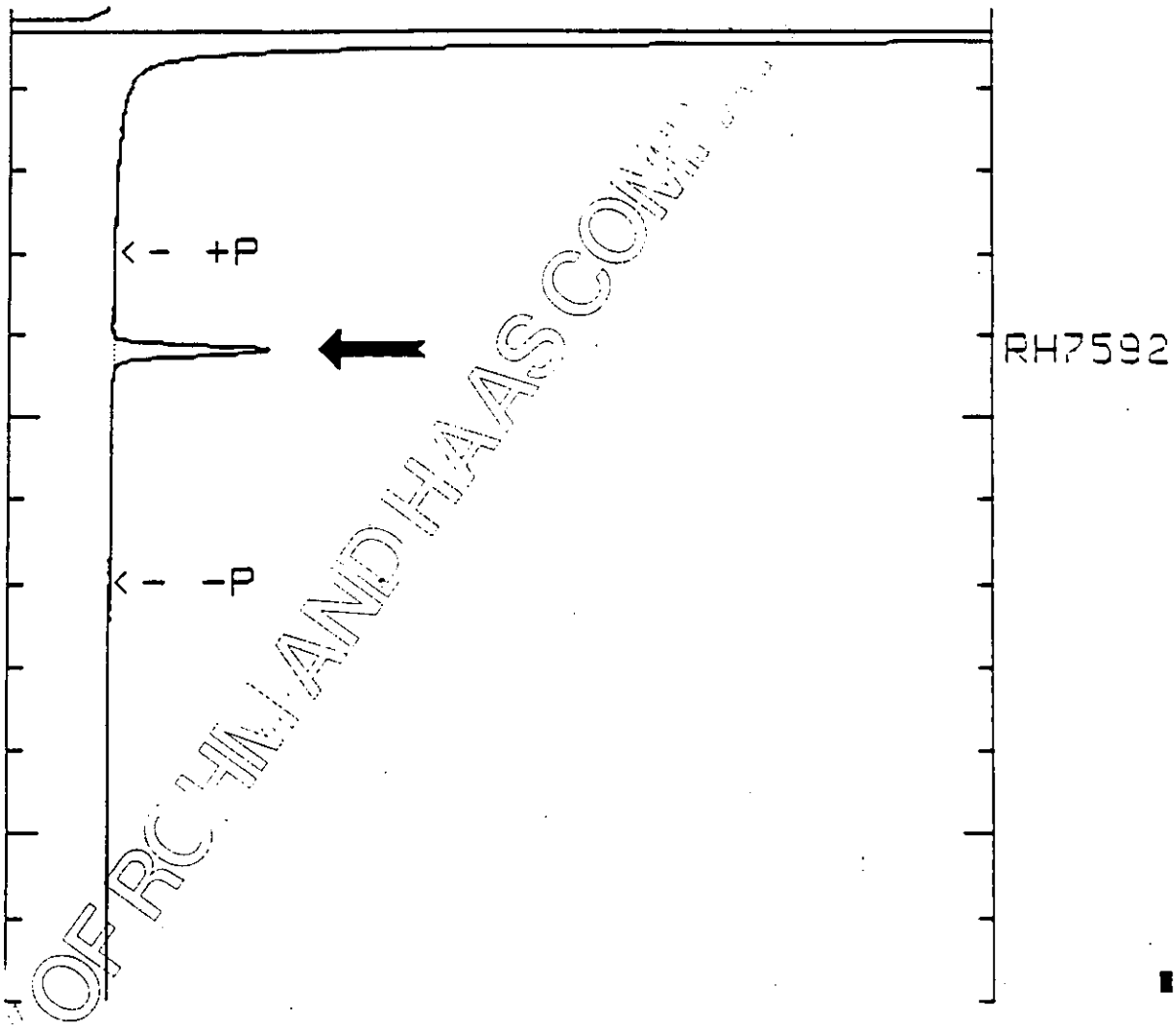
Prog Slope (# or Linear): Inj Port Temp: 255

Flowrate/Gas: 18.7 Split Ratio:

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

Plot times: 0 to 12 minutes

Plot range: 20 millivolts (-3.3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.19	RH7592	0.500	26.60	310.00

Figure 2

0.50 ug/ml RH-7592 Standard

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TR 34S-88-20

Data file: B9148829 Type: STANDARD

Sample Name: PEACHES Cal. Curve: 09/14/88

Date: 15 Sep 1988 01:05 Method: M7592m Operator: TFB

Interface: 711 Cycle#: 29 Channel#: A Vial#: 29

000017

Instrument: VARIAN 3500(18-87)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

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

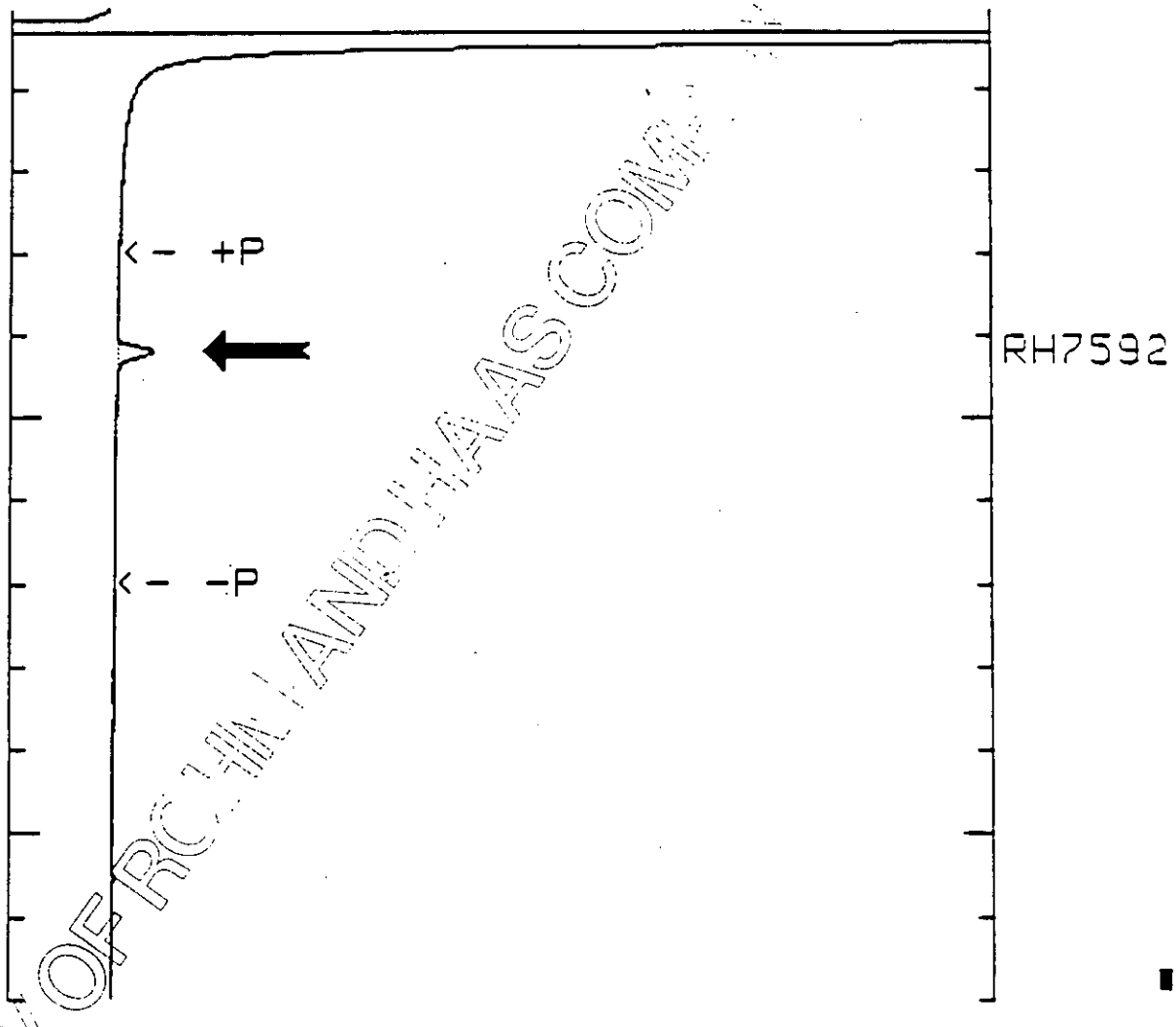
Prog Slope (# or Linear): Inj Port Temp: 255

Flowrate/Gas: 18.7 Split Ratio:

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

Plot times: 0 to 12 minutes

Plot range: 20 millivolts (-3.3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.19	RH7592	0.100	6.24	69.10

Figure 3

0.10 ug/ml RH-7592 Standard

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TR 34S-88-20

Data file: B9148816 Type: STANDARD

Sample Name: PEACHES Cal. Curve: 09/14/88

Date: 14 Sep 1988 21:25 Method: M7592m Operator: TFB

Interface: 711 Cycle#: 16 Channel#: A Vial#: 16

000018

Instrument: VARIAN 3500(18-87)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

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

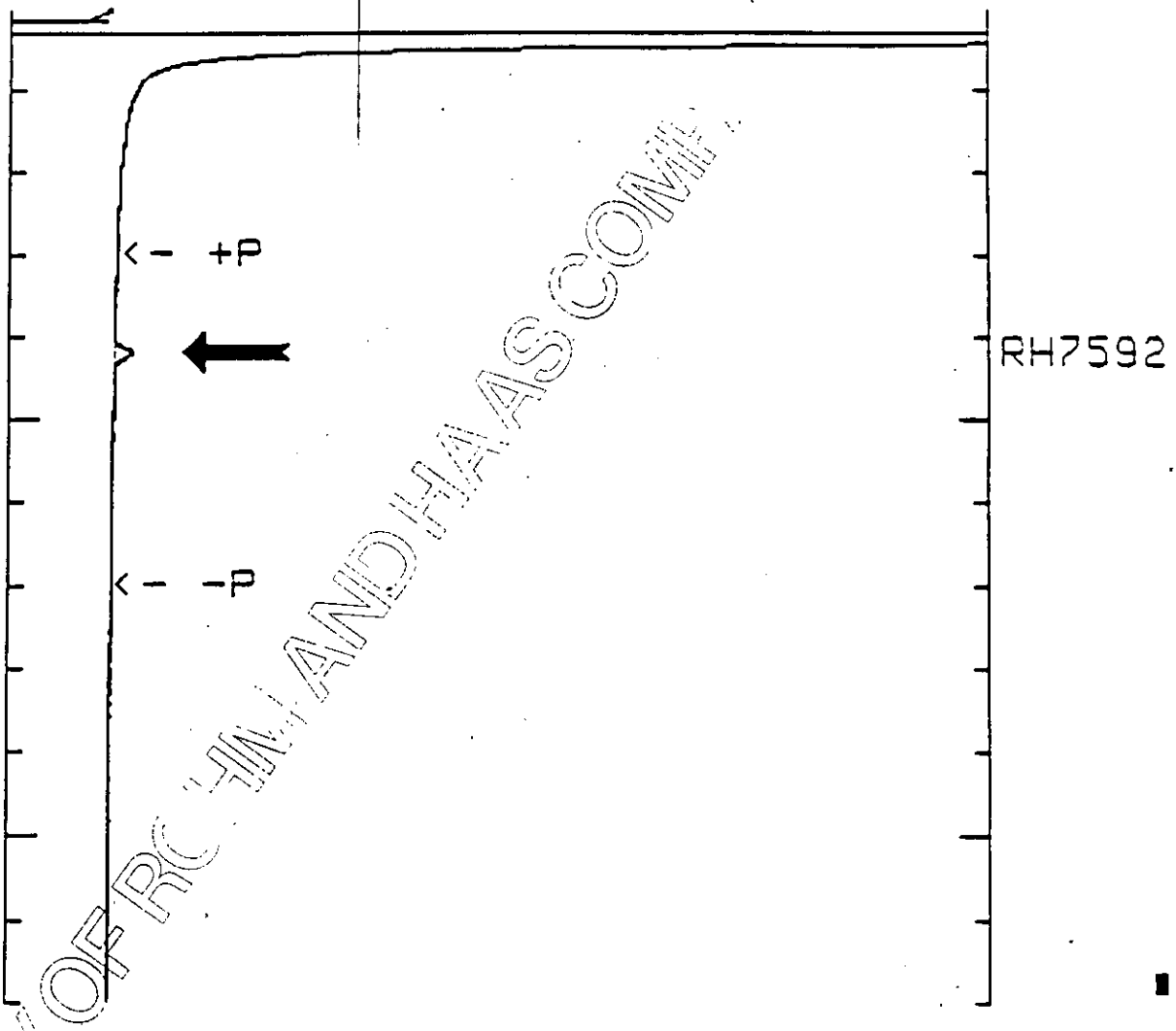
Prog Slope (# or Linear): Inj Port Temp: 255

Flowrate/Gas: 18.7 Split Ratio:

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

Plot times: 0 to 12 minutes

Plot range: 20 millivolts (-3.3 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.19	RH7592	0.050	2.58	32.00

Figure 4

0.05 ug/ml RH-7592 Standard

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TR 34S-88-20

Data file: B9148835 Type: STANDARD

Sample Name: PEACHES Cal. Curve: 09/14/88

Date: 15 Sep 1988 02:47 Method: M7592m Operator: TFB

Interface: 711 Cycle#: 35 Channel#: A Vial#: 35

000019

Instrument: VARIAN 3500(18-87)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

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

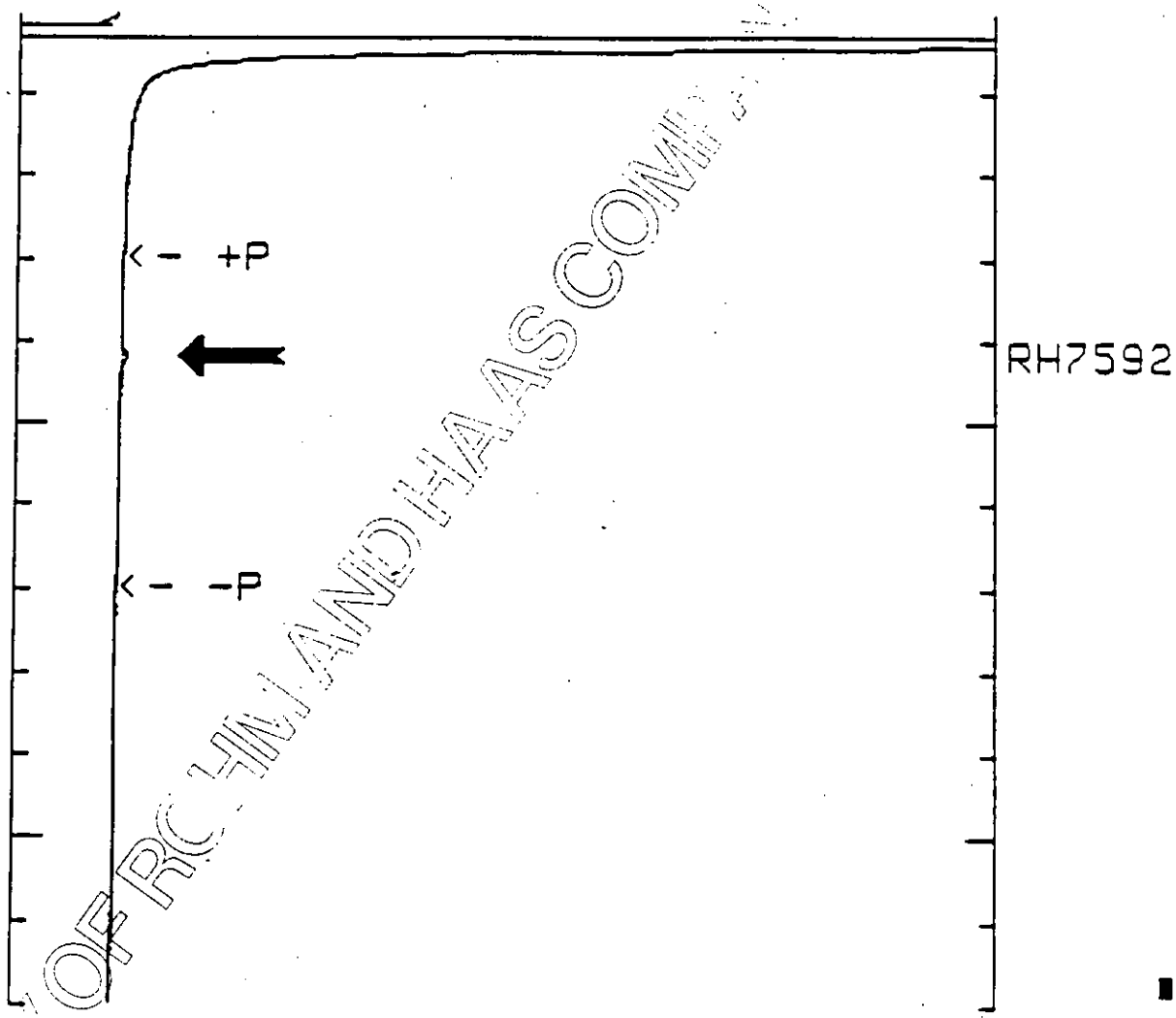
Prog Slope (# or Linear): Inj Port Temp: 255

Flowrate/Gas: 18.7 Split Ratio:

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

Plot times: 0 to 12 minutes

Plot range: 20 millivolts (-3.3 mv offset)

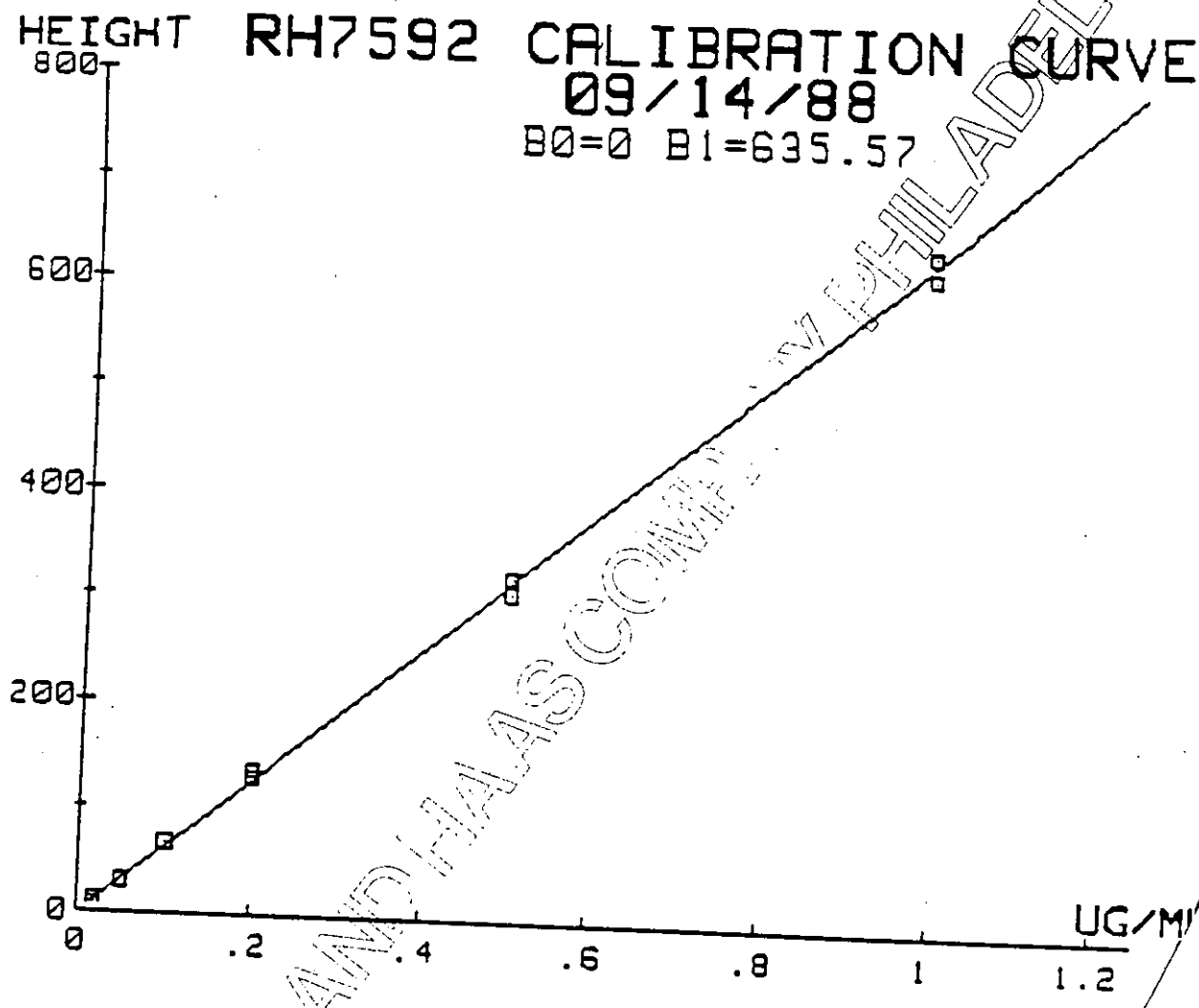


Retention Time	Compound Name	PPM Injected	Area	Height
4.20	RH7592	0.020	.84	11.30

Figure 5

0.02 ug/ml RH-7592 Standard

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA



Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML)
obtained by linear regression of standard injection data
with constrained origin.

Figure 6
Calibration Curve 09/14/88

LIBRARY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data file: B914884
Method file: M7592m
Type: SAMPLE

TR 34S-88-20

RAR number: 87-0170
Sample No: 029
Component: FRUIT

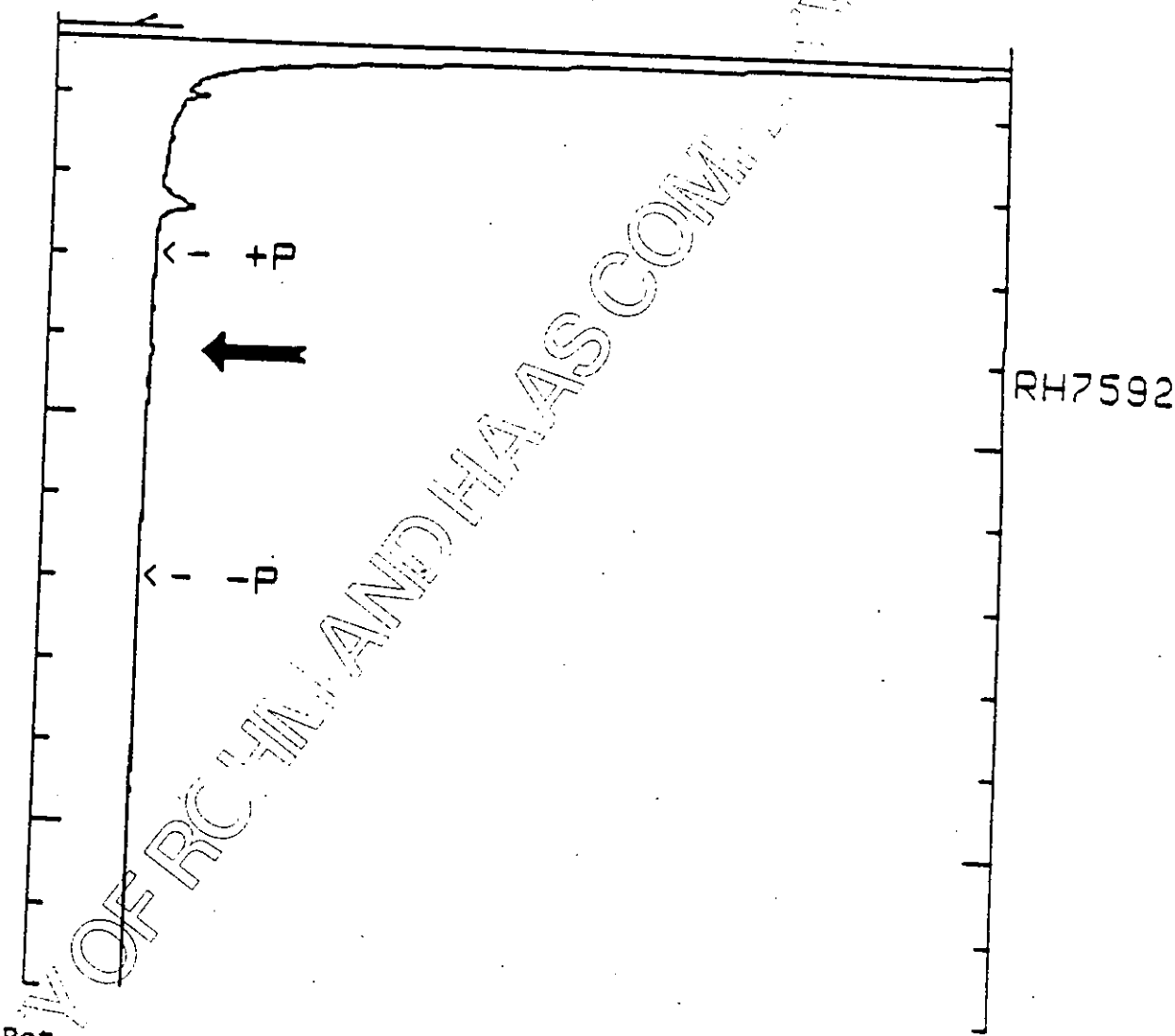
000021

Sample Name: PEACHES
Date: 14 Sep 1988 18:47 Method: M7592m
Interface: 711 Cycle#: 4 Channel#: A Vial#: 4

Cal. Curve: 09/14/88
Operator: TFB

Instrument: VARIAN 3500(18-87)
Column: SPB-608
Column Length: 15 Meters
Start Temp-Time (deg-min): 245
Ramp Hold (deg-min):
Program Rate (deg/min):
End Time-Temp (deg-min):
Prog Slope (# or Linear):
Inj Port Temp: 255
Flourate/Gas: 18.7
Split Ratio:
Det 1-Type & Temp: TSD/300
Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
4.19	RH7592	0.0	0.0	5.00	25.0

Figure 7
Control Peach Sample No. 87-0170-029

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

FORM

IR 345-88-20

Data file: B914885
Method file: M7592m
Type: FORTIFICATION

RAR number: 87-0170
Sample No: 029
Component: FRUIT

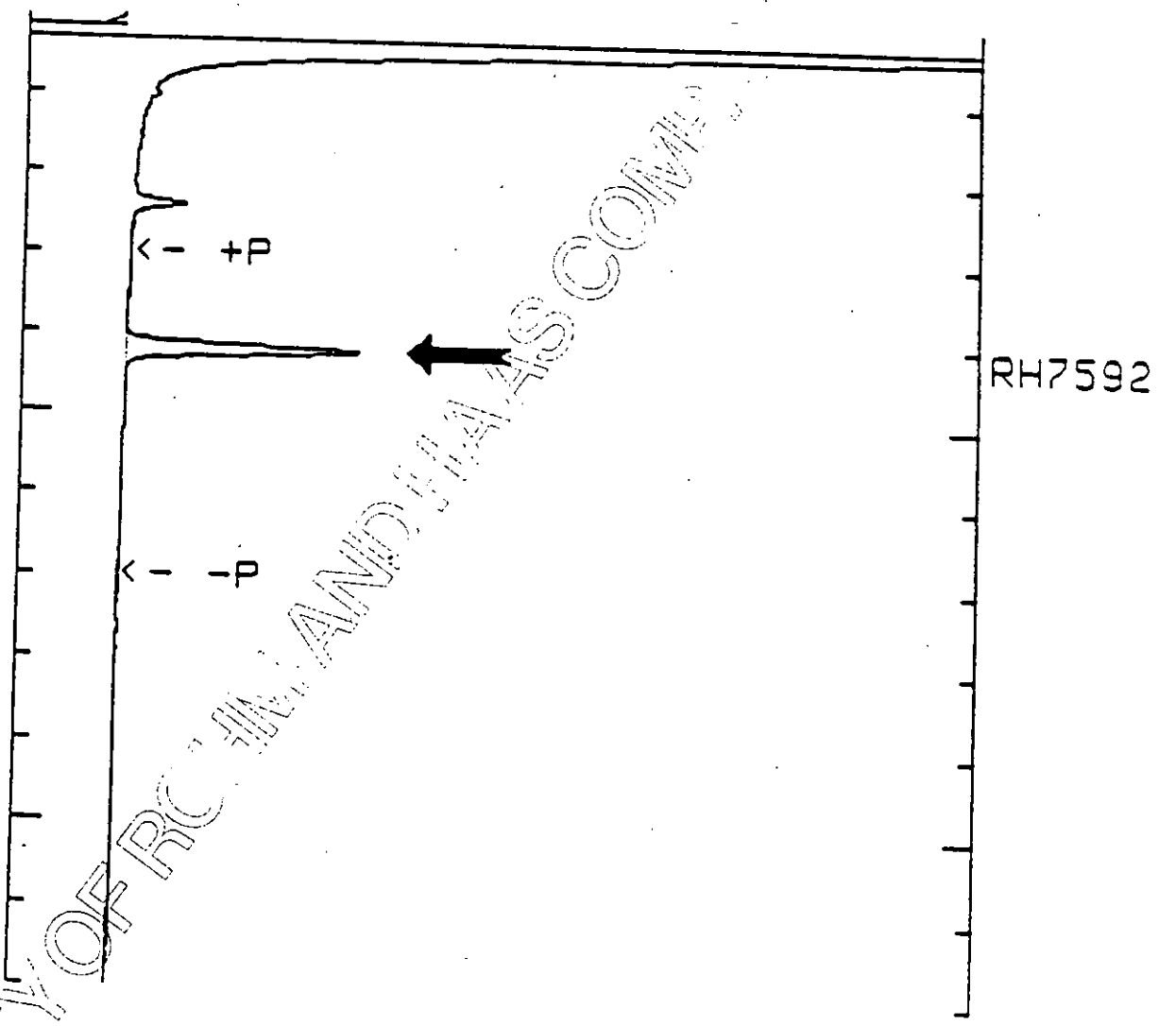
000022

Sample Name: PEACHES
Date: 14 Sep 1988 18:50 Method: M7592m
Interface: 711 Cycle#: 5 Channel#: A

Cal. Curve: 09/14/88
Operator: TFB
Vial#: 5

Instrument: VARIAN 3500(18-87)
Column: SPB-608
Column Length: 15 Meters
Start Temp-Time (deg-min): 245
Ramp Hold (deg-min):
Program Rate (deg/min):
End Time-Temp (deg-min):
Prog Slope (# or Linear):
Inj Port Temp: 255
Flowrate/Gas: 18.7
Split Ratio:
Det 1-Type & Temp: TSD/300
Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	u Add
4.19	RH7592	40.3	484.0	20.0	1

Figure 8

Control Peach Sample No. 87-0170-029 fortified with 0.65 ppm RH-7592

UNIVERSITY OF RICHMAND HAS COMPANY PHILADELPHIA

FORM 4685 PE

Method file: M7592m
Type: SAMPLE

TR 34S-88-20

RAR number: 87-0170
Sample No: 017
Component: FRUIT

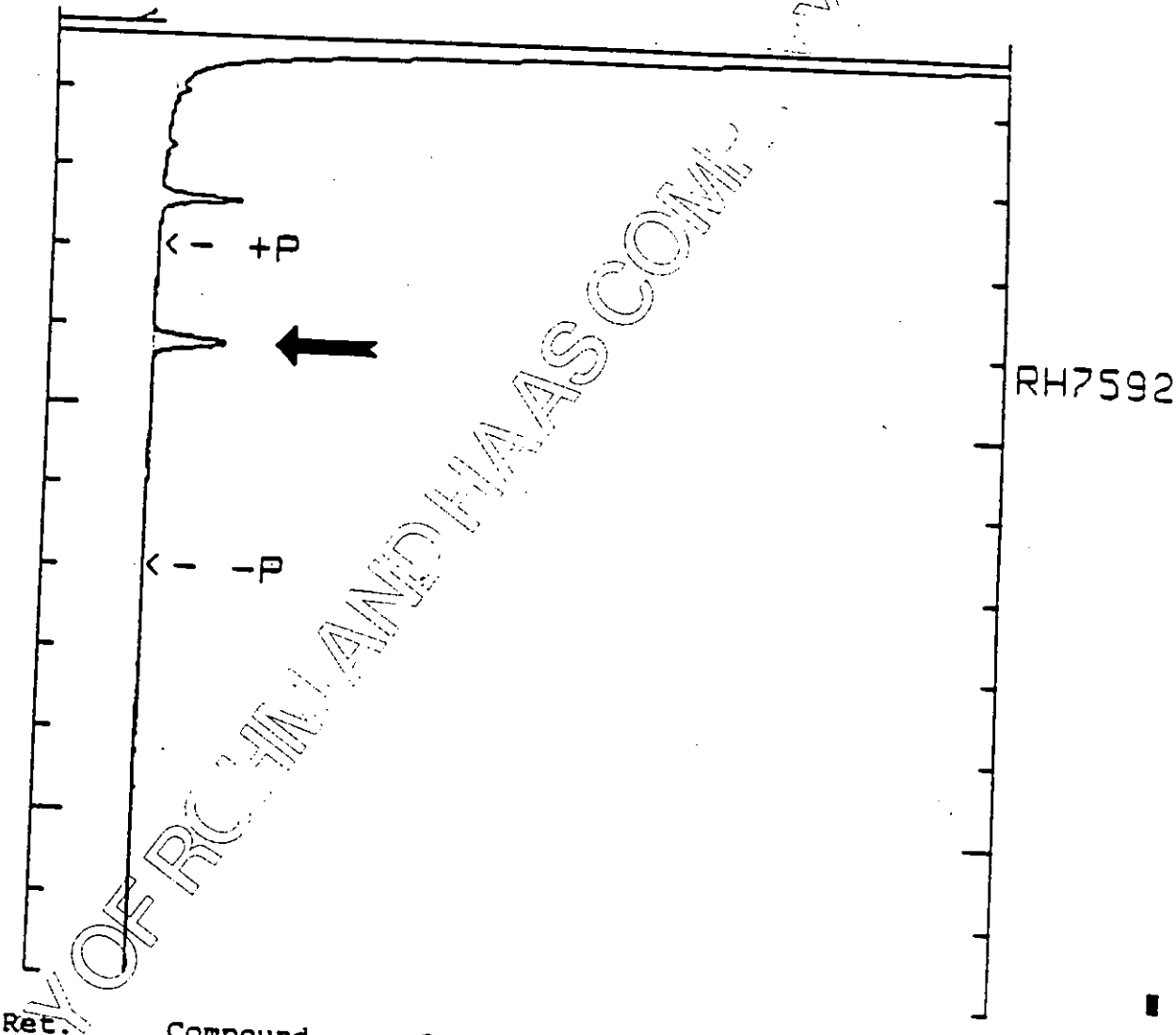
Sample Name: PEACHES
Date: 14 Sep 1988 20:17 Method: M7592m
Interface: 711 Cycle#: 12 Channel#: A

Cal. Curve: 09/14/88
Operator: TFB
Vial#: 12

000023

Instrument: VARIAN 3500(18-87)
Column: SPB-608
Column Length: 15 Meters
Start Temp-Time (deg-min): 245
Ramp Hold (deg-min):
Program Rate (deg/min):
End Time-Temp (deg-min):
Prog Slope (# or Linear):
Inj Port Temp: 255
Flowrate/Gas: 18.7
Split Ratio:
Det 1-Type & Temp: TSD/300
Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
4.20	RH7592	12.1	145.0	10.0	25.0

Figure 9

Treated Peach Sample No. 87-0170-017

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

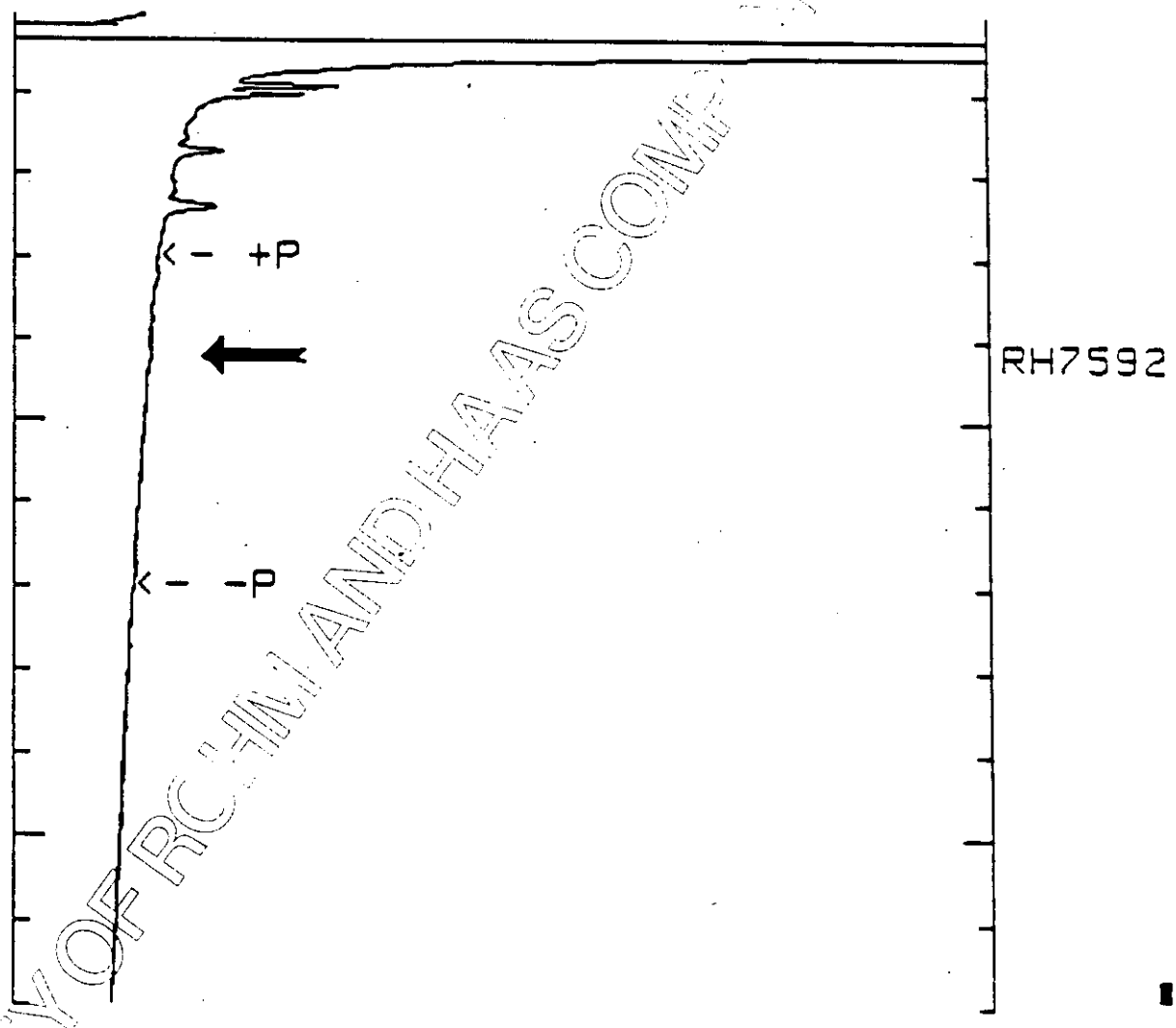
Data file: M016886 TR 34S-88-20 RAR number: 87-0333
Method file: M7592m Sample No: 025
Type: SAMPLE Component: FRUIT

Sample Name: RH7592 Cal. Curve: 09/16/88
Date: 16 Sep 1988 16:21 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 6 Channel#: A Vial#: 6

000024

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 10 millivolts (-4 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.20	RH7592	0.0	0.0	0.00	5.00	25.0	.90	0.00

Figure 10

Control Cherry Sample No. 87-0333-025

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

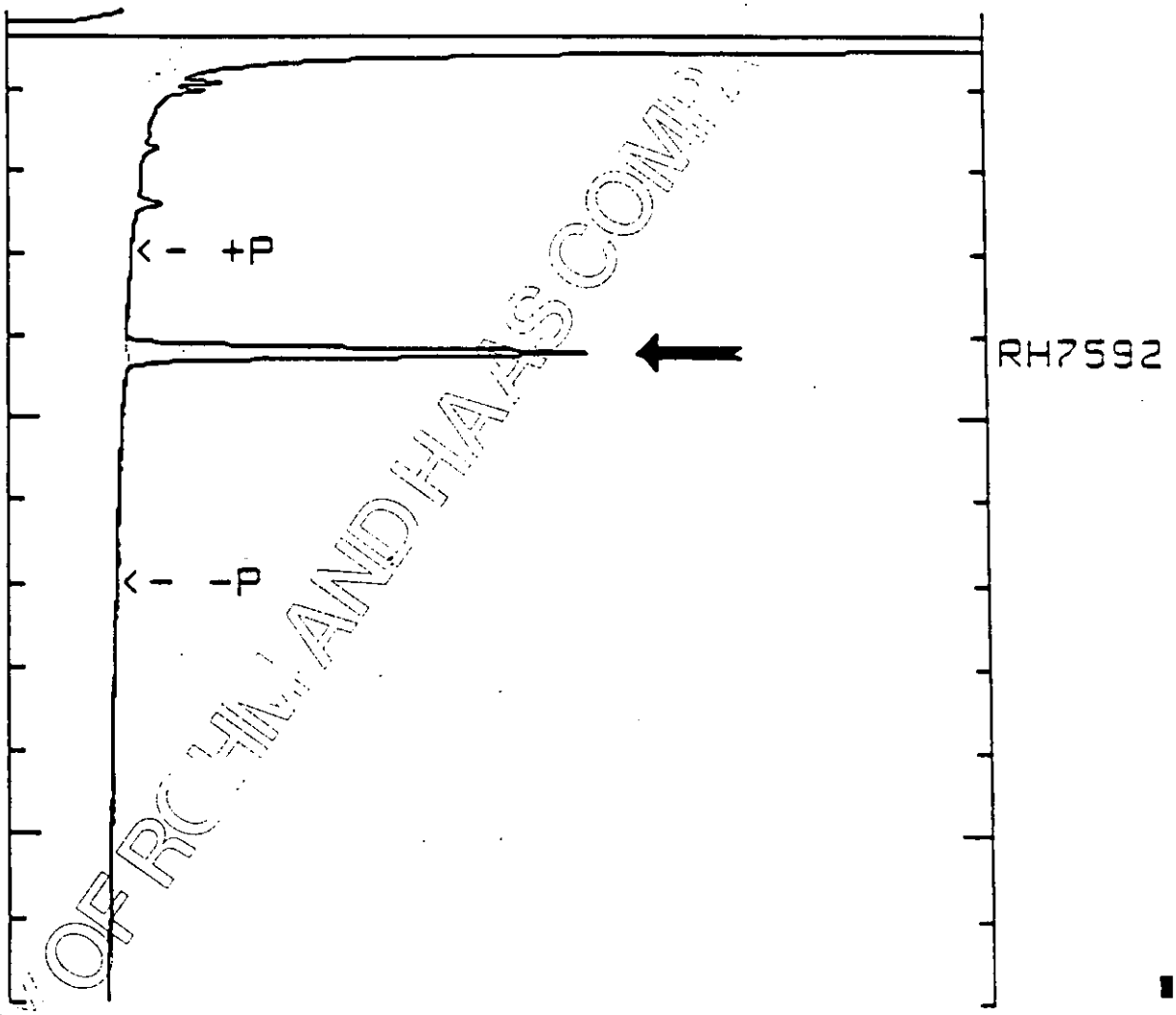
Data file: M916887 **TR 34S-88-20** RAR number: 87-0333
Method file: M7592m Sample No: 025
Type: FORTIFICATION Component: FRUIT

Sample Name: RH7592 Cal. Curve: 09/16/88
Date: 16 Sep 1988 16:38 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 7 Channel#: A Vial#: 7

000025

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 10 millivolts (-4.1 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Ctl Corr.	ug Added	Pct Recov
4.20	RH7592	37.6	469.0	0.760	15.0	0.000	12.4	92

Figure 11

Control Cherry Sample No. 87-0333-025 fortified with 0.49 ppm RH-7592

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

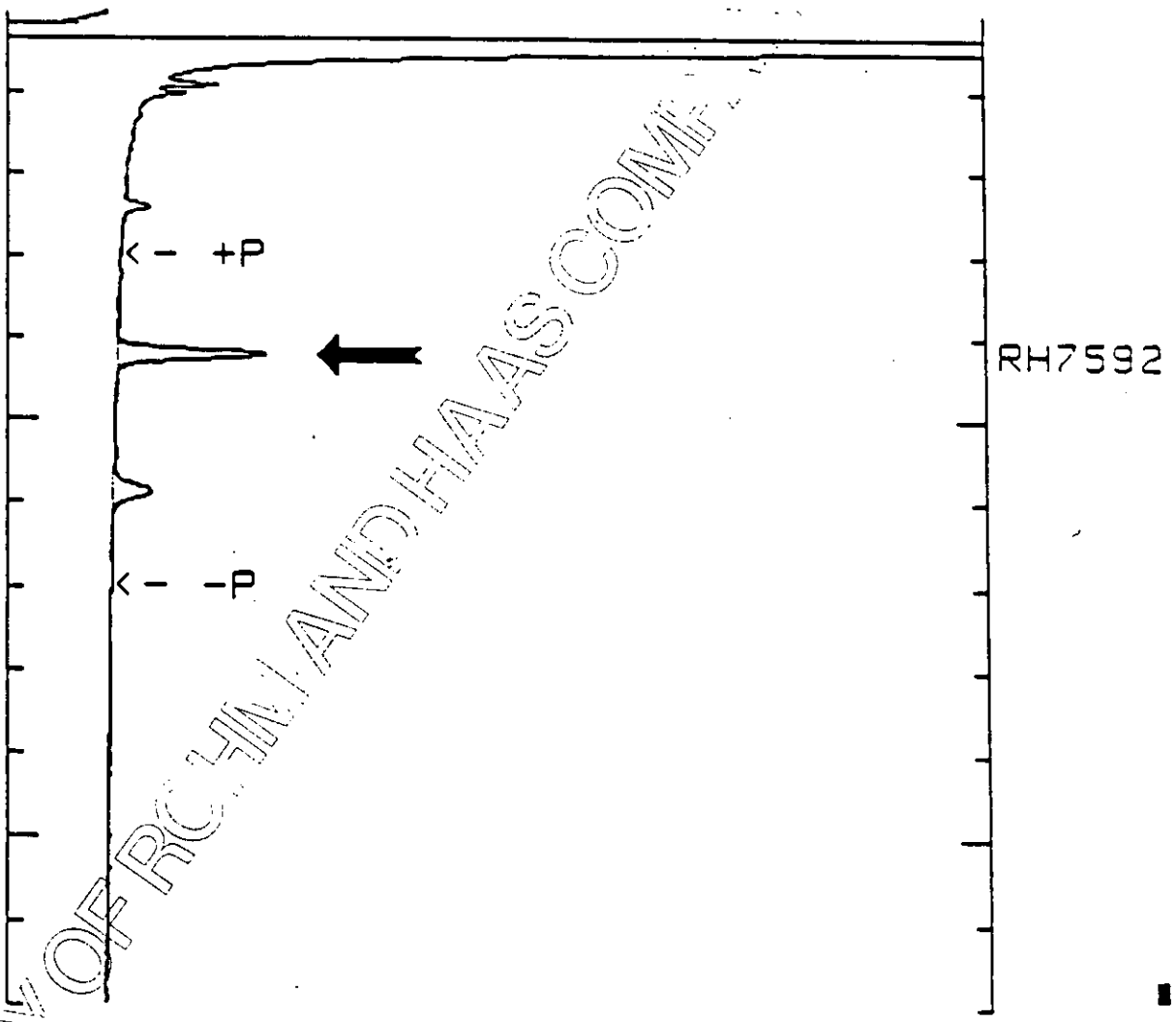
Data file: M9168810 **TR 34S-88-20** RAR number: 87-0333
Method file: M7592m Sample No: 006
Type: SAMPLE Component: FRUIT

000026

Sample Name: RH7592 Cal. Curve: 09/16/88
Date: 16 Sep 1988 17:28 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 10 Channel#: A Vial#: 10

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 10 millivolts (-4.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.21	RH7592	12.3	150.0	0.243	10.0	25.0	.90	0.108

Figure 12

Treated Cherry Sample No. 87-0333-006

PROPERTY OF RHONE-POULENC AGRO COMPANY PHILADELPHIA

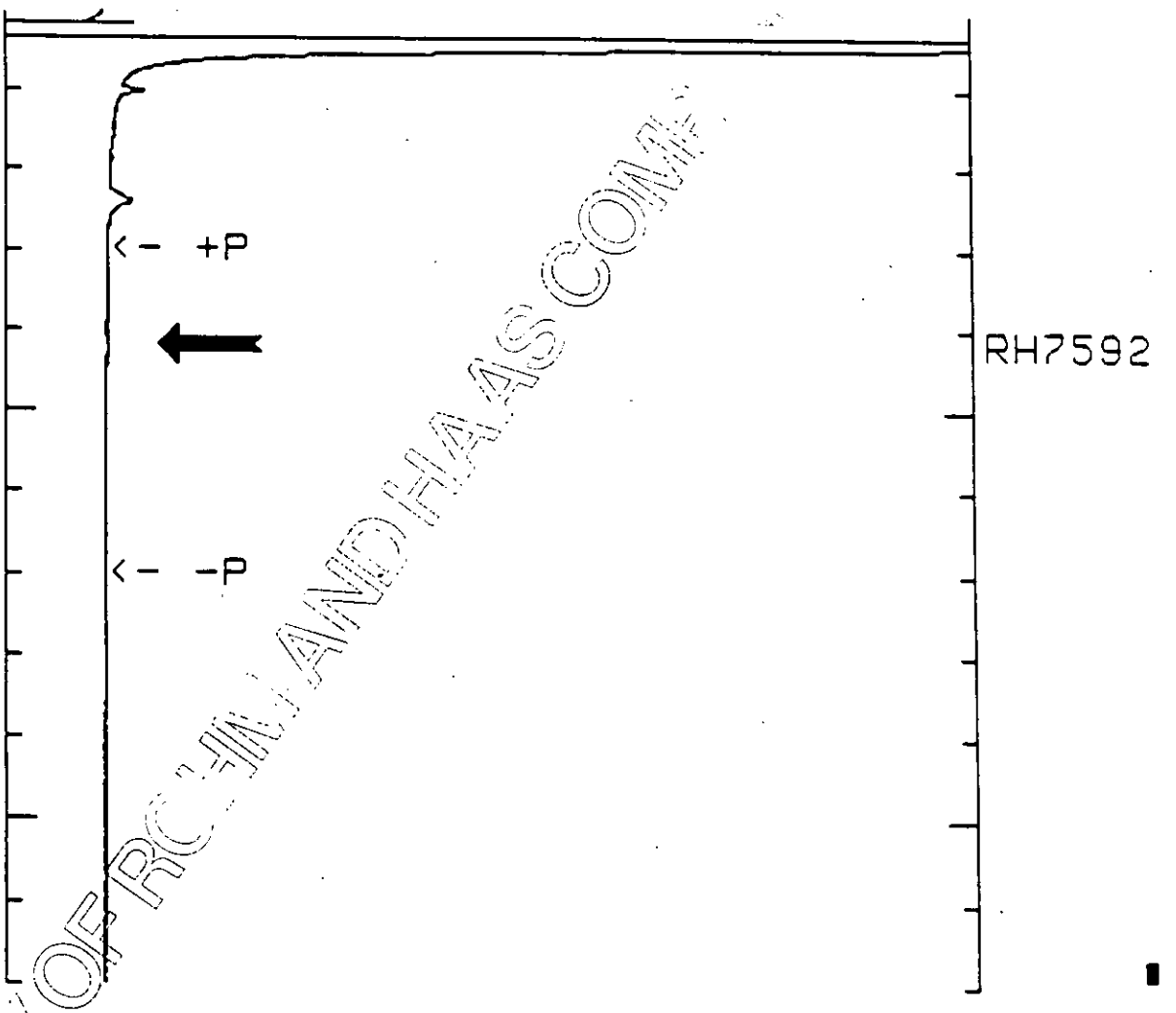
Data file: MO03886 TR 34S-88-20 RAR number: 87-0206
Method file: M7592m Sample No: 009
Type: SAMPLE Component: FRUIT

000027

Sample Name: RH7592 Cal. Curve: 10/03/88
Date: 4 Oct 1988 06:35 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 6 Channel#: A Vial#: 6

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
4.19	RH7592	0.0	0.0	5.00	25.0

Figure 13

Control Plum Sample No. 87-0206-009

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

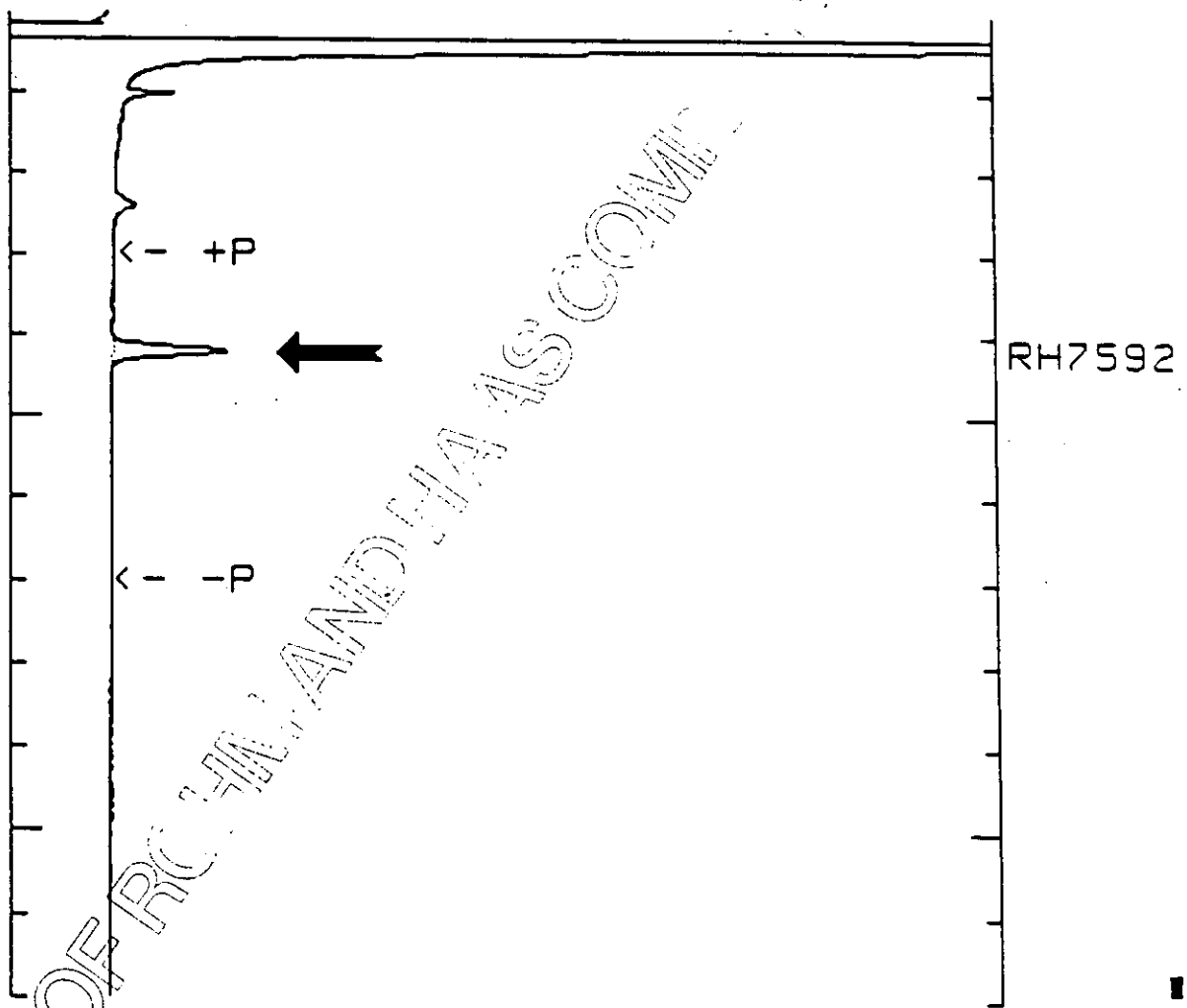
Data file: MO03887 TR 34S-88-20 RAR number: 87-0206
Method file: M7592m Sample No: 009
Type: FORTIFICATION Component: FRUIT

000028

Sample Name: RH7592 Cal. Curve: 10/03/88
Date: 4 Oct 1988 06:37 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 7 Channel#: A Vial#: 7

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	ug Added
4.20	RH7592	18.6	231.0	5.00	2.50

Figure 14

Control Plum Sample No. 87-0206-009 fortified with 0.10 ppm RH-7592

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

TR 34S-88-20

Data file: M003888
Method file: M7592m
Type: SAMPLE

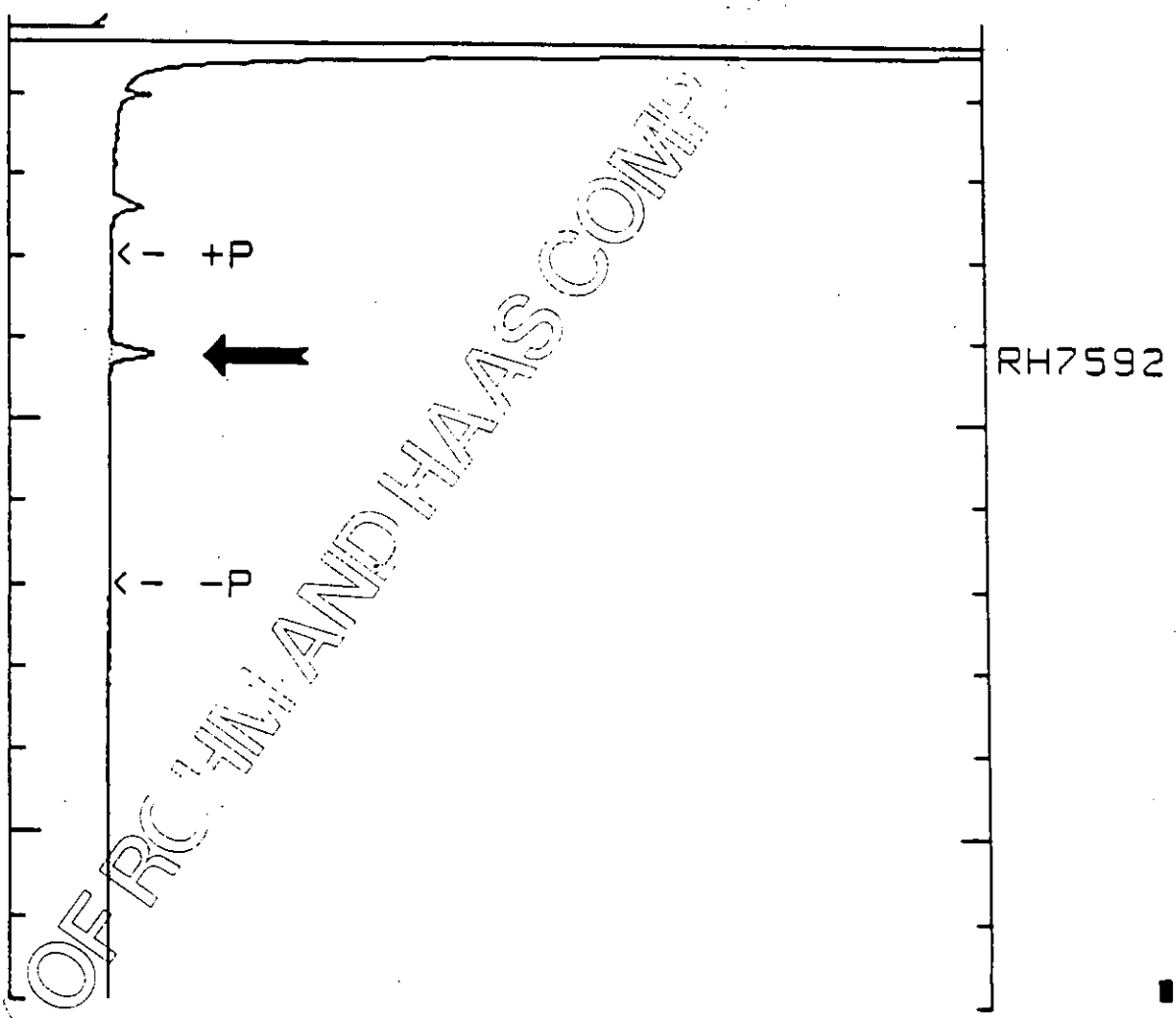
RAR number: 87-0206
Sample No: 012
Component: FRUIT

000029

Sample Name: RH7592 Cal. Curve: 10/03/88
Date: 4 Oct 1988 06:39 Method: M7592m Operator: JJM
Interface: 711 Cycle#: 8 Channel#: A Vial#: 8

Instrument: VARIAN 3500(18-87)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 255
Flowrate/Gas: 18.7 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 12 minutes
Plot range: 20 millivolts (-3.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
4.20	RH7592	7.1	87.4	5.00	25.0

Figure 15

Treated Plum Sample No. 87-0206-012

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Appendix A

Material Safety Data Sheet for RH-7592

ROHM AND HAAS COMPANY TR 34S-88-20

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

FIRE 1 REAC 0 TOXICITY 1 SPECIAL

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		

000031

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=
Insoluble	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 (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 particulate: 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.

[X] STABLE [ ] UNSTABLE | NA

HAZARDOUS DECOMPOSITION PRODUCTS
None known

ZARDOUS POLYMERIZATION
[ ] MAY OCCUR [X] WILL NOT OCCUR

CONDITIONS TO AVOID
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 cleaning 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.

OTECTIVE GLOVES
- pervious

EYE PROTECTION
Chemical splash goggles (ANSI Z87.1)

ROTECTIVE 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

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