US EPA ARCHIVE DOCUMENT

450121-11

STUDY TITLE

Tolerance Enforcement Method for Parent RH-117,281 in Cucurbits

DATA REQUIREMENT

Guideline 860.1340

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STUDY COMPLETED ON

November 30, 1999

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Rohm and Haas Technical Report No 34-99-85

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FORM 4686 REV 10/78

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).

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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 environment of United States Environmental Protection Agency FIFRA Good Laboratory Practices

The recovery data from the field residue analysis were obtained under GLP

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Quality Assurance Statement

This report and the original raw data 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.

QA activities for this study

Date	Activity	Date Reported
05/17-18/99	Audit of Preliminary Method	05/18/99
08/05/99	Radiovalidation Data Audit	08/09/99
11/17&11/30/99	Final Report Audit	11/17&11/30/99

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1. Summary

This tolerance enforcement method determines RH-7281 residues in the cucurbit vegetable group, i.e. cucumber, cantaloupe, and zucchini

RH-7281 residues are extracted from the cucurbit matrices by blending with acetonitrile The extract is partially purified by a liquid-liquid partition, a carbon Solid Phase Extraction (SPE), and an Alumina-B SPE

Quantitation is performed by gas-liquid chromatography using electron capture detection (GC/ECD) and confirmation is done by mass selective detection (GC/MSD) The limit of quantitation (LOQ) is 0 010 ppm and established by actual fortifications at this level The limit of detection (LOD) is estimated at 0.003 ppm The mean recoveries of the fortification samples, in the concentration range of 0 010 - 0.10 ppm, are as follows

Matrices	Recovery ± S.D. (%)	No. of Samples
Cucumber	89 8±10 4	25
Cantaloupe	89.9±9.82	15
Zucchini	88 4±9 14	22
Overall	89.3±9.71	62

The accountability of the method for RH-7281 in cucumber is 106% as demonstrated during radiovalidation The radiovalidation also indicates a very good agreement in the concentration of RH-7281 between the analytical method and metabolism study results

2. Introduction

RH-7281 is a fungicide recently developed at Rohm and Haas Company and targeted to be used on a variety of crops Based on the metabolism study [Ref.1], the parent is the only major residue present in cucurbit matrices. This tolerance enforcement method was developed to determine the residue of the parent compound in cucurbits to establish tolerances for registration of the compound

The report compiles data from the preliminary analytical method [Ref 2], data from residue trials [Ref 3], and data from radiovalidation which was conducted during the method development.

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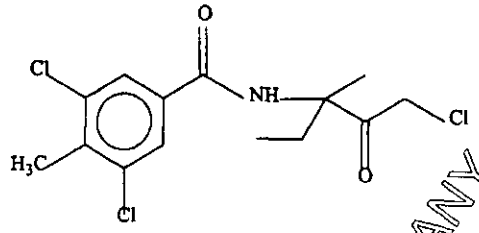
3. Experimental Compound

The structure of RH-7281 is shown below.



CAS Name 3,-5-dichloro-N-(3-chloro-1-ethyl-1-methyl-2-oxopropyl)-4-methylbenzamide

CAS Number: 156052-68-5

Proposed Generic Name. zoxamide

R&H I.D.	Lot #	Purity	Appearance	Expiration Date
RH-117281*	ELM1157	97.9%	White Solid	Nov. 11, 2001

*The full R&H number is RH-117281; the designation typically used in studies is the four digit abbreviation RH-7281

4 Chemicals/Prepared Solutions

Chemicals	Grade	Supplier*
Acetonitrile	HPLC	Baker
Alumina B Cartridge	LC-Alumina B (0.5 g, 6 mL)	Supelco Cat # 57085
Anhydrous Sodium Sulfate	Analytical	Fisher Chemical
Carbon Cartridge	ENVI-Carb (0.5 g, 6 mL)	Supelco Cat #.57094
Ethyl Acetate	HPLC	Baker

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Hexane	HPLC	Baker
Sodium Chloride (NaCl)	A C S. Reagent	Baker
Water	Mill-Q	Millipore System

* Other manufacturer brands may be substituted if shown to be suitable

Prepared Solutions

0 1N NaCl solution was prepared by dissolving 5 84 g of NaCl into 1000 mL of Mill-Q water

5/95 (v/v) ethyl acetate/hexane solution was prepared by mixing 50 mL of ethyl acetate and 950 mL of hexane in a 1000 mL flat bottom flask

30/70 (v/v) ethyl acetate/hexane solution was prepared by mixing 300 mL of ethyl acetate and 700 mL of hexane in a 1000 mL flat bottom flask

5. Equipment

Equipment	Description	Source*
Blender	Model 31BL91	Waring Comm.
Buchner Funnel	200 mL	Coors
Filter Flask	500 mL	Pyrex
Filter Paper	# 3	Whatman
Flat Bottom Flask, 24/40 ST	1000 mL	Fisher Scientific
Rotary Evaporator	Model R114	Brinkman
Round Bottom Flasks, 24/40 ST	250 mL and 500 mL	Fisher Scientific
Separatory Funnels	500 mL	Fisher Scientific
Sonicator	Model 1510	Branson
Visprep SPE Vacuum Manifold	Manifold	Supelco

Standard laboratory equipment, balance, beakers, etc

* Other manufacturer brands may be substituted if shown to be suitable.

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6. Method

6 1 Method Flow Diagram

10 g Cucurbit Sample

↓

Extraction (acetonitrile, blending)

↓

Liquid-Liquid Partition (EtOAc/0.1N NaCl)

↓

Carbon SPE

↓

Alumina B SPE

↓

GC/ECD or GC/MSD Quantitation

6 2 Sample Preparation

Prepare frozen cucurbits, i.e cucumber, cantaloupe, and zucchini, by chopping the samples in a food chopper with dry ice. Allow the dry ice to sublime overnight in a freezer. Store the processed samples in a freezer prior to use

6.3 Sample Extraction

Weigh 10 g of homogenized cucurbit sample in a weighing dish and transfer to a blender jar. At this time, spike fortification samples with a known amount of RH-7281 in hexane Allow the solvent to evaporate in a fume hood for about 30 minutes until the sample is free of solvent

Add 100 mL acetonitrile to the blender jar (the blender can be substituted with a tissuemizer) Blend the slurry for one minute at a medium speed. Filter the slurry through a Buchner funnel mounted with a #3 Whatman filter paper into a 500 mL filtration flask. Rinse the blender and the filter cake with 50 mL acetonitrile Combine the filtrates (~150 mL) in a 500 mL round bottom flask and concentrate to about 10 mL on a rotovap at about 40 °C under diminished pressure (about 50 mm Hg)

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6.4 Liquid-Liquid Partition

Add the extract to a 500 mL separatory funnel containing 100 mL of 0.1N NaCl aq. solution. Rinse the flask with ~5 mL of water and 100 mL of ethyl acetate and add all rinsates to the separatory funnel. Shake the funnel for one minute with frequent venting and allow two phases to separate. Drain the aqueous phase to the 500 mL round bottom flask Transfer the organic phase to a clean 500 mL round bottom flask Partition the aqueous phase with another 50 mL of ethyl acetate in the same manner Discard the aqueous phase to waste Combine both organic phases to the 500 mL round bottom flask and concentrate to dryness on a rotovap at about 40°C under diminished pressure (about 50 mm Hg) Dissolve the residues with 10 mL of hexane with sonication for 10 seconds

6.5 Carbon Solid Phase Extraction

Top the carbon cartridge with about 0.5 cm of anhydrous sodium sulfate and rinse the cartridge with 5 mL of 5/95 (v/v) ethyl acetate/hexane Discard the effluent at this stage Apply the 10 mL of residues in the 500 mL flask to the cartridge. Rinse the 500 mL round bottom flask with 30 mL of 5/95 (v/v) ethyl acetate/hexane and apply the rinsate to the cartridge Collect the eluate and transfer it to a 250 mL round bottom flask Note: RH-7281 is eluted from the cartridge with 5/95 (v/v) ethyl acetate/hexane. The elution profile should be checked for each new lot of carbon cartridge and minimum volume of elution solution used to elute RH-7281 from the cartridge See Note on page 13 for the elution profile check.

Concentrate the eluate to dryness on a rotovap at about 40°C under diminished pressure (about 50 mm Hg). Dissolve the residue with 10 mL of hexane with sonication for 10 seconds

6.6 Alumina-B Solid Phase Extraction

Wash the Alumina-B cartridge with 5 mL of ethyl acetate and then 10 mL of hexane. Apply the 10 mL of residue in the 250 mL round bottom flask to the cartridge Rinse the flask with 15 mL of 5/95 (v/v) ethyl acetate/hexane and apply the rinsates to the cartridge. Discard the effluent at this stage. Elute RH-7281 from the cartridge with 40 mL of 30/70 (v/v) ethyl acetate/hexane and transfer the eluate to a clean 250 mL round bottom flask Note. The elution profile should be checked for each new lot of Alumina-B cartridge and the minimum volume of elution solution used to elute RH-7281 from the cartridge See Note on page 13 for the elution profile check.

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Concentrate the eluate to dryness on a rotovap at about 40°C under diminished pressure (about 50 m Hg). Dissolve the residue in 10 mL of hexane with sonication for 10 seconds

Note.

The analyte(s) elution profile should be checked for each new lot of carbon and Alumina-B Minimum volume of solvent should be used to elute most of the target analyte from the cartridge An example procedure for testing the elution profile for a new lot of Alumina-B is as follows

1) Using 10 mL of 0.10 µg/mL RH-7281 standard, follow the elution schemes as outlined under section 6 6

2) Collect the pre-elution cuts as well as the target elution cut Wash the column with 30 mL of ethyl acetate and collect this post-elution cut

3) Concentrate the pre-elution cuts, the target cut, and the post-elution cut to dryness

4) Dissolve the cuts in 10 mL of hexane and inject them as outlined in the GC/ECD quantitation section

5) If the target cut contains a minimum of 85% of RH-7281, it may be considered acceptable

6) If the 85% of recovery criteria for standard is not met, the following adjustment in elution scheme should be made:

a) For case where the pre-wash cut contains significant standard, either the pre-wash amount or eluting solvent percentage may be decreased.

b). For cases where the post-wash cut contains significant standard, the percentage of the more polar component in the eluting solvent may be increased

7. Quantitation

Quantitation can be done with either GC/ECD or GC/MSD depending on the availability of instrumentation.

7.1 Instrumentation and Conditions

Gas Chromatography/Electron Capture Detector

GC/ECD HP 6890/Micro-ECD

Any other suitable GC/ECD system may be used for analysis after verifying system suitability.

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Injector	HP 6890 Auto-Sampler	
	On-column mode	
	Injection temp.:	150°C
	Injection volume:	1 0 µL
Column	Rtx-5 (0.25mm I D x 30 m; 0 25µm film)	
	Carrier gas:	Helium
	Head Pressure	13 5 psi
	Flow rate	1.0 mL/min
Detector	Temperature	300°C
	Make Up Gas:	Nitrogen
	Total Flow	60 mL/min.

Oven temperature program
Initial temp. = 80°C, hold for 1 min.
Ramp at 20°C/min to 220°C, hold for 5 min
Ramp at 30°C/min. to 280°C, hold for 3 min

Typical Retention Time.

RH-7281: ~12 min.

Gas Chromatography/Mass Selective Detector

GC/MSD HP6890/5973 MSD

Any other suitable GC/MSD system may be used for analysis after verifying system suitability.

Injector:	HP6890 Auto-Sampler	
	Splitless mode (purge time = 1.0 min.)	
	Injection temp :	180°C
	Injection volume	1 0 µL
Column.	HP-MS5 (0.25mm I D. x 30 m; 0 25 µm film)	
	Carrier gas:	Helium
	Head Pressure:	10 4 psi
	Flow rate	1 0 mL/min.

Oven temperature program:
Initial temp. = 100 °C, hold for 1 min
Ramp at 15 °C/min to 250 °C, hold for 5 min

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Ramp at 30 °C/min. to 280 °C, hold for 3 min.

SIM m/z 187, 189, 258 (quantitation ion)

Typical Retention Time:

RH-7281 10-13 min.

7 2 Preparation of Standard Curves

7 2.1 Stock Standard Solution

Weigh about 10 mg of RH-7281 standard on an analytical balance and correct for purity. Transfer the material into a 100 mL volumetric flask and add adequate amount of ethyl acetate to make a 100 µg/mL solution Store the stock solution in the refrigerator. Shelf life of the 100 ppm stock solution is 6 months (see reference [4] for details).

7.2 2 Intermediate Standard Solution

Prepare a 1.0 µg/mL intermediate standard solution by diluting 1 0 mL of the 100 µg/mL stock solution to 100 mL in a volumetric flask with hexane Store the solution a refrigerator. Shelf life of the solution is 6 months

7 2.3 Working Standard Solution

Prepare working standard solutions by diluting the intermediate standard solution with hexane

Concentration	Aliquot size and source	Final Volume
0.10 µg/mL	10 mL of 1 0 µg/mL	100 mL
0.050 µg/mL	5 0 mL of 1 0 µg/mL	100 mL
0.020 µg/mL	2 0 mL of 1.0 µg/mL	100 mL
0 010 µg/mL	1.0 mL of 1 0 µg/mL	100 mL

Store working standard solutions in a refrigerator and prepare fresh every six months or when they are needed.

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7.2.4. Spiking Standard Solution

Use 0.10 µg/mL and 1.0 µg/mL standard solutions to fortify 10 g of cucurbit samples using the following procedure

Fortification (µg/g)	Solution (µg/mL)	Volume Taken (mL)
0 010	0 1	1 0
0 020	0 1	2 0
0 050	1 0	0 50
0 10	1.0	1 0

7 3 Preparation of Standard Curves

A minimum of four standard solutions are prepared in a desired concentration range Standards and samples are preferably quantitated by peak area, although height may be used A linear regression is used to fit the instrument response into the equation $Y = mX + b$ A quadratic regression can also be used $Y = nX^2 + mX + b$. The concentrations of RH-7281 in samples are then determined from the standard curves

7 4 Fortification Recovery

Control samples are fortified with a known amount of RH-7281 prior to extraction Percent recovery is calculated by measuring the peak area or peak height, calculating the µg/mL found from the standard curves, and correcting any background (if the residue level is ≥ LOQ) from the control sample as shown in Equation 1.

$$\frac{\text{Found (µg/mL)} \times \text{Final Vol.(mL)}}{\text{Fortification Amount (µg)}} \times 100\% = \%\ \text{Recovery} \qquad \text{Eq.1}$$

7 5 Sample Analysis

Injection volumes are the same for samples and standards If necessary, the samples are diluted to an appropriate volume to give a final concentration within the standard curve range.

The residue concentration in ppm is determined as follows.

$$\frac{\text{Concentration (µg/mL)} \times \text{Final Volume (mL)}}{\text{Sample Weight (g)}} = \text{µg/g} = \text{ppm} \qquad \text{Eq 2}$$

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7.6 Sample Calculation

A typical calculation using Eq.1 for the recovery of 0 10 ppm fortification of a cucumber sample (Table 1 and Figure 12) is demonstrated as follows

$$\frac{0.0840\ (\mu g/mL) \times 10\ (mL)}{1\ 0\ \mu g} \times 100\% = 84\ 0\%$$

The residue concentration in ppm is then determined using Eq 2 as follows

$$\frac{0.0840\ (\mu g/mL) \times 10\ (mL)}{10\ g} = 0\ 0840\ \mu g/g = 0\ 0840\ ppm$$

7 7 Run Time of a Set of Samples

The time required to run a set of 6 samples is about 18 hours including data acquisition

8. Radiovalidation

8 1 Sample Preparation

Cucumber samples (RAR. 98-0280-002) used in the radiovalidation were obtained from Grayson Research facilities in North Carolina [Ref. 1], where the field portion of the metabolism study was conducted. The control cucumber sample (RARNo. 98-0280-001) was also collected from the same site The cucumber samples were processed and stored frozen prior to use.

8.2 Sample Analysis

Sample extraction, clean up, and quantitation by GC/ECD were conducted by following the preliminary analytical method [Ref. 2].

The Liquid Scintillation Counting (LSC) procedures were conducted according to RMEF SOP RES.10 01 as follows.

Prior to analysis, weigh replicates from different areas (sample size ~400 mg) of the C^{14} sample and control replicates of the control for combustion to determine Total Radioactive Residue (TRR).

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At extraction, measure the total volume for each control and C^{14} sample replicates and then take 1.0 mL of aliquot in triplicate from the control and C^{14} replicates for liquid scintillation counting. Air dry the filter cakes and collect the solids that remain after extraction Weigh the total weight and take replicates for combustion

After Alumina-B SPE, measure the total volume for each control and C^{14} sample replicates and take triplicate of 1 0 mL aliquots for liquid scintillation counting

9 Results and Discussion

9 1 The Analytical Method

Both GC/ECD and GC/MSD quantitation have acceptable sensitivity for RH-7281 and generate a linear response to RH-7281 in the concentration range of 0 010 - 0.10 µg/mL (Figure 1 to Figure 5 and Figure 45 to Figure 49)

The limit of quantitation (LOQ) for cucurbit samples is established at 0 010 ppm by analysis of fortifications at that target level (Table 1). LOD is estimated at 0 003 ppm.

No significant interference was found at the retention time of RH-7281 in all three matrices, using a 30 m Rtx-5 column (Figures 6, 19, and 32).

The mean recovery of RH-7281 for fortified samples is 89.3±9 71% (n=62, Table 1) by GC/ECD.

GC/ECD is recommended as the primary analytical method because of its high sensitivity (attributable to the three chlorines in the compound). Confirmation of the detection is demonstrated by GC/MSD detection monitoring three typical ions, i.e. 187, 189, and 258 of the compound.

The GC inlet system must be clean and conditioned to minimize the degradation of RH-7281 A temperature program, e g increasing injection temperature to 30°C below the maximum column bleeding temperature for one minute after the elution of the parent peak, is found effective to remove the impurities that remain in the inlet system Removal of 0 5 m of the analytical column, sonicating of the insert in methanol for 5 minutes, and replacing the septa after about 100 injections were also effective to keep the system clean

If poor chromatography still persists after these changes, a new analytical column and a new insert may be required.

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9 2 Radiovalidation

The results from radiovalidation are compared with that of the metabolism study (Table 2) in order to demonstrate whether the residues of RH-7281 presented in the samples from the metabolism study were adequately measured

At the combustion step, the initial material used in the analytical method shows an average activity of 61,600 dpm/g . It matches very well with 60,089 dpm/g from the metabolism study

At the extraction step, the analytical method demonstrates a 106% in extraction efficiency comparing with 119% from the metabolism study.

At the final quantitation step, the analytical method determines the final concentration of RH-7281 to be 1 28 µg/g This result matches very well with the 1 33 µg/g RH-7281 from the metabolism study.

Since there is a good agreement between two studies in the concentration of the parent RH-7281, the radiovalidation is claimed successful and validity of the analytical method is proven.

10. References

[1] TR 34-99-57, "^{14}C RH-117,281: Nature of the Residue in Cucumbers" Ashok Sharma, Concurrent Submission.

[2] TR 34-99-43, "Preliminary Analytical Method for RH-7281 in Cucurbits" Ipin Guo, Ernest Burdge, Ken Kurilla, Charles Hofmann, May 21, 1999

[3] TR 34-99-160, "RH-117,281 Fungicide Field Residue Trials in Cucurbit Vegetable Group". Deborah Graves, Concurrent Submission

[4] LM 34-99-12, "Storage Stability of the Standard Solutions RH-7281, RH-0721, RH-1452, RH-1455, RH-7391, and RH-7399" Ipin Guo, March 18, 1999

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11. Project Information

Title	Tolerance Enforcement Method for Parent RH-7281 in Cucurbits
Project Number.	1401.15
Sponsor	Rohm and Haas Company Research Laboratories 727 Norristown Road Spring House, PA 19477-0904
Dates	Work Initiated: January, 1999 Work Completed: November, 1999
Technical Personnel.	Ipin Guo, Ernie Burdge, Ken Kurilla, Charles Hofmann
Note Book.	RH-IG-003 (#68298)
Peer Reviewer	Dennis Martin

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Table 1 Fortification Recovery Results

Fortification (µg/g)	Reference	Recovery, % GC/ECD (MSD)	Date mm/dd/yy
LOQ			
0 010 (Cucumber)	TR 34-99-43	90 0	03/12/99
0 010	TR 34-99-43	93 6	03/12/99
0 010	TR 34-99-43	105	03/17/99
0 010	TR 34-99-43	98 3	03/17/99
0 010	TR 34-99-43	90 8	03/22/99
0 010	TR 34-99-43	94 3	03/22/99
0 010	TR 34-99-43	88 2 (67.2)	04/26/99
0 010	TR 34-99-43	83.6 (105)	04/26/99
0 010	TR 34-99-160	122	09/12/99
0.010 (Cantaloupe)	TR 34-99-43	92 0	03/01/99
0.010	TR 34-99-43	93 4	03/01/99
0 010	TR 34-99-43	73 0 (74 2)	03/26/99
0 010	TR 34-99-43	99 2 (97.3)	03/26/99
0.010	TR 34-99-160	83 4	06/07/99
0 010 (Zucchini)	TR 34-99-43	83.9	03/03/99
0 010	TR 34-99-43	82 9	03/03/99
0 010	TR 34-99-43	100 (88 2)	04/05/99
0 010	TR 34-99-43	93 0 (98 7)	04/05/99
0 010	TR 34-99-160	107	08/10/99
0 010	TR 34-99-160	82 8	08/25/99
0 010	TR 34-99-160	90.2	08/26/99
LOQ Mean ± Standard Deviation		92.7±10.5 (88.5±14.9)	
LOQ Number of Samples		21 (6)	

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Table 1 Fortification Recovery Results (cont'd)

Fortification (µg/g)	Reference	Recovery, % GC/ECD (MSD)	Date mm/dd/yy
0.02-0.10ppm			
0.020 (Cucumber)	TR 34-99-43	98 4	03/12/99
0 020	TR 34-99-43	92 3	03/17/99
0 020	TR 34-99-43	75 2	03/22/99
0 020	TR 34-99-43	85.1 (97.8)	04/26/99
0 020	TR 34-99-160	76 0	09/17/99
0 020	TR 34-99-160	98 0	09/20/99
0 020 (Cantaloupe)	TR 34-99-43	85.0	03/01/99
0 020	TR 34-99-43	82.7 (77 8)	03/26/99
0 020	TR 34-99-160	115	07/23/99
0 020 (Zucchini)	TR 34-99-43	86.0	03/03/99
0 020	TR 34-99-43	100 (78 1)	04/05/99
0 020	TR 34-99-160	80 7	08/04/99
0.020	TR 34-99-160	77 0	10/04/99
0 020	TR 34-99-160	96.0	10/12/99
0 050 (Cucumber)	TR 34-99-43	87 9	03/12/99
0 050	TR 34-99-43	75.1	03/17/99
0.050	TR 34-99-43	88 1	03/22/99
0.050	TR 34-99-43	101 (79.4)	04/26/99
0 050	TR 34-99-160	82.5	09/17/99
0 050 (Cantaloupe)	TR 34-99-43	94 4	03/01/99
0 050	TR 34-99-43	84 5 (104)	03/26/99
0 050	TR 34-99-160	79 1	06/07/99
0 050 (Zucchini)	TR 34-99-43	89.3	03/03/99
0 050	TR 34-99-43	96 0 (76.4)	04/05/99
0 050	TR 34-99-160	103	08/10/99
0 050	TR 34-99-160	85 3	08/26/99
0 050	TR 34-99-160	78.9	09/10/99
0 10 (Cucumber)	TR 34-99-43	88 9	03/12/99
0.10	TR 34-99-43	82.9	03/17/99
0 10	TR 34-99-43	84 0	03/22/99
0 10	TR 34-99-43	81 6 (88.7)	04/26/99
0 10	TR 34-99-160	81 4	09/12/99
0 10 (Cantaloupe)	TR 34-99-43	95 4	03/01/99
0 10	TR 34-99-43	93 6 (91.3)	03/26/99
0.10	TR 34-99-160	89.2	07/15/99
0.10	TR 34-99-160	88 1	07/23/99
0.10 (Zucchini)	TR 34-99-43	94 9	03/03/99
0 10	TR 34-99-43	79 9 (85 3)	04/05/99
0.10	TR 34-99-160	76.4	08/04/99
0.10	TR 34-99-160	83 5	08/25/99
0 10	TR 34-99-160	77 2	10/04/99
Total Mean ± Standard Deviation		89.3±9.71 (87.3±11 6)	
Total Number of Samples		62 (15)	

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Table 2 Radiovalidation Results

LSC Results

Fraction	Metabolism[a] (dpm/g)	The Method (dpm/g)
Combustion		
Initial Material	60089±27,600 (n=8)	61600±20100 (n=10)
TRR (µg/g)[b]	1.53±0 70	1 57±0 51
Extraction		
Extract	71235	65000±6823 (n=3)
PES	0	1130±36 (n=3)
Extraction Efficiency	119%	106%
Quantitation		
Final Solution	69620	54133±2159 (n=3)
TRR%	116%	87.9%

TLC and GC Results

Compounds	Metabolism *(TLC, µg/g)*	The Method *(GC, µg/g)*
RH-7281	1 33±0 61	1.28±0.16 (n=3)

[a] Data cited from [Ref. 1]
[b] Specific Activity = 39,274 dpm/µg

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Appendix Chromatograms

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\2\DATA\032299\STDA1 D
Operator      IG
Acquired    : 3-22-99 17 34 50  using AcqMethod RH7281.M
Instrument      GC ECD
Sample Name   std 0 01ppm
Misc Info     0.01 ppm 7281 std
Vial Number   2
```

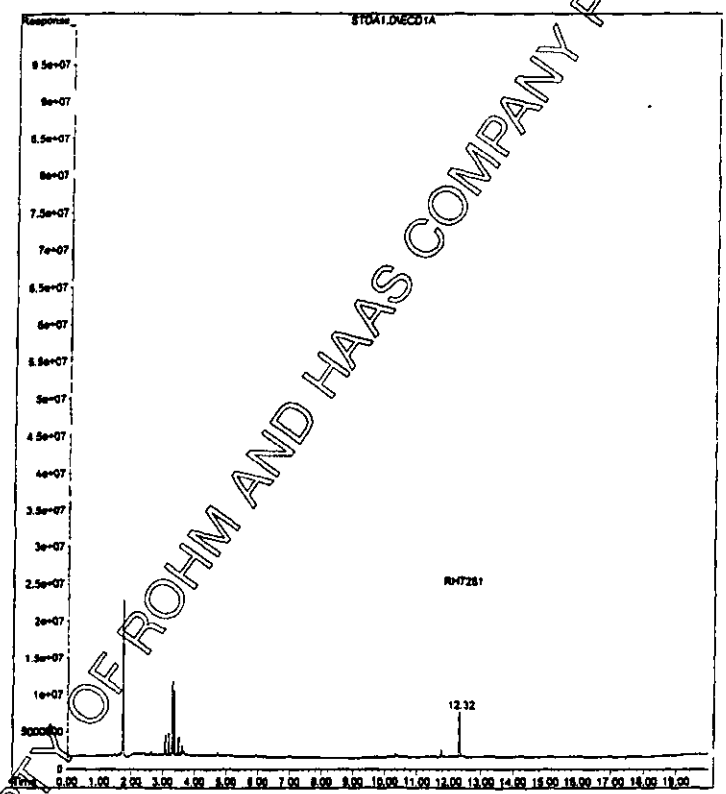


Figure 1 0 010 µg/mL Standard RH-7281 (GC/ECD, Peak Area = 123500669)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032299\STDA2 D
Operator      IG
Acquired      3-22-99 18 00:12  using AcqMethod RH7281 M
Instrument .    GC ECD
Sample Name   std 0 02ppm
Misc Info     0 02 ppm 7281 std
Vial Number   3
```

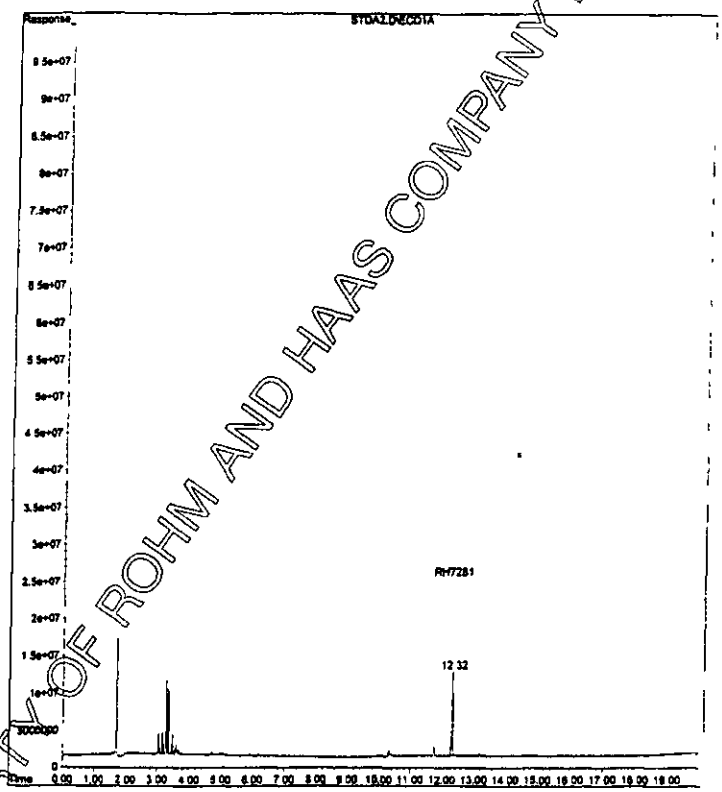


Figure 2 0 020 µg/mL Standard RH-7281 (GC/ECD, Peak Area = 224743915).

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        . C:\HPCHEM\2\DATA\032299\STDAJ D
Operator    . IG
Acquired    : 3-22-99 18:25:32  using AcqMethod RH7281 M
Instrument    GC ECD
Sample Name   std 0 05ppm
Misc Info     0 05 ppm 7281 std
Vial Number   4
```

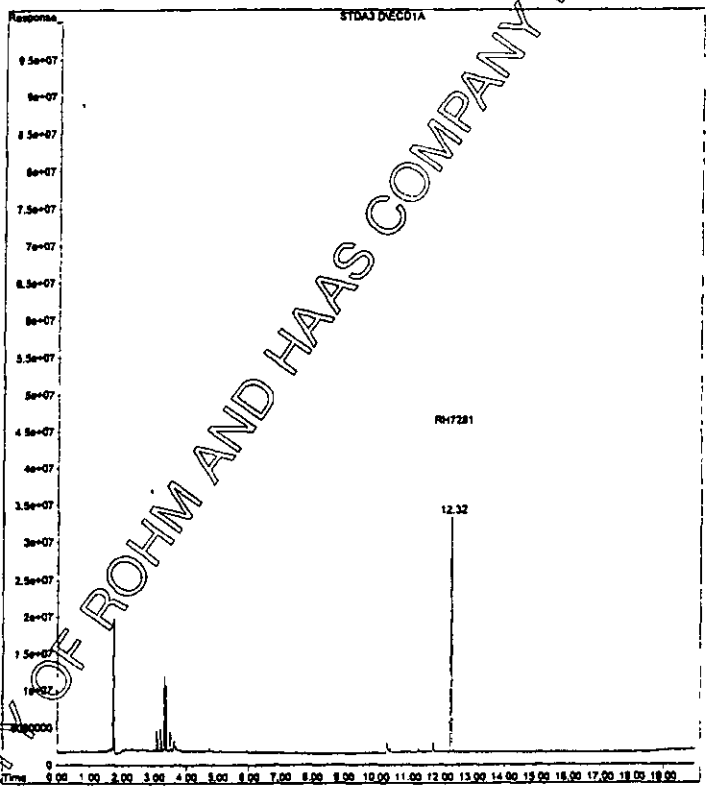


Figure 3 0 050 µg/mL Standard RH-7281 (GC/ECD, Peak Area = 589528318)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032299\STDA4 D
Operator      IG
Acquired      3-22-99 18 50 55  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   std 0.10ppm
Misc Info     0 10 ppm 7281 std
Vial Number   5
```

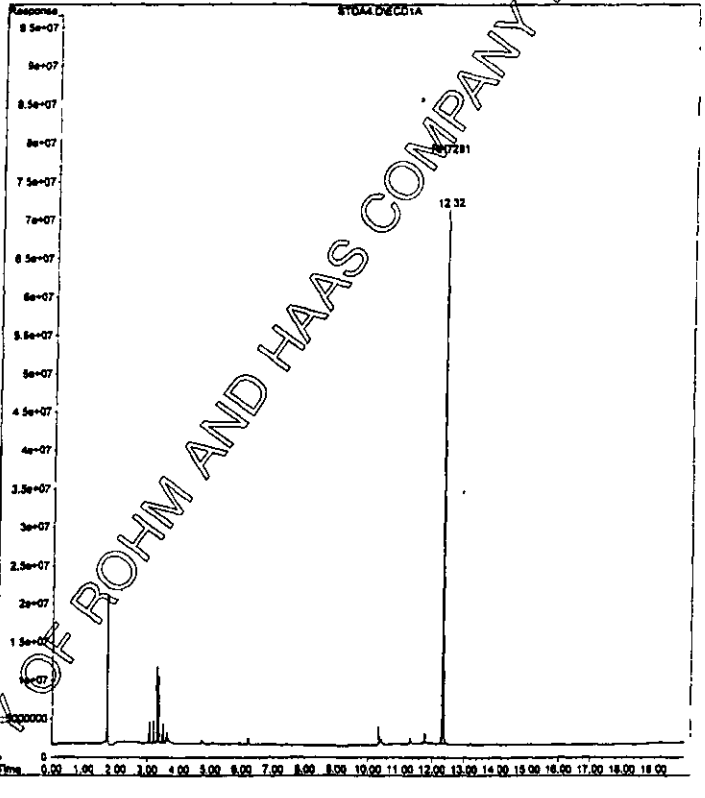
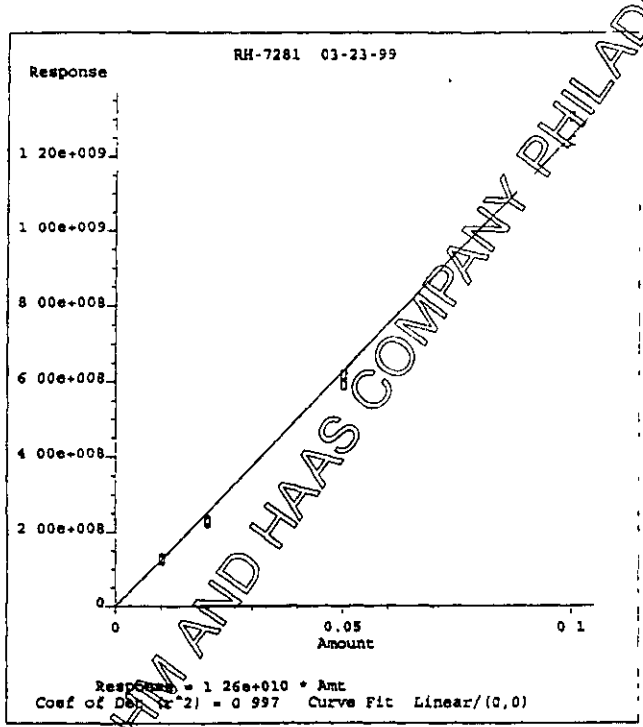


Figure 4 0 10 µg/mL Standard RH-7281 (GC/ECD, Peak Area = 1242287208)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA



Method Name C.\HPCHEM\2\METHODS\RH7281 M
Calibration Table Last Updated Tue Mar 23 09 20·01 1999

Figure 5 Calibration Curve for RH-7281 (GC/ECD, 03-23-1999)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032299\CONTROL D
Operator      IG
Acquired      3-22-99 19 41 36  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   control, cucumber
Misc Info     Cucumber control
Vial Number   6
```

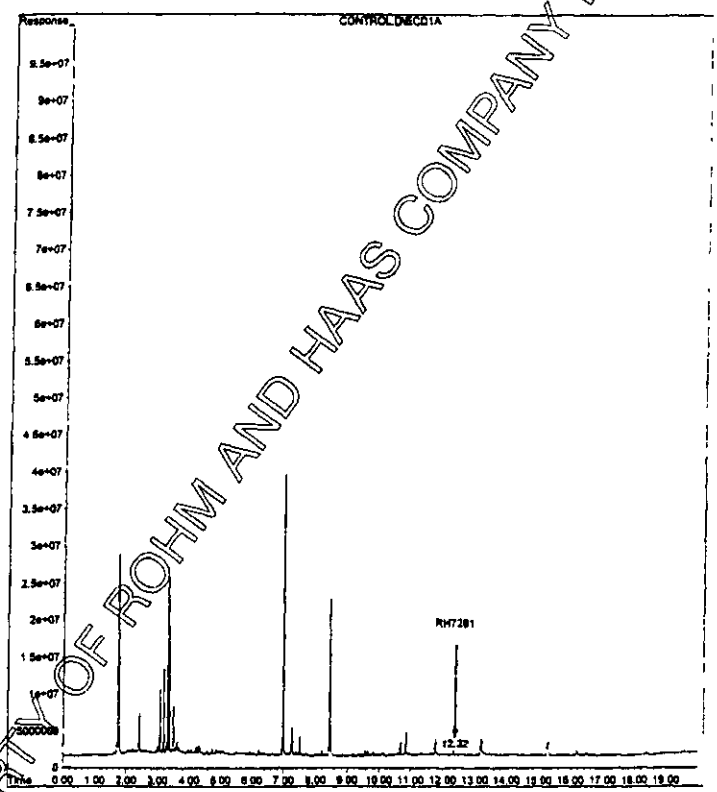


Figure 6 Control Cucumber (GC/ECD, TR 34-99-43, Peak Area = 0).

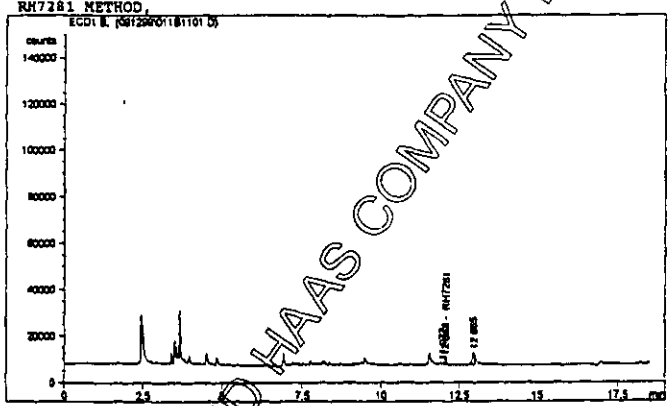
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091299\011B1101.D Sample Name: 98-0081-1 Cntr

RAR 98-0081-1 Control Cucumberi GLP18722C

Injection Date . 9/12/1999 5:59:17 PM Seq. Line : 11
Sample Name : 98-0081-1 Cntrl Vial : 11
Acq. Operator : wos Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0912 S
Acq. Method C.\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/12/1999 1:55:27 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed 9/14/1999 9:33:48 AM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 9/14/1999 9:32:38 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.865	VB	9228.12891	6.08454e-7	5.61490e-3		RH7281

Totals : 5.61490e-3

Results obtained with enhanced integrator!

Figure 7. Control Cucumber (GC/ECD, TR 34-99-160, Peak Area > LOQ)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

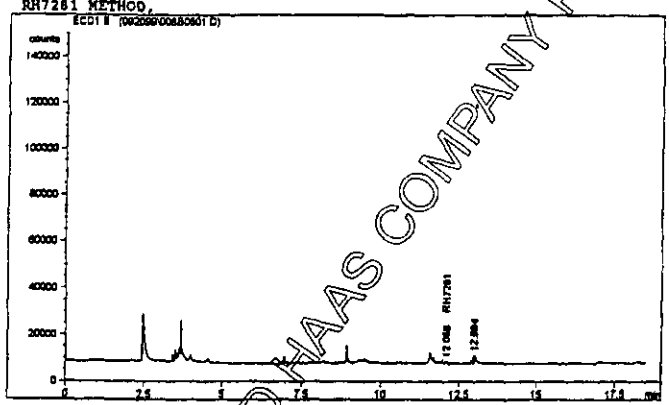
Data File C:\HPCHEM\2\DATA\092099\00680601.D Sample Name: 98-0081-10 Cntrl

RAR 98-0081-10 Control Cucumberi GLP18724C

Injection Date : 9/20/1999 12:12:36 PM
Sample Name : 98-0081-10 Cntrl
Acq. Operator : wos

Seq Line : 6
Vial : 6
Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0920.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/20/1999 10:10:10 AM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/21/1999 11:19:29 AM by wos
RH7281 METHOD



External Standard Report

Sorted By : Signal
Calib. Data Modified : 9/21/1999 11:18:25 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.088	BB	4697.85547	0.00000	0.00000		RH7281

Totals : 0.00000

Results obtained with enhanced integrator!
1 Warnings or Errors :

Instrument 2 9/21/1999 11:20:02 AM wos Page 1 of 2

Figure 8. Control Cucumber (GC/ECD, TR 34-99-160, Peak Area = 0).

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        . C \HPCHEM\2\DATA\032299\SPKZ-1 D
Operator      IG
Acquired      3-22-99 20 06 55  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name  0 01ppm spkie, cucumber
Misc Info   . 0.01ppm spike cucumber
Vial Number. 7
```

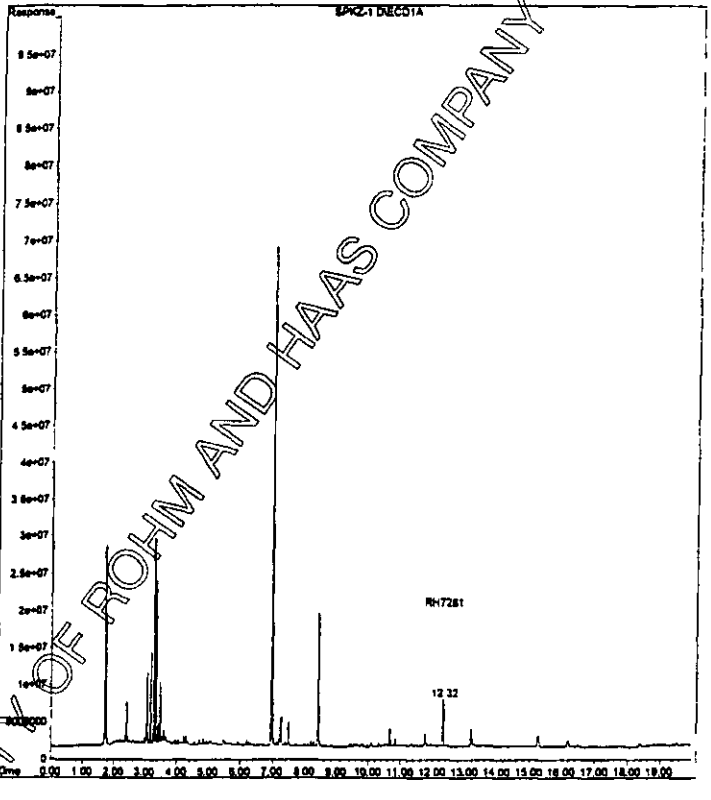


Figure 9. Fortification Cucumber at 0.010 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 113724481, Recovery = 90 8%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032299\SPKZ-3 D
Operator      IG
Acquired      3-22-99 20 57 32  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0 02ppm spkie, cucumber
Misc Info   - 0 02ppm spike cucumber
Vial Number   9
```

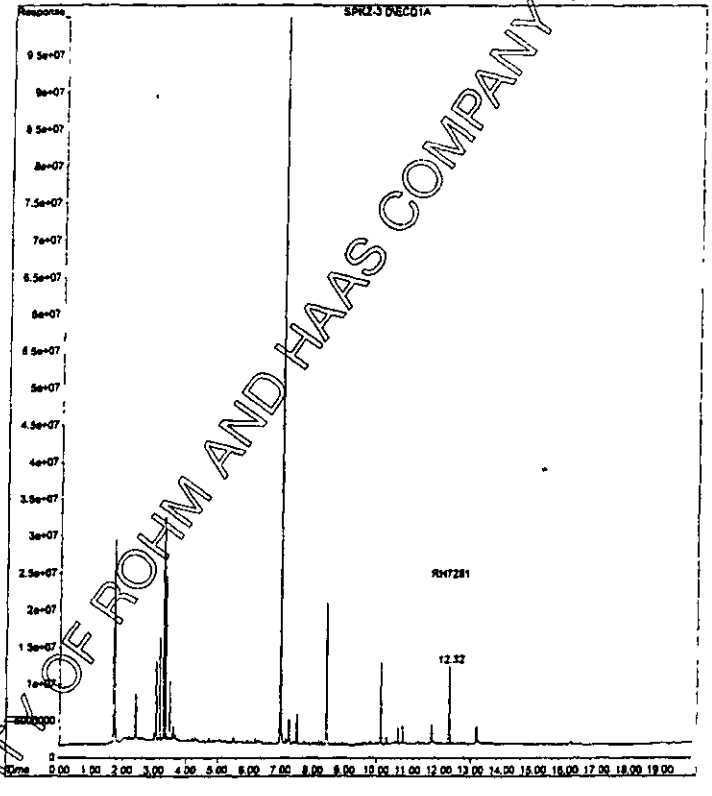


Figure 10 Fortification Cucumber at 0.020 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 194723622; Recovery = 75 2%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C:\HPCHEM\2\DATA\032299\SPKZ-4 D
Operator
Acquired      3-23-99 9 44.08  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0 05ppm spkie, cucumber
Misc Info     0 05ppm spike cucumber
Vial Number   10
```

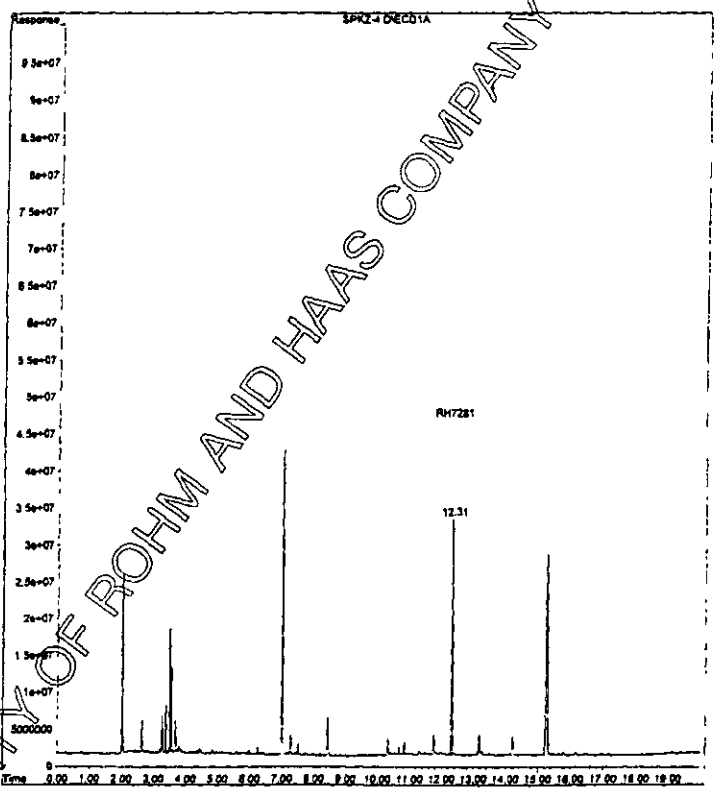


Figure 11 Fortification Cucumber at 0.050 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 556894100, Recovery = 88 1%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          : C \HPCHEM\2\DATA\032299\SPKZ-5 D
Operator        IG
Acquired        3-22-99 21 48:11  using AcqMethod RH7281 M
Instrument ·      GC ECD
Sample Name     0 10ppm spkie, cucumber
Misc Info       0 10ppm spike cucumber
Vial Number     11
```

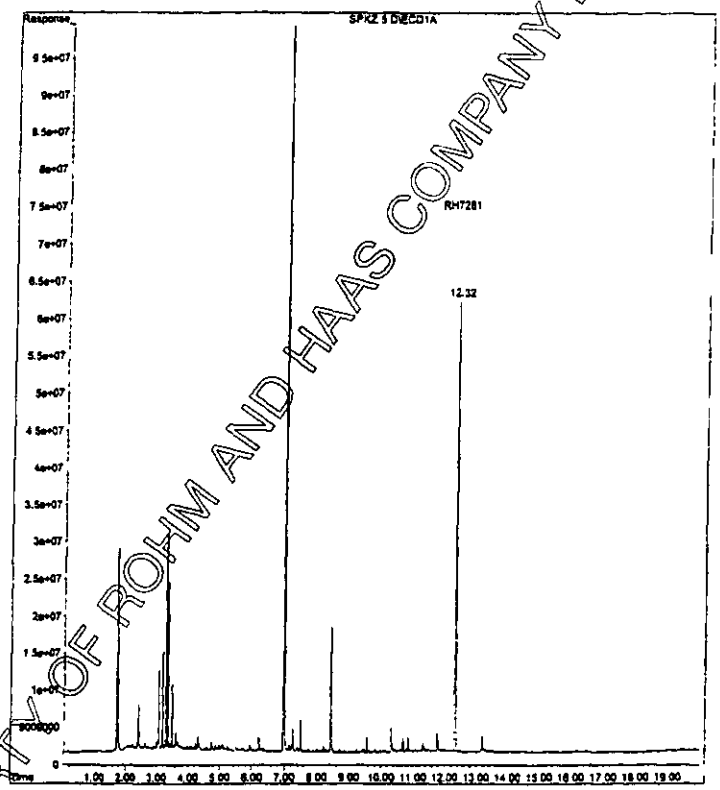


Figure 12 Fortification Cucumber at 0 10 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 1057632744, Recovery = 84.0%

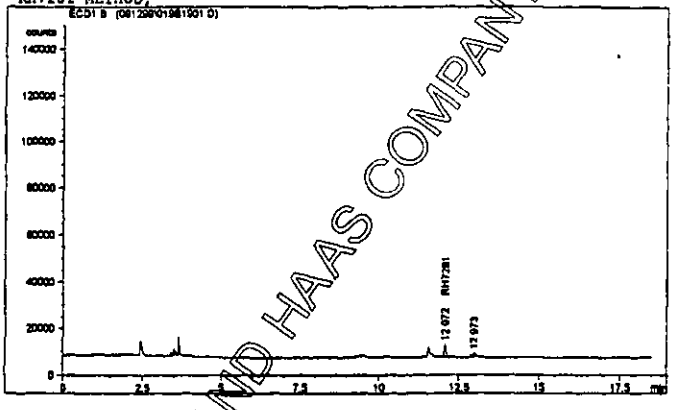
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091299\019B1901.D Sample Name: 98-0081-4 Trd#4A

RAR 98-0081-4 Treated#4 Cucumber GLP18722T4A
Diluted 1:10 with hexane

Injection Date : 9/12/1999 9:12:46 PM Seq. Line : 19
Sample Name : 98-0081-4 Trd#4A Vial : 19
Acq. Operator : wos Inj : 1
Inj Volume : 2 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0912.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/12/1999 1.55:27 PM by wos
Analysis Method C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/14/1999 9:33:48 AM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 9/14/1999 9:32:38 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.072	BB	2.08203e4	5.72173e-7	1.19128e-2		RH7281
Totals :				1.19128e-2		

Results obtained with enhanced integrator!

Instrument 2 9/14/1999 9.35.04 AM wos Page 1 of 2

Figure 13. Treated Cucumber Sample (GC/ECD, TR 34-99-160)
Concentration of RH-7281 = 0 0119 µg/g

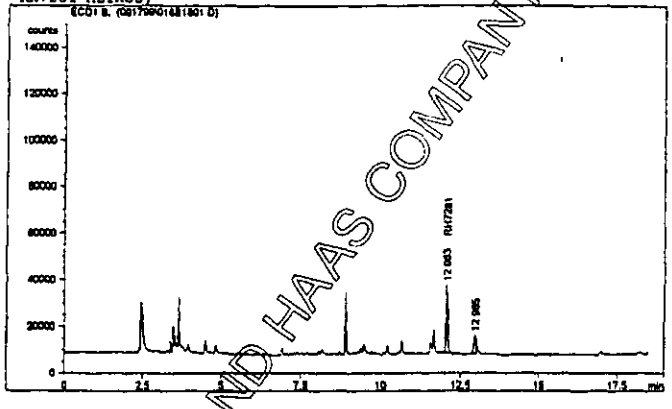
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091799\018B1801.D Sample Name: 98-0081-7 Trd#7

RAR 98-0081-7 Treated#7 Cucumber GLP18723T7

```
Injection Date  : 9/17/1999 10:05:40 PM          Seq. Line :  18
Sample Name     : 98-0081-7 Trd#7                     Vial :  18
Acq  Operator   : wos                                  Inj :   1
                                                Inj Volume : 5 µl

Sequence File   : C.\HPCHEM\1\SEQUENCE\WS0917.S
Acq. Method     : C:\HPCHEM\2\METHODS\RH7281.M
Last changed    : 9/17/1999 3:03:13 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed    : 9/20/1999 9:31.20 AM by wos
 RH7281 METHOD,
```



External Standard Report

```
Sorted By              :      Signal
Calib. Data Modified   :      9/20/1999 9:30.50 AM
Multiplier             :      1.0000
Dilution               :      1.0000

Signal 1: ECD1 B,
```

RetTime (min)	Type	Area counts*s	Amt/Area	Amount (ng/ul)	Grp	Name
12.063	PB	1 11914e5	5.13046e-7	5.74172e-2		RH7281
Totals :				5.74172e-2		

Results obtained with enhanced integrator!

Instrument 2 9/20/1999 9:32:45 AM wos Page 1 of 2

Figure 14 Treated Cucumber Sample (GC/ECD, TR 34-99-160)
Concentration of RH-7281 = 0.0571 µg/g.

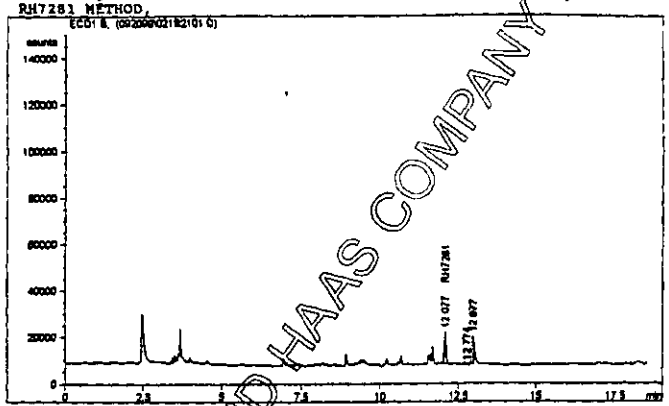
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\092099\021B2101.D Sample Name: 98-0081-15Trd#1

RAR 98-0081-15 Treated#15 Cucumber GLP18724T15

```
Injection Date  . 9/20/1999 6:15:46 PM          Seq. Line :  21
Sample Name     . 98-0081-15Trd#15                   Vial :  21
Acq. Operator   . wos                                 Inj .   1
                                                Inj Volume . 1 µl

Sequence File   : C:\HPCHEM\1\SEQUENCE\WS0920.S
Acq. Method     : C:\HPCHEM\2\METHODS\RH7281.M
Last changed    : 9/20/1999 10:10:10 AM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed    : 9/21/1999 11.19:29 AM by wos
 RH7281 METHOD,
```



External Standard Report

```
Sorted By                 :      Signal
Calib. Data Modified      :      9/21/1999 11:18:25 AM
Multiplier                :      1.0000
Dilution                  :      1.0000

Signal 1: ECD1 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          counts*s                [ng/ul]
-------|------|----------|----------|----------|--|-----------------
 12.077 BB     5.30727e4  5.69105e-7 3.02040e-2     RH7281

Totals :                               3.02040e-2

 Results obtained with enhanced integrator!
```

Instrument 2 9/21/1999 11:20:52 AM wos Page 1 of 2

Figure 15. Treated Cucumber Sample (GC/ECD, TR 34-99-160) Concentration of RH-7281 = 0 0301 µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\2\DATA\032699\CONTROL D
Operator    : IG
Acquired      3-26-99 20:35:17  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   control, cantaloupe
Misc Info   : Cantaloupe control
Vial Number : 7
```

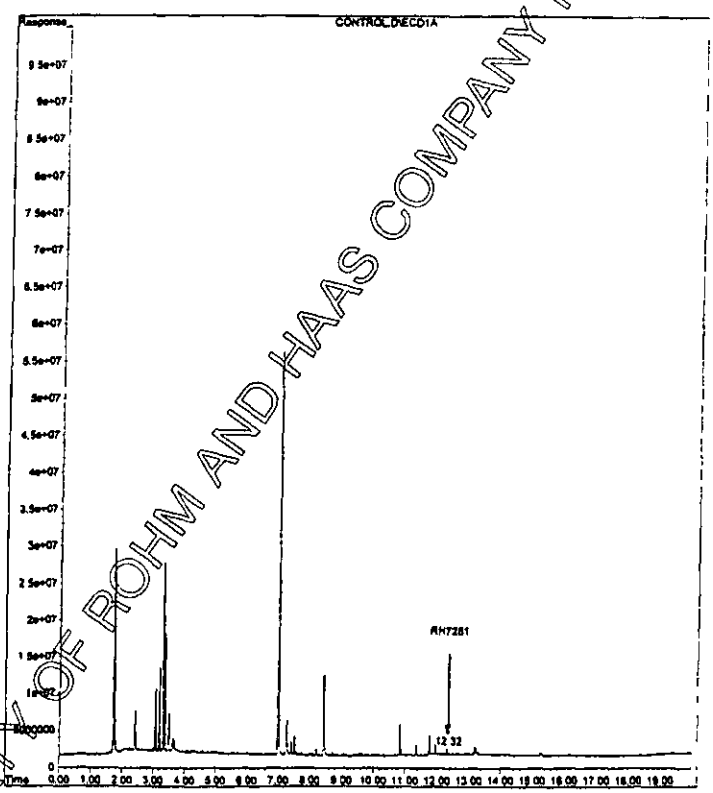


Figure 16. Control Cantaloupe (GC/ECD, TR 34-99-43, Peak Area < LOQ)

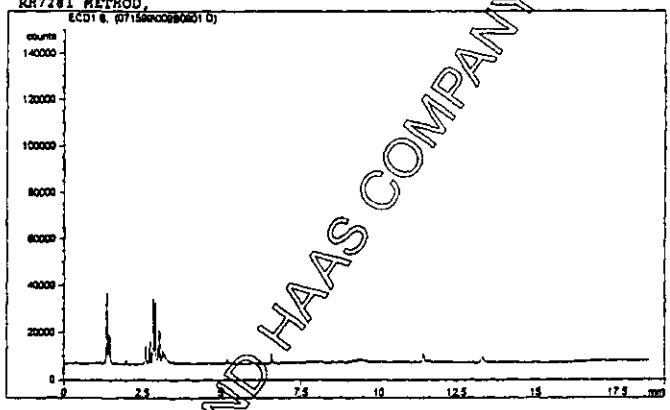
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\071599\009B0901.D Sample Name: 98-0070-1 Cntr

98-0070-1 Control Cantaloupe

Injection Date : 7/15/1999 7:33:12 PM Seq Line : 9
Sample Name : 98-0070-1 Cntrl Vial : 9
Acq. Operator : wos Inj : 1
Inj Volume : µl

Sequence File : C:\HPCHEM\2\SEQUENCE\WS0715.S
Acq. Method : C \HPCHEM\1\METHODS\RH7281 M
Last changed : 7/15/1999 4:07:36 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 8/17/1999 9:56:15 AM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 8/17/1999 9 55:38 AM
Multiplier : 1.0000
Dilution : 1 0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.295		-	-	-		RH7281

Totals : 0.00000

Results obtained with enhanced integrator!
1 Warnings or Errors :

Instrument 2 8/17/1999 9:59:24 AM wos Page 1 of 2

Figure 17 Control Cantaloupe (GC/ECD, TR 34-99-160, Peak Area = 0)

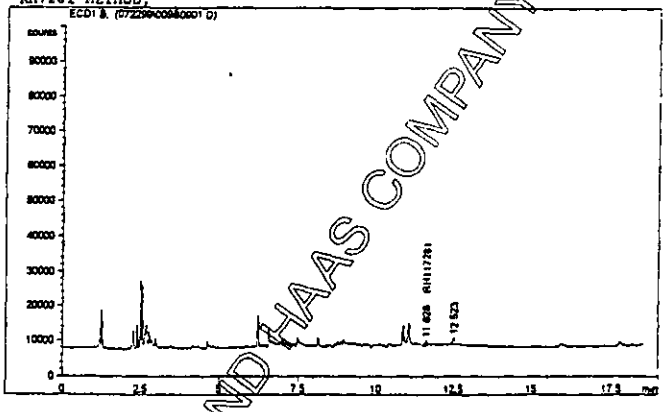
PROPERTY ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\072299\009B0901 D Sample Name: 98-0072-1 Cntr

RAR 98-0072-1 Control Cantaloupe GLP1875C

Injection Date : 7/23/1999 4:57:08 PM Seq. Line : 9
Sample Name : 98-0072-1 Cntrl Vial : 9
Acq. Operator : WOS Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\WS0723.S
Acq. Method : C.\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/23/1999 1:20:21 PM by WOS
Analysis Method : C:\HPCHEM\2\METHODS\RH7281 M
Last changed : 7/26/1999 7:53 32 AM by WOS
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 7/26/1999 7:50:54 AM
Multiplier : 1 0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
11.628	PB	5695 54297	6.41602e-7	3.65427e-3		RH117281
Totals				3.65427e-3		

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 2 7/26/1999 7:54.08 AM WOS Page 1 of 1

Figure 18. Control Cantaloupe (GC/ECD,-TR 34-99-160, Peak Area > LOQ)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C:\HPCHEM\2\DATA\032699\SPK1 D
Operator      IG
Acquired      3-26-99 21.00 36  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0 01ppm spike, cantaloupe
Misc Info     0 01ppm spike cantaloupe
Vial Number   8
```

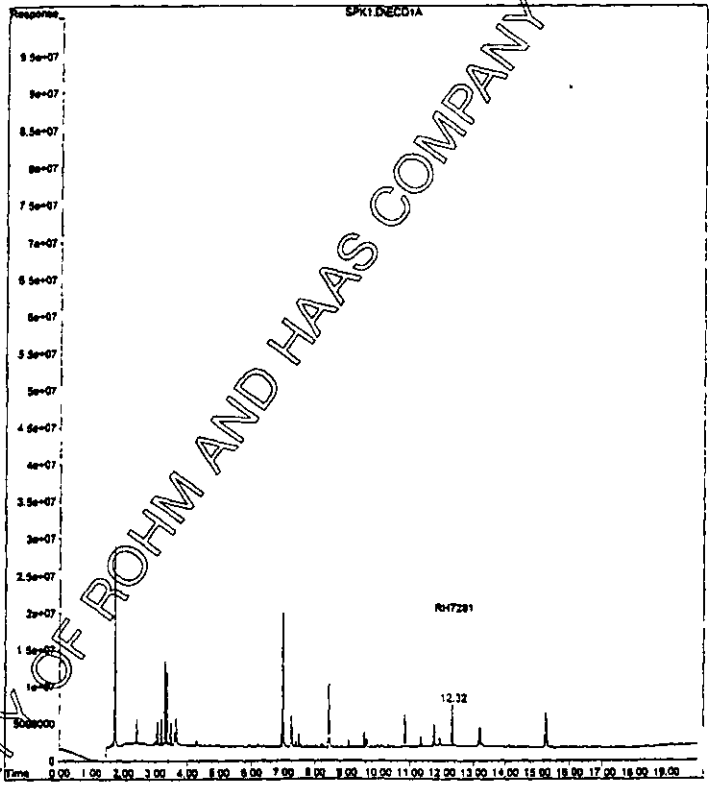


Figure 19. Fortification Cantaloupe at 0.010 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 96038128, Recovery = 73 0%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\2\DATA\032699\SPK3 D
Operator      IG
Acquired      3-26-99 21 51.16  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0.02ppm spike, cantaloupe
Misc Info     0 02ppm spike cantaloupe
Vial Number   10
```

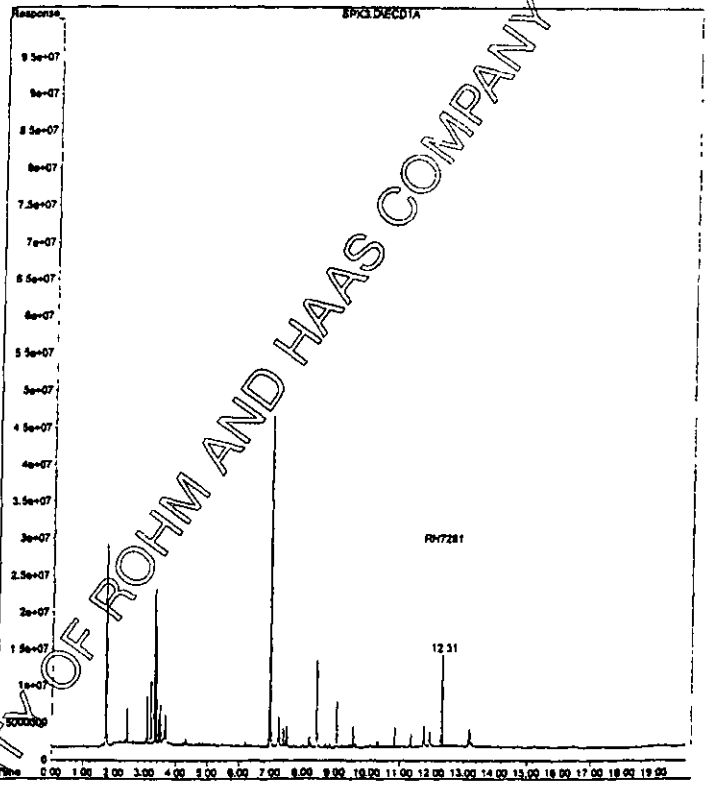


Figure 20 Fortification Cantaloupe at 0 020 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 219084323, Recovery = 82 7%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032699\SPK4 D
Operator    . IG
Acquired      3-26-99 22 16:33  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0 05ppm spike, cantaloupe
Misc Info     0 05ppm spike cantaloupe
Vial Number   11
```

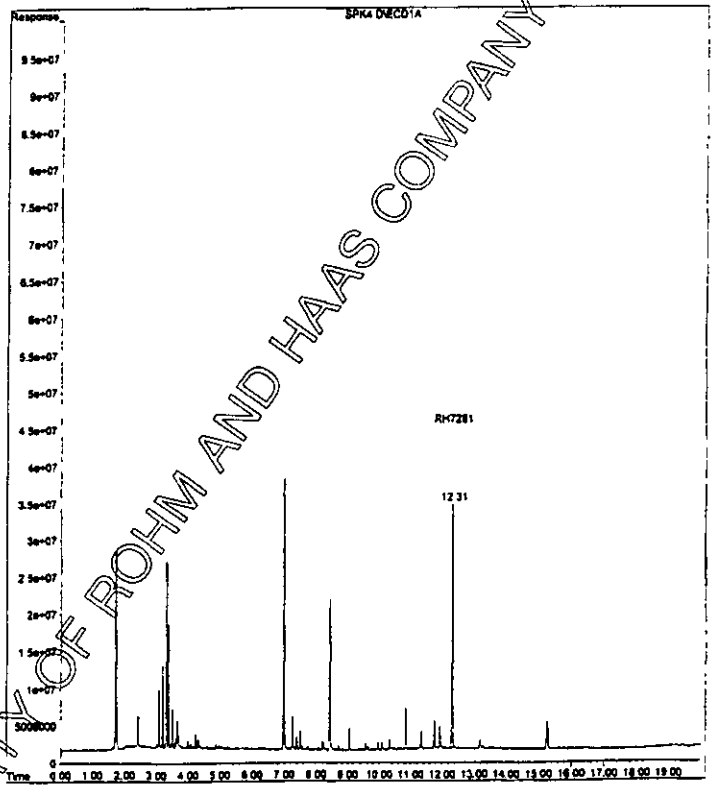


Figure 21 Fortification Cantaloupe at 0 050 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 560842721; Recovery = 84.5 %

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\032699\SPK5 D
Operator      IG
Acquired      3-26-99 22 41 52  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   0.10ppm spike, cantaloupe
Misc Info     0 10ppm spike cantaloupe
Vial Number   12
```

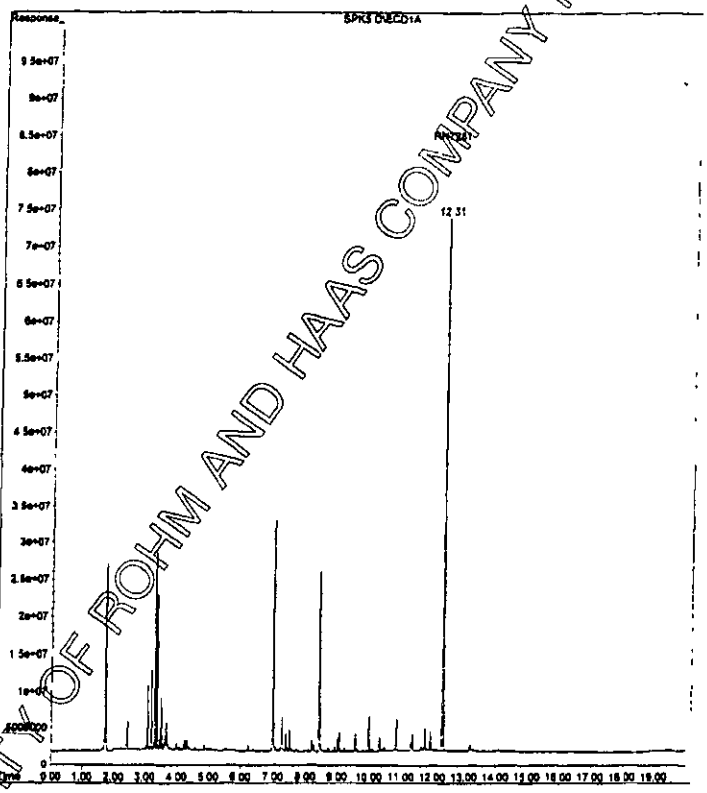


Figure 22 Fortification Cantaloupe at 0 10 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 1243317039, Recovery = 93 6%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

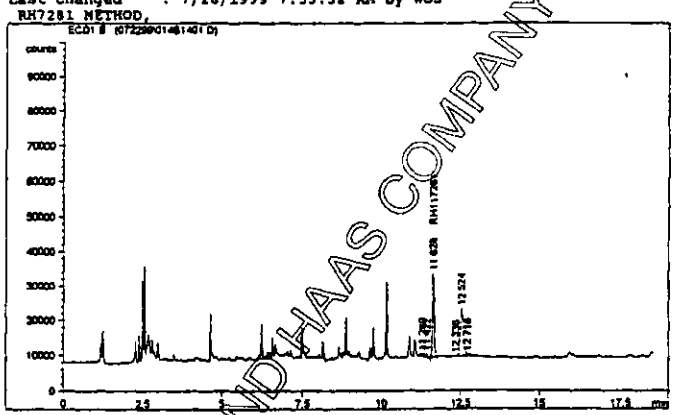
Data File C:\HPCHEM\2\DATA\072299\014B1401.D Sample Name: 98-0072-3 Trtd#3

RAR 98-0072-3 Cantaloupe treated #3 GLP1875T3

Injection Date : 7/23/1999 6:58:33 PM
Sample Name : 98-0072-3 Trtd#3
Acq. Operator : WOS

Seq. Line : 14
Vial : 14
Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\2\SEQUENCE\WS0723.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/23/1999 1:20:23 PM by WOS
Analysis Method : C.\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/26/1999 7:53:32 AM by WOS
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib Data Modified : 7/26/1999 7 50 54 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime (min)	Type	Area counts*s	Amt/Area	Amount (ng/ul)	Grp	Name
11.628	BB	8.94142e4	6.41602e-7	5.73683e-2		RH117281

Totals : 5.73683e-2

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 2 7/26/1999 7:54:24 AM WOS Page 1 of 1

Figure 23 Treated Cantaloupe Sample (ECD, TR 34-99-160)
Concentration of RH-7281 = 0.0572 µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

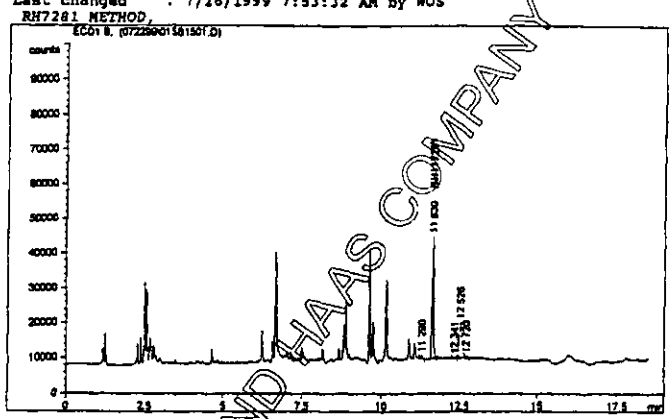
Data File C:\HPCHEM\2\DATA\072299\015B1501.D Sample Name: 98-0072-4 Trtd#4

RAR 98-0072-4 Cantaloupe treated #4 GLP1875T4

Injection Date : 7/23/1999 7:22:51 PM
Sample Name : 98-0072-4 Trtd#4
Acq. Operator : WOS

Seq Line : 15
Vial : 15
Inj : 1
Inj Volume : 1 µl

Sequence File : C.\HPCHEM\2\SEQUENCE\WS0723.S
Acq. Method : C \HPCHEM\2\METHODS\RH7281.M
Last changed : 7/23/1999 1:20:23 PM by WOS
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/26/1999 7:53:32 AM by WOS
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 7/26/1999 7:50.54 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
11.630	PB	1.29845e5	6.41602e-7	8.33086e-2		RH117281

Totals : 8.33086e-2

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 2 7/26/1999 7:54.27 AM WOS Page 1 of 1

Figure 24 Treated Cantaloupe Sample (ECD, TR 34-99-160)
Concentration of RH-7281 = 0 0829 µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

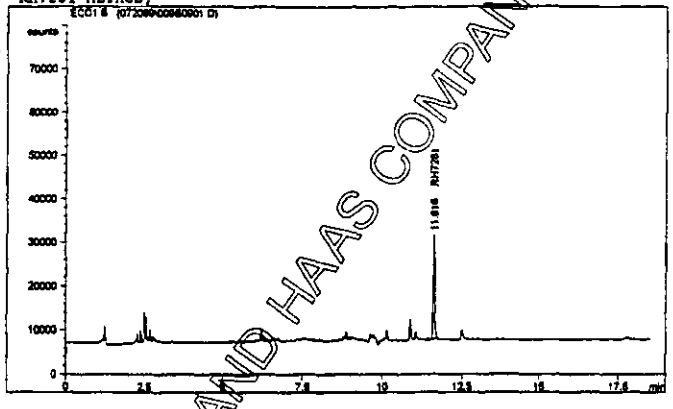
Data File C:\HPCHEM\2\DATA\072099\00980901.D Sample Name: 98-0070-4 Trtd#4

RAR 98-0070-4 Cantaloupe treated #4 diluted GLP1873T4 (1:10)

Injection Date : 7/20/1999 6:32:51 PM
Sample Name : 98-0070-4 Trtd#4
Acq. Operator : WOS

Seq. Line : 9
Vial : 9
Inj : 1
Inj Volume : µl

Sequence File : C \HPCHEM\2\SEQUENCE\WS0720.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/20/1999 3:17:14 PM by WOS
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 7/21/1999 12.08:26 PM by ig
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 7/21/1999 12:07.01 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
11.616	PB	8.94653e4	8.18523e-7	7.32294e-2		RH7281

Totals : 7.32294e-2

Results obtained with enhanced integrator!

Instrument 2 7/21/1999 12:09.32 PM wos Page 1 of 1

Figure 25. Treated Cantaloupe Sample (ECD, TR 34-99-160)
Concentration of RH-7281 ≈ 0.0731 µg/g

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        . C.\HPCHEM\2\DATA\040599\CONTR-Z D
Operator
Acquired      4-4-99 21 08.07  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name· zucchini control
Misc Info
Vial Number  21
```

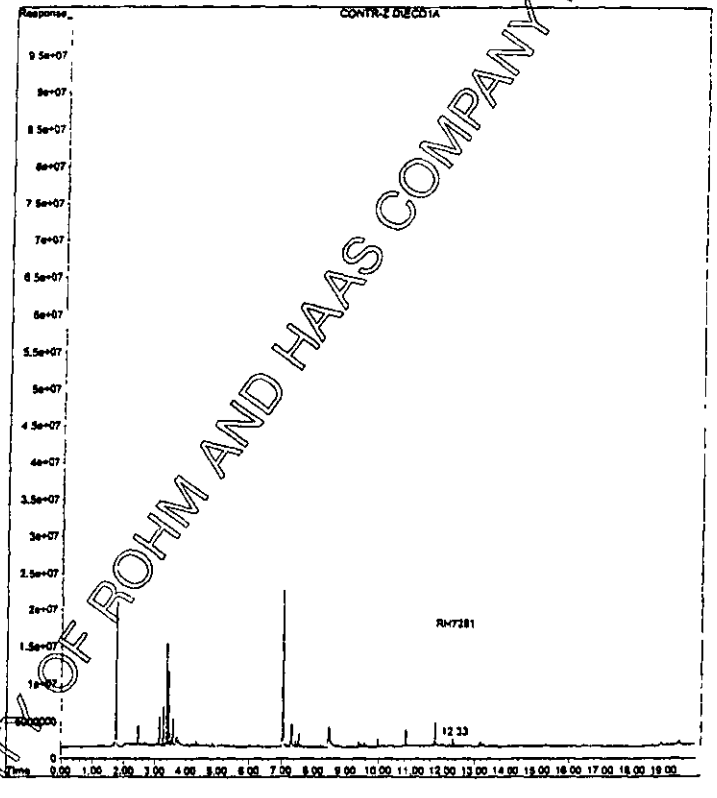


Figure 26 Control Zucchini (GC/ECD, TR 34-99-43, Peak Area < LOQ)

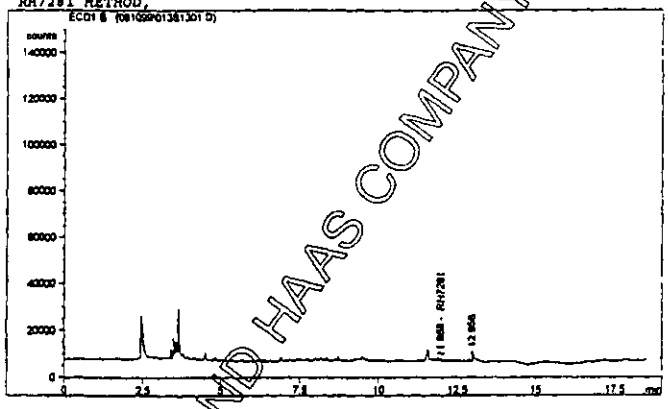
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091099\013H1301.D Sample Name: 99-0026-18 Cntrl

RAR 99-0026-18 Control Zucchini GLP18720C

Injection Date . 9/10/1999 3:06 02 PM Seq. Line : 17
Sample Name . : 99-0026-18 Cntrl Vial : 13
Acq. Operator . wos Inj : 1
Inj Volume : µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0910.S
Acq Method : C.\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/2/1999 1:47:45 PM by wos
Analysis Method : C·\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/12/1999 1·08:11 PM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 9/12/1999 1:07.00 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
11.968	PP	1133 22522	0.00000	0.00000		RH7281
Totals :				0.00000		

Results obtained with enhanced integrator!
1 Warnings or Errors .

Instrument 2 9/12/1999 1·09:02 PM wos Page 1 of 2

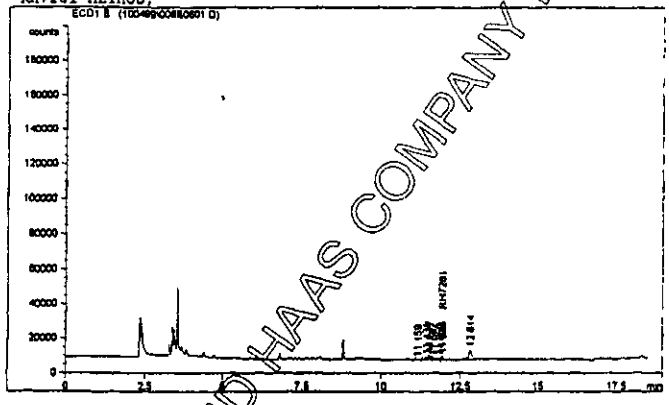
Figure 27 Control Zucchini (GC/ECD, TR 34-99-160, Peak Area = 0).

PROPERTY ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\100499\006B0601.D Sample Name. 98-0078-1 Cntr

RAR 98-0078-1 Control Zucchini GLP18728C

```
Injection Date  : 10/4/1999 5:39:04 PM          Seq  Line .
Sample Name     : 98-0078-1 Cntrl                    Vial :
Acq. Operator   : wos                                 Inj .
                                                 Inj Volume . 1 µl
Sequence File   : C \HPCHEM\2\DATA\WS1004 S
Acq. Method     . C.\HPCHEM\2\METHODS\RH7281.M
Last changed    · 10/4/1999 3:35:53 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed    : 10/5/1999 8.24:43 AM by wos
RH7281 METHOD,
```



External Standard Report

```
Sorted By              :      Signal
Calib. Data Modified   :      10/5/1999 8:24:25 AM
Multiplier             :      1.0000
Dilution               :      1.0000

Signal 1: ECD1 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          counts*s                [ng/ul]
--------|------|----------|----------|----------|--|-----------------
 11.815 VB      7550 74805    0.00000    0.00000     RH7281

Totals :                                 0.00000

 Results obtained with enhanced integrator!
 1 Warnings or Errors :

Instrument 2 10/5/1999 8 25:10 AM wos                    Page 1 of 2
```

Figure 28 Control Zucchini (GC/ECD, TR 34-99-160, Peak Area = 0).

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C.\HPCHEM\2\DATA\040599\SPK1.D
Operator
Acquired    : 4-4-99 21 33.24  using AcqMethod RH7281 M
Instrument :   GC ECD
Sample Name  zucchini spike 0 01ppm
Misc Info
Vial Number  22
```

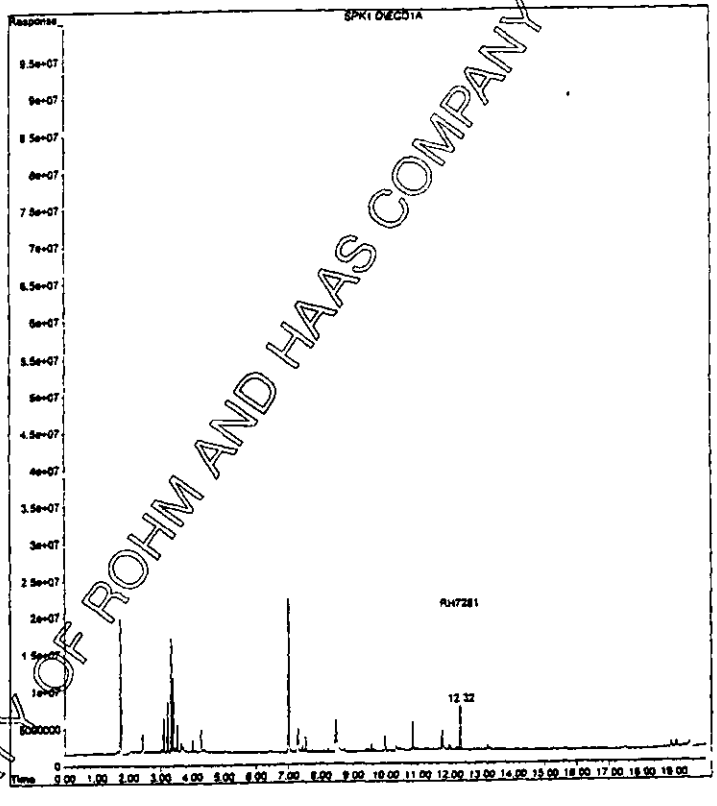


Figure 35 Fortification Zucchini at 0 010 µg/g (GC/ECD, TR 34-99-43), Peak Area = 113599847, Recovery = 100%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C:\HPCHEM\2\DATA\040599\SPK3.D
Operator
Acquired      4-4-99 22 23 54  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name   zucchini spike 0 02ppm
Misc Info
Vial Number.  24
```

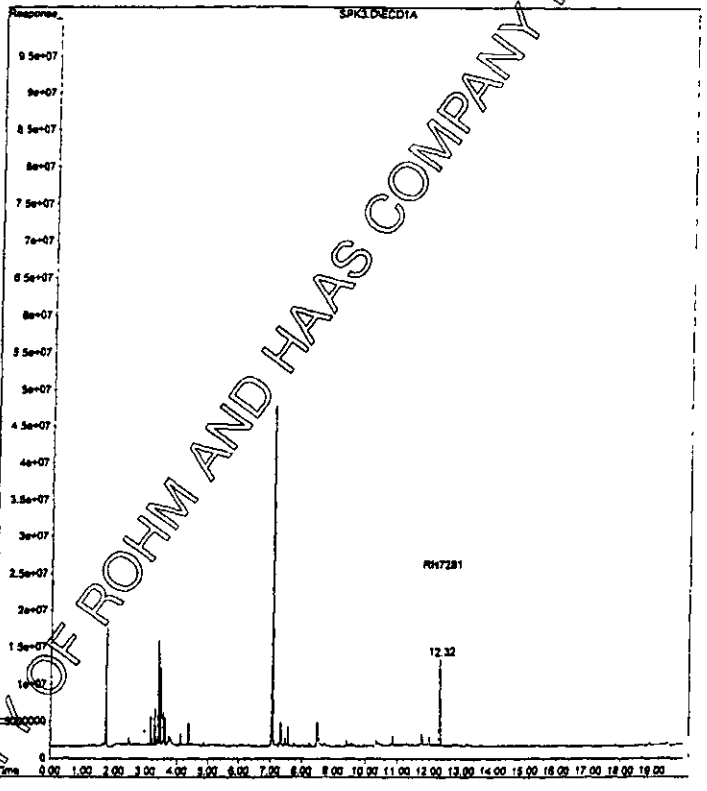


Figure 30 Fortification Zucchini at 0 020 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 229488640, Recovery = 100 %.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\2\DATA\040599\SPK4 D
Operator    :
Acquired      4-4-99 22 49 09  using AcqMethod RH7281 M
Instrument .    GC ECD
Sample Name   zucchini spike 0 05ppm
Misc Info
Vial Number. 25
```

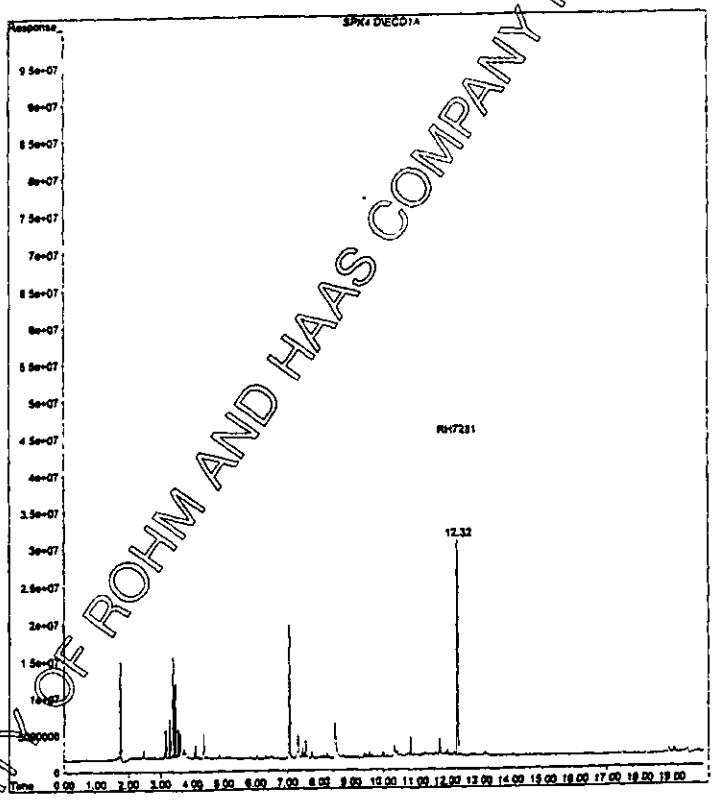


Figure 31 Fortification Zucchini at 0 050 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 540411258, Recovery = 96.0%

```
File          C \HPCHEM\2\DATA\040599\SPK5 D
Operator
Acquired      4-4-99 23 14 25  using AcqMethod RH7281 M
Instrument      GC ECD
Sample Name·  zucchini spike 0 10ppm
Misc Info
Vial Number   26
```

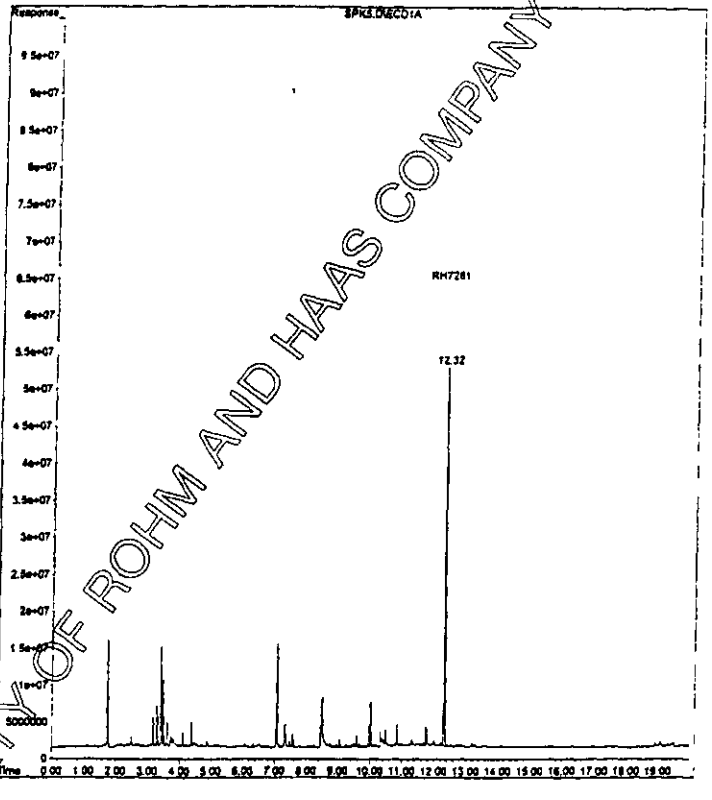


Figure 32 Fortification Zucchini at 0 10 µg/g (GC/ECD, TR 34-99-43)
Peak Area = 900294120, Recovery = 79 9%

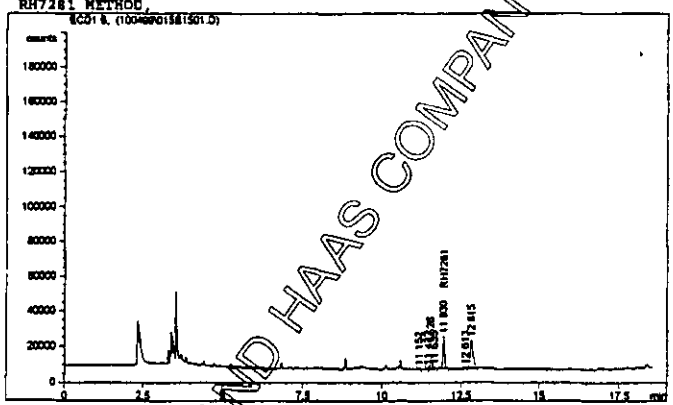
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\100499\015B1501 D Sample Name: 98-0078-3 Trd#

RAR 98-0078-3 Treated#3 Zucchini GLP18728T3

Injection Date : 10/4/1999 9:17:10 PM Seq. Line : 15
Sample Name : 98-0078-3 Trd#3 Vial : 15
Acq. Operator : wos Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\2\DATA\WS1004.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 10/4/1999 3:35:53 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281 M
Last changed : 10/5/1999 8:24:43 AM by wos
RH7281 METHOD



External Standard Report

Sorted By : Signal
Calib. Data Modified : 10/5/1999 8:24:25 AM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
11.930	BB	6.80288e4	3.49473e-7	2.37742e-2		RH7281

Totals : 2.37742e-2

Results obtained with enhanced integrator!

Instrument 2 10/5/1999 8:25:40 AM wos Page 1 of 2

Figure 33. Treated Zucchini Sample (GC/ECD, TR 34-99-160)
Concentration of RH-728 = 0.0236 µg/g.

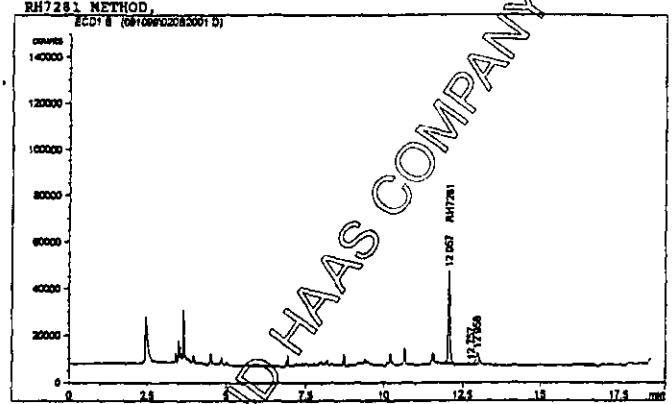
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091099\02082001.D Sample Name: 99-0026-16 Trd16

RAR 99-0026-16 Treated#16 Zucchini GLP18720T16

Injection Date : 9/10/1999 5:56:08 PM Seq. Line : 20
Sample Name : 99-0026-16 Trd16 Vial : 20
Acq. Operator : wos Inj : 1
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0910.S
Acq Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/2/1999 1:47:45 PM by wos
Analysis Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/12/1999 1:08:11 PM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib. Data Modified : 9/12/1999 1:07:00 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.057	PB	1.52320e5	5.26000e-7	8.01204e-2		RH7281

Totals : 8.01204e-2

Results obtained with enhanced integrator!

Instrument 2 9/12/1999 1:09:25 PM wos Page 1 of 2

Figure 34 Treated Zucchini Sample (GC/ECD, TR 34-99-160)
Concentration of RH-7281 = 0.0799 µg/g.

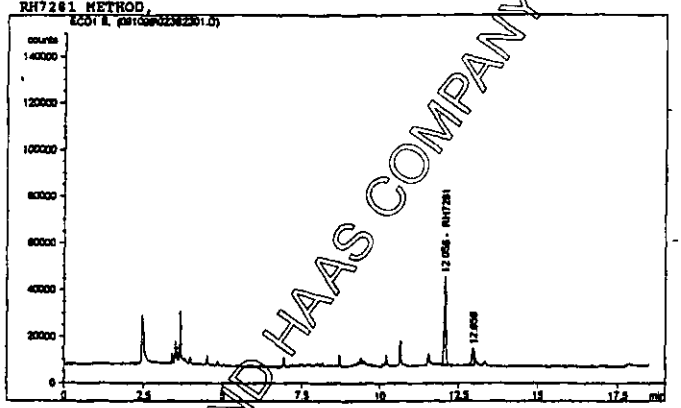
PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Data File C:\HPCHEM\2\DATA\091099\023B2301.D Sample Name: 99-0026-19 Trd1

RAR 99-0026-19 Treated#19 Zucchini GLP18720T19

Injection Date : 9/10/1999 7:09:02 PM Seq. Line : 23
Sample Name : 99-0026-19 Trd19 Vial : 23
Acq Operator : wos Inj : 2
Inj Volume : 1 µl

Sequence File : C:\HPCHEM\1\SEQUENCE\WS0910.S
Acq. Method : C:\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/2/1999 1:47:45 PM by wos
Analysis Method : C.\HPCHEM\2\METHODS\RH7281.M
Last changed : 9/12/1999 1:08:11 PM by wos
RH7281 METHOD,



External Standard Report

Sorted By : Signal
Calib Data Modified : 9/12/1999 1:07.00 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 B,

RetTime [min]	Type	Area counts*s	Amt/Area	Amount [ng/ul]	Grp	Name
12.056	BB	1.45514e5	5.25314e-7	7.64405e-2		RH7281

Totals : 7.64405e-2

Results obtained with enhanced integrator!

Instrument 2 9/12/1999 1:09:36 PM wos Page 1 of 2

Figure 35. Treated Zucchini Sample (GC/ECD, TR 34-99-160)
Concentration of RH-7281 = 0.0765 µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C.\HPCHEM\1\DATA\040199\STDB1.D
Operator
Acquired       1 Apr 99  15:37      using AcqMethod 7281SIM
Instrument       5973
Sample Name   std 0 01ppm
Misc Info
Vial Number·  2
```

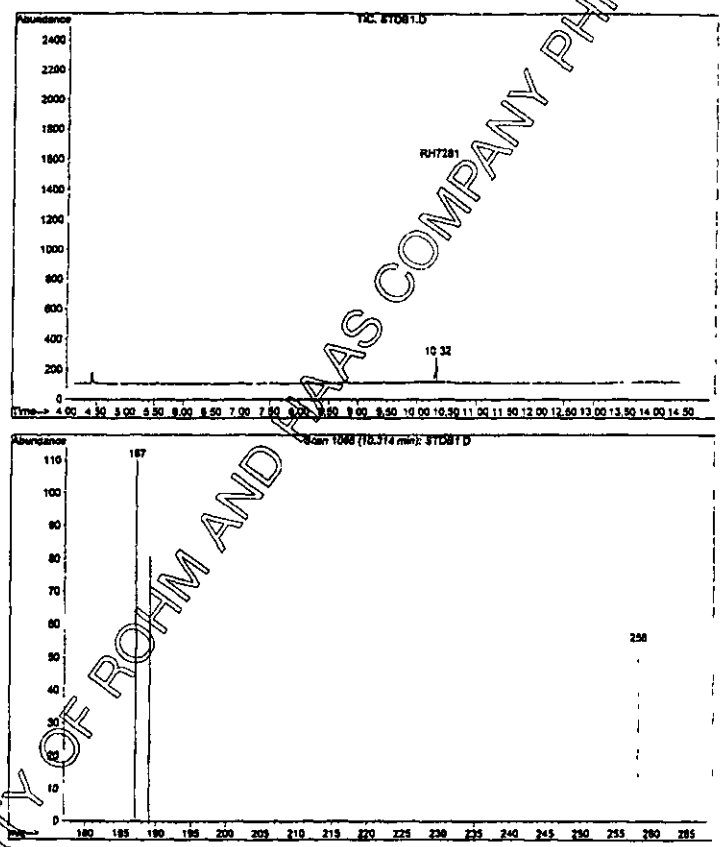


Figure 36. 0.010 µg/mL Standard RH-7281 (GC/MSD, Peak Area = 413)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C.\HPCHEM\1\DATA\040199\STDB2 D
Operator    :
Acquired      1 Apr 99  16:00      using AcqMethod 7281SIM
Instrument      5973
Sample Name. std 0.02ppm
Misc Info   :
Vial Number: 3
```

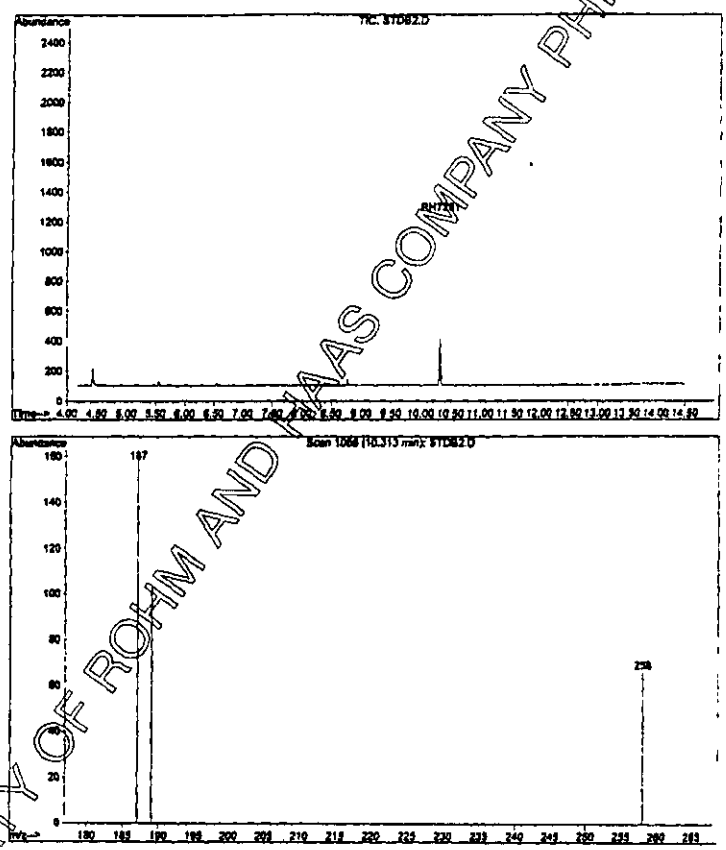
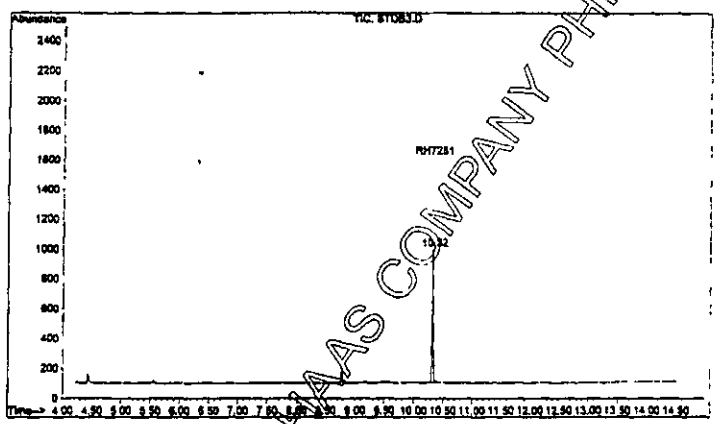


Figure 37. 0 020 µg/mL Standard RH-7281 (GC/MSD, Peak Area = 800)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\040199\STDB3.D
Operator
Acquired      1 Apr 99  16:22      using AcqMethod 7281SIM
Instrument      5973
Sample Name   std 0.05ppm
Misc Info
Vial Number   4
```



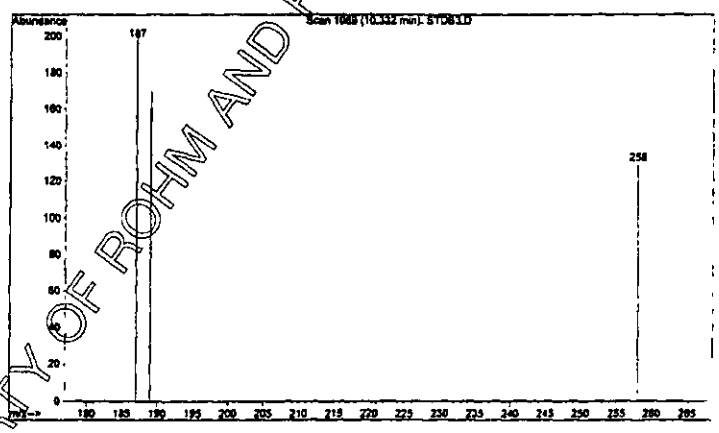


Figure 38 0.050 µg/mL Standard RH-7281 (GC/MSD, Peak Area = 2220).

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\1\DATA\040199\STDB4 D
Operator    :
Acquired      1 Apr 99  16:44     using AcqMethod 7281SIM
Instrument      5973
Sample Name. std 0.10ppm
Misc Info
Vial Number. 5
```

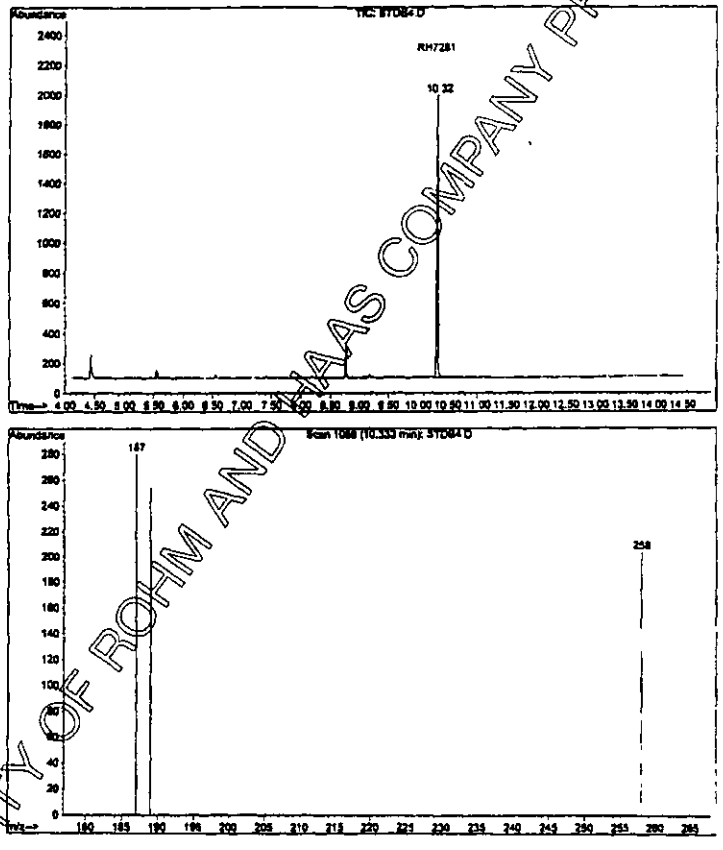
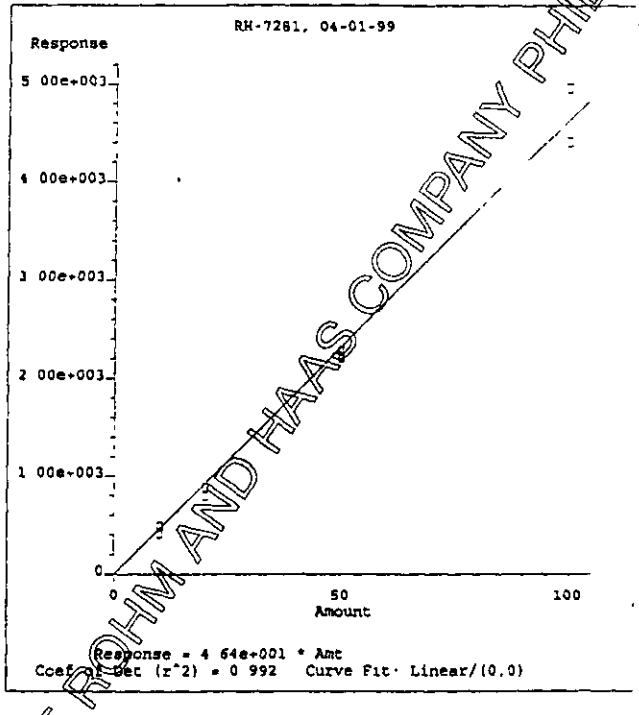


Figure 39. 0 10 µg/mL Standard RH-7281 (GC/MSD, Peak Area = 4413)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA



Method Name C \HPCHEM\1\METHODS\7281SIM M
Calibration Table Last Updated. Tue May 04 12 53-10 1999

Figure 40 Calibration Curve for RH-7281 (GC/MSD, 04-01-1999).

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        . C:\HPCHEM\1\DATA\050699\CONTROL.D
Operator      IG
Acquired       6 May 99  19.31     using AcqMethod 7281SIM
Instrument ·     5973
Sample Name:  control cucumber
Misc Info
Vial Number  6
```

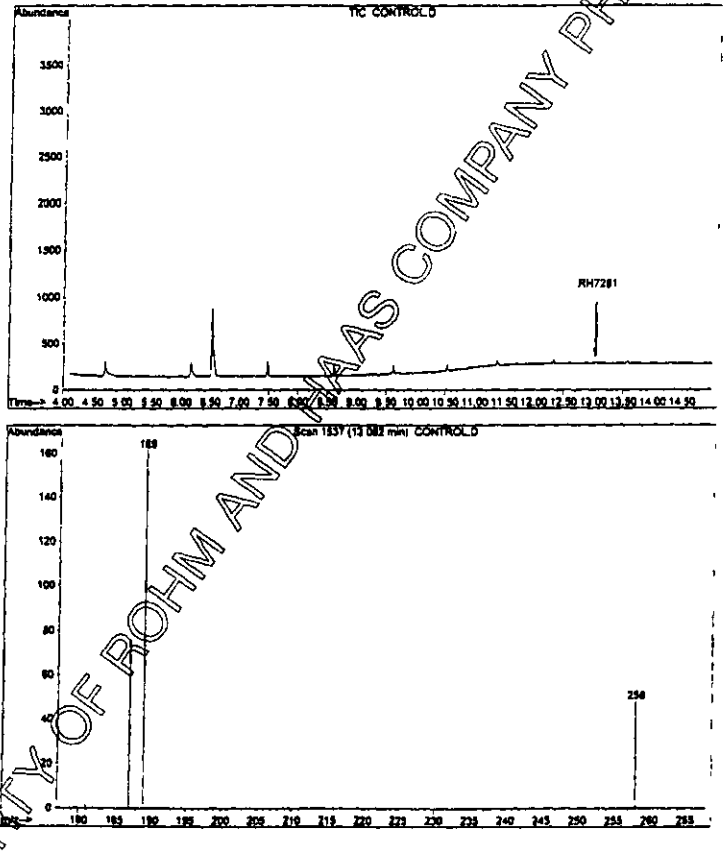
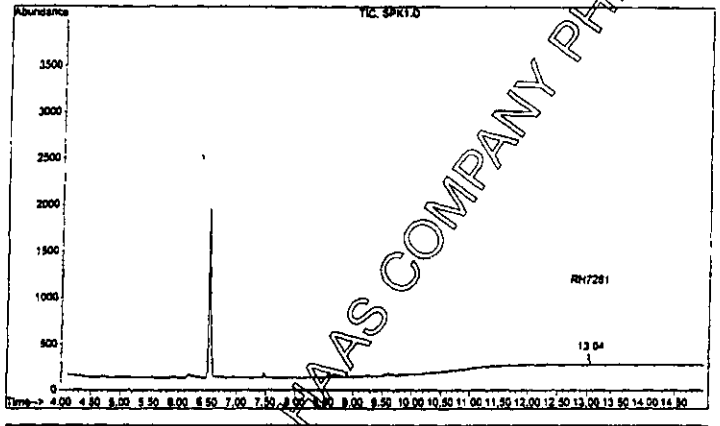


Figure 41. Control Cucumber (GC/MSD, TR 34-99-43, Peak Area = 0)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C:\HPCHEM\1\DATA\050699\SPK1 D
Operator    : IG
Acquired      6 May 99  19 55     using AcqMethod 7281SIM
Instrument .    5973
Sample Name: spike cucumber 0 01 ppm
Misc Info  .
Vial Number: 7
```



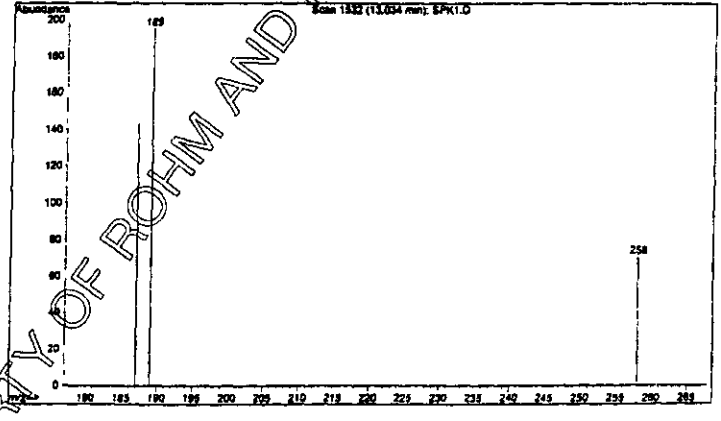
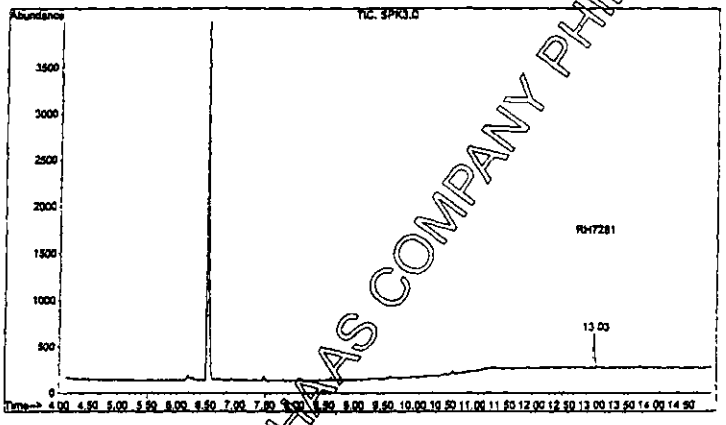


Figure 42 Fortification Cucumber at 0 010 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 470; Recovery = 67 2%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C:\HPCHEM\1\DATA\050699\SPK3 D
Operator      IG
Acquired    .  6 May 99  20 43      using AcqMethod 7281SIM
Instrument       5973
Sample Name:  spike cucumber 0 02 ppm
Misc Info
Vial Number   9
```



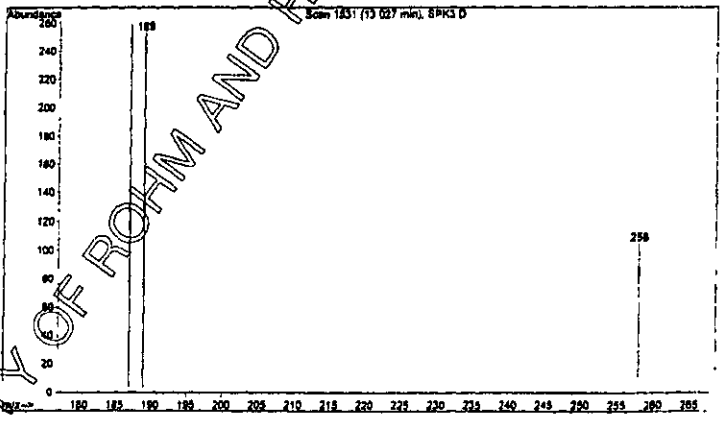
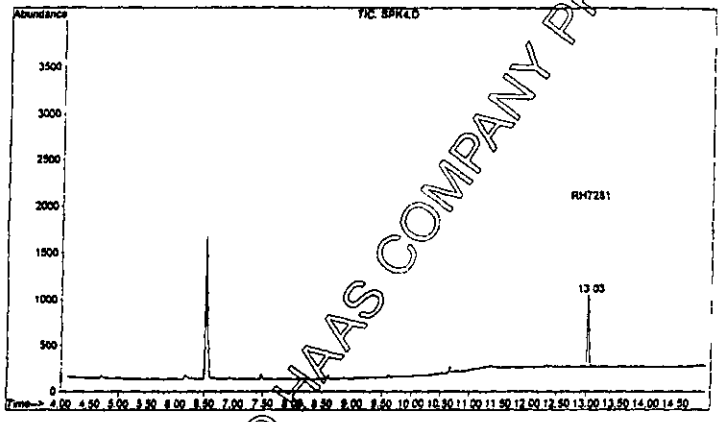


Figure 43 Fortification Cucumber at 0.020 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 1367, Recovery = 97.8%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\050699\SPK4.D
Operator    . IG
Acquired       6 May 99  21 07      using AcqMethod 7281SIM
Instrument       5973
Sample Name: spike cucumber 0.05 ppm
Misc Info
Vial Number  10
```



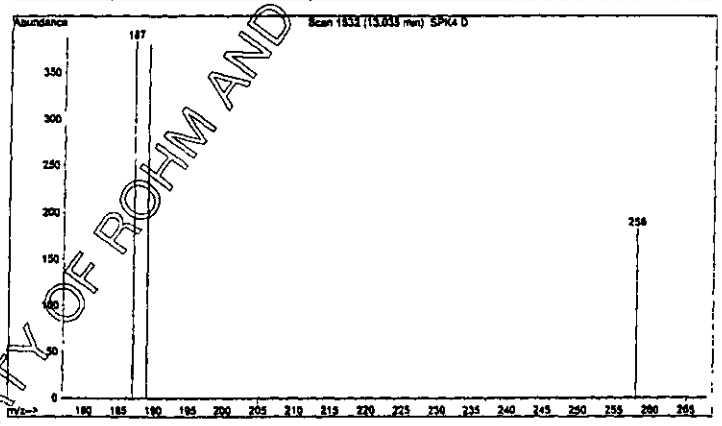
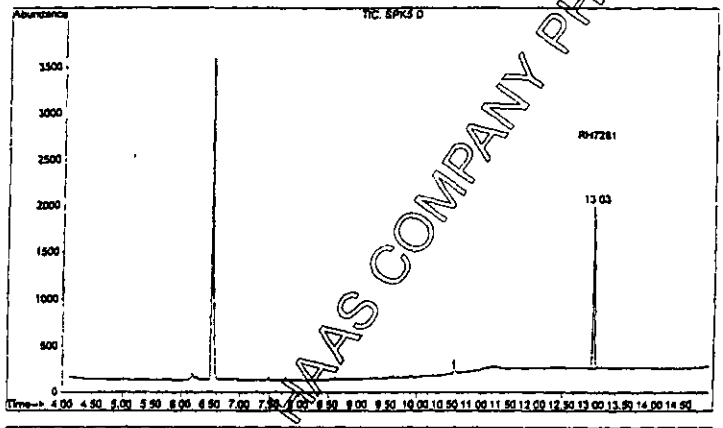


Figure 44. Fortification Cucumber at 0.050 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 2776; Recovery = 79.4%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\050699\SPK5.D
Operator    . IG
Acquired    · 6 May 99  21 31      using AcqMethod 7281SIM
Instrument .    5973
Sample Name: spike cucumber 0 10ppm
Misc Info  ·
Vial Number  11
```



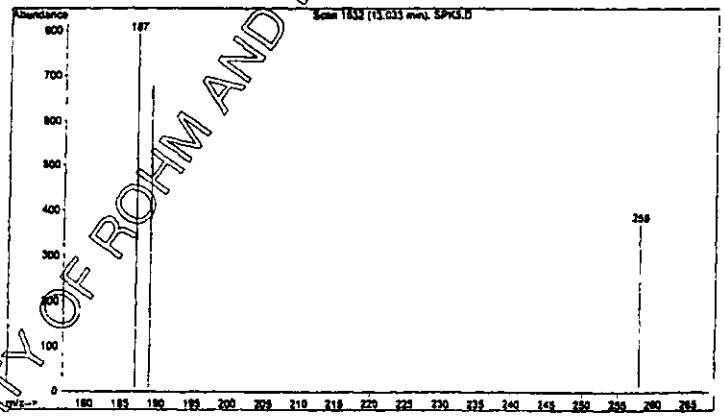
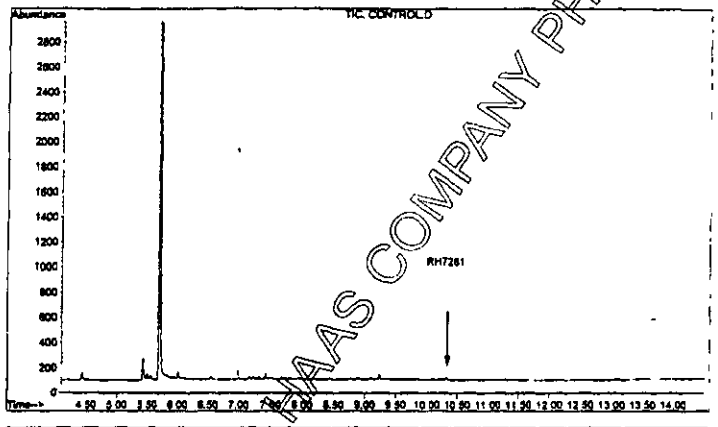


Figure 45 Fortification Cucumber at 0.10 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 6195; Recovery = 88 7%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\040199\CONTROL.D
Operator    -
Acquired    .  1 Apr 99  13 01      using AcqMethod 7281SIM
Instrument  ·     5973
Sample Name. control cantaloupe
Misc Info
Vial Number  6
```



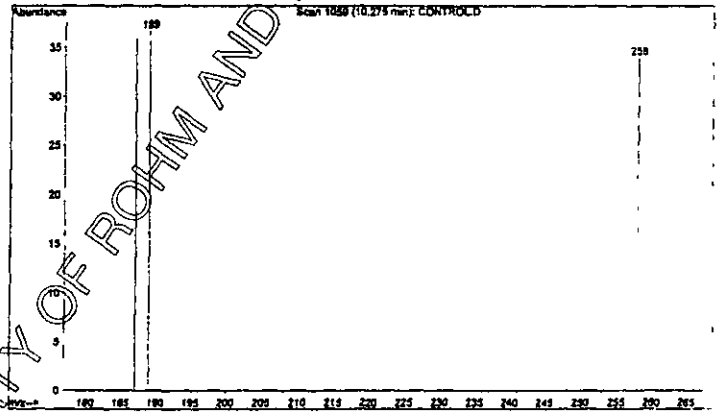
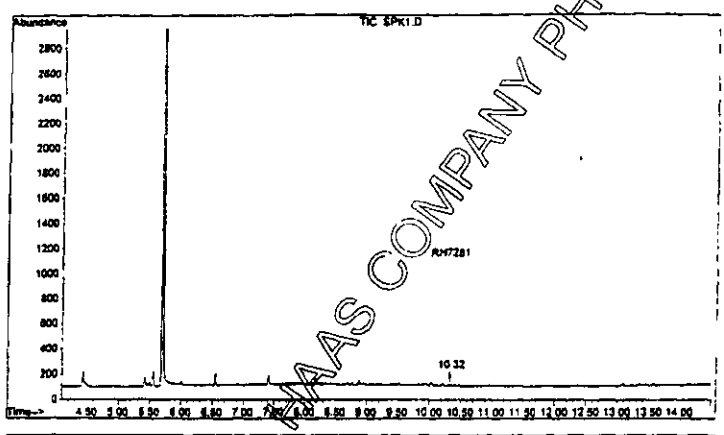


Figure 46 Control Cantaloupe (GC/MSD, TR 34-99-43, Peak Area = 0)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\040199\SPK1 D
Operator
Acquired       1 Apr 99  13 23      using AcqMethod 7281SIM
Instrument ·     5973
Sample Name  spike cantaloupe 0.01 ppm
Misc Info
Vial Number  7
```



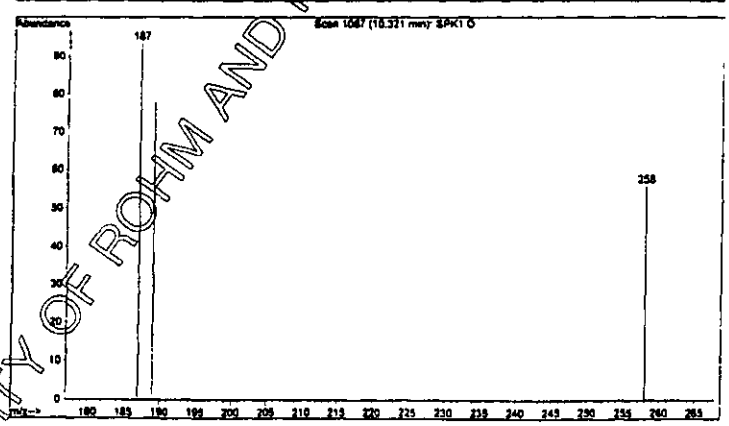
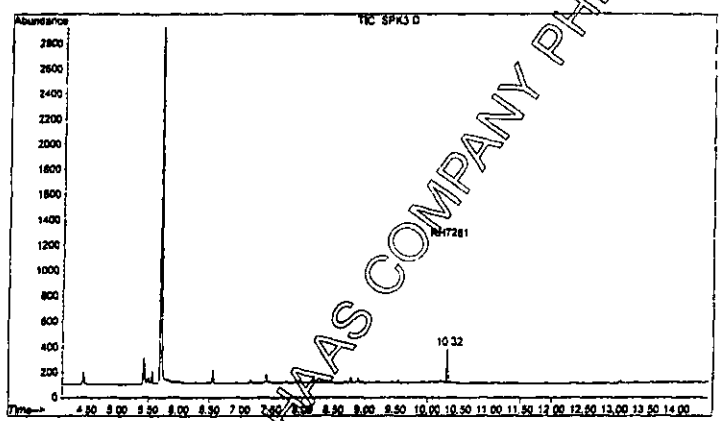


Figure 47. Fortification Cantaloupe at 0.010 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 361, Recovery = 74 2%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\1\DATA\040199\SPK3 D
Operator
Acquired    .  1 Apr 99  14 08      using AcqMethod 7281SIM
Instrument  :    5973
Sample Name  spike cantaloupe 0.02 ppm
Misc Info
Vial Number  9
```



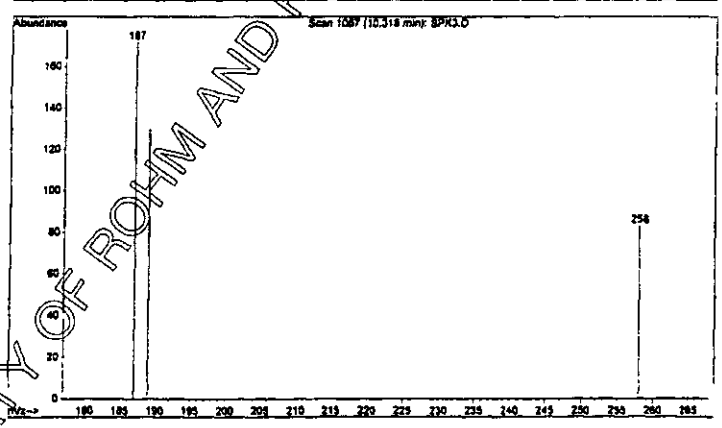


Figure 48 Fortification Cantaloupe at 0 020 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 757; Recovery = 77 8%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C.\HPCHEM\1\DATA\040199\SPK4 D
Operator
Acquired       1 Apr 99  14.30      using AcqMethod 7281SIM
Instrument :     5973
Sample Name  spike cantaloupe 0 05 ppm
Misc Info
Vial Number  10
```

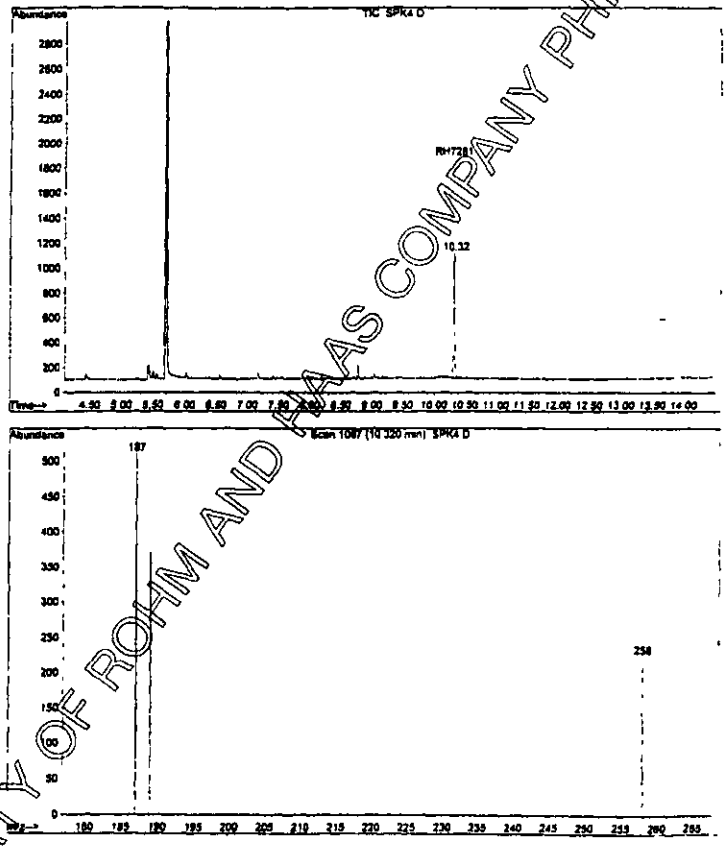


Figure 49. Fortification Cantaloupe at 0.050 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 2537, Recovery = 104 %

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\1\DATA\040199\SPK5 D
Operator
Acquired    :  1 Apr 99  14 53      using AcqMethod 7281SIM
Instrument .    5973
Sample Name  spike cantaloupe 0 10ppm
Misc Info   :
Vial Number: 11
```

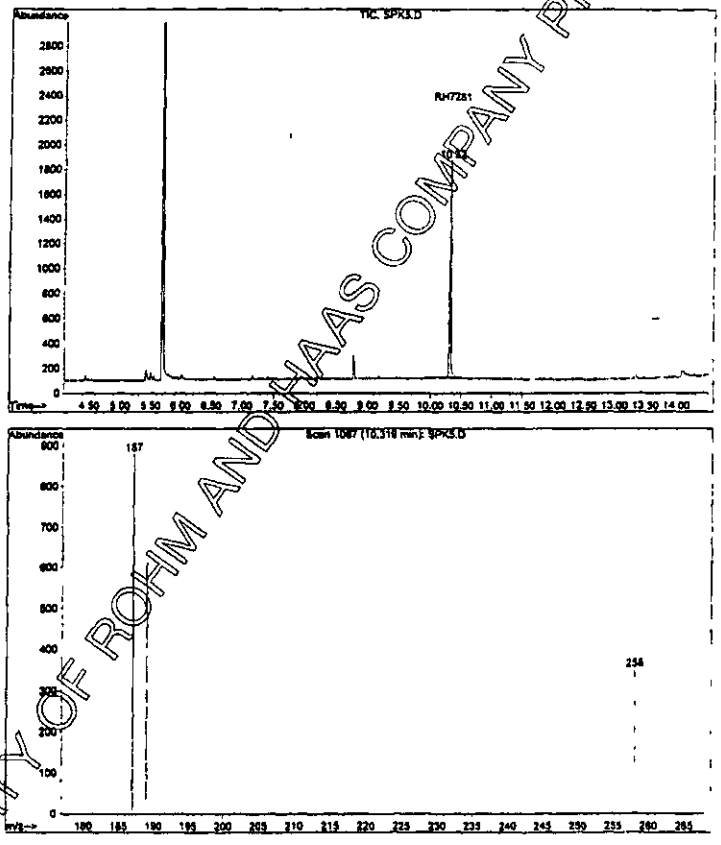
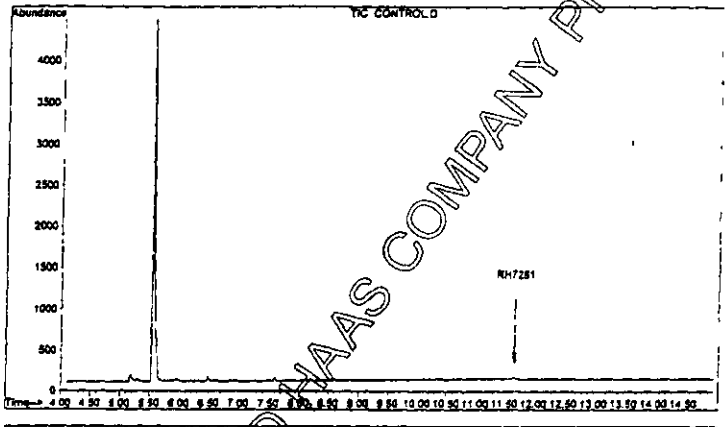


Figure 50 Fortification Cantaloupe at 0.10 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 4447; Recovery = 91 3%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        . C.\HPCHEM\1\DATA\042699\CONTROL D
Operator
Acquired      26 Apr 99  10:42      using AcqMethod 7281SIM
Instrument       5973
Sample Name: control zucchini
Misc Info   .
Vial Number  6
```



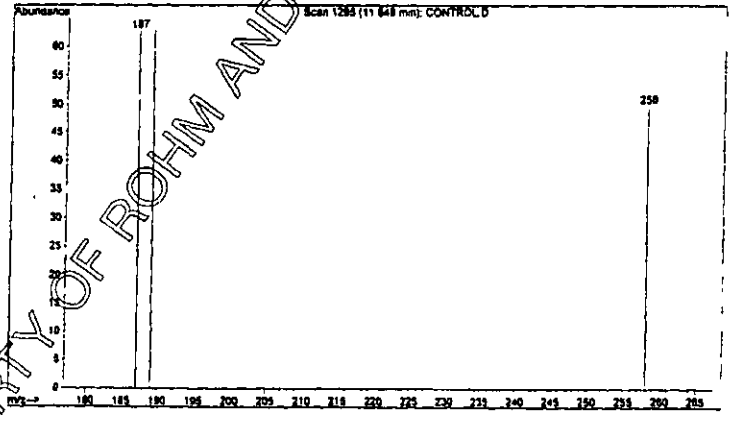
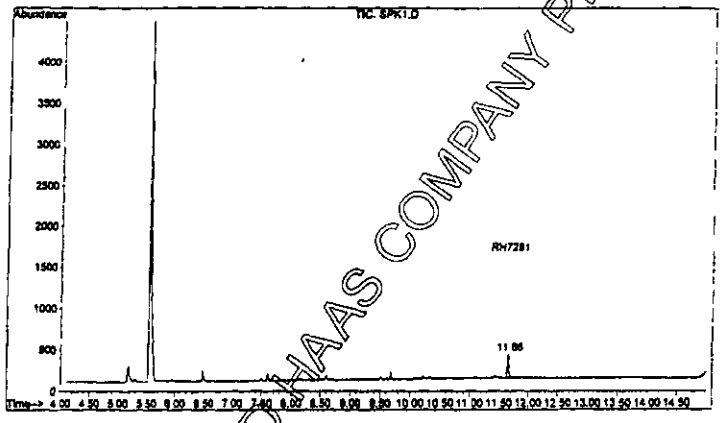


Figure 51 Control Zucchini (GC/MSD, TR 34-99-43, Peak Area = 0)

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C.\HPCHEM\1\DATA\042699\SPK1 D
Operator
Acquired      26 Apr 99  11:06      using AcqMethod 7281SIM
Instrument .     5973
Sample Name:  spike zucchini 0 01 ppm
Misc Info
Vial Number   7
```



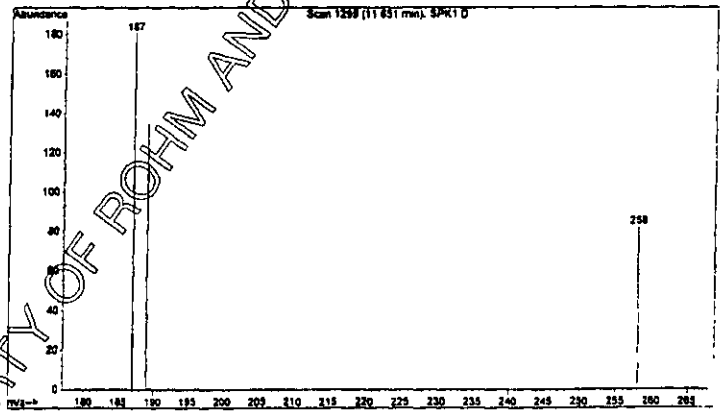
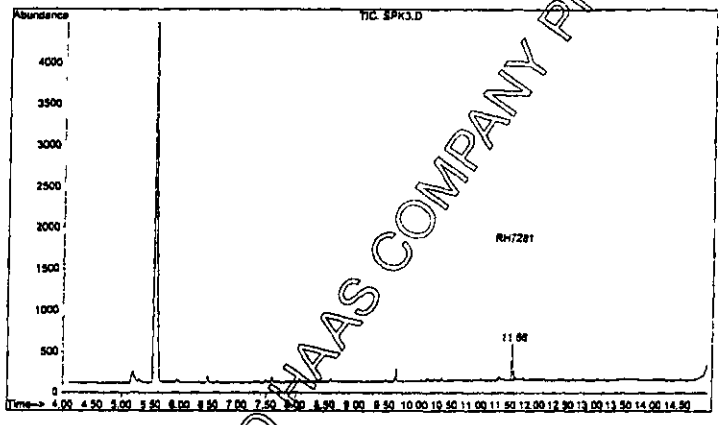


Figure 52 Fortification Zucchini at 0 010 µg/g (GC/MSD, TR 34-99-43); Peak Area = 777, Recovery = 88.2%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File          C \HPCHEM\1\DATA\042699\SPK3 D
Operator
Acquired      26 Apr 99  11 54      using AcqMethod 7281SIM
Instrument       5973
Sample Name.  spike zucchini 0.02 ppm
Misc Info
Vial Number   9
```



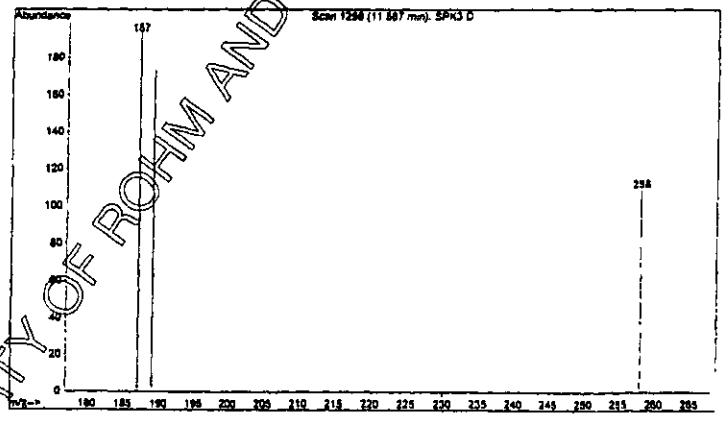
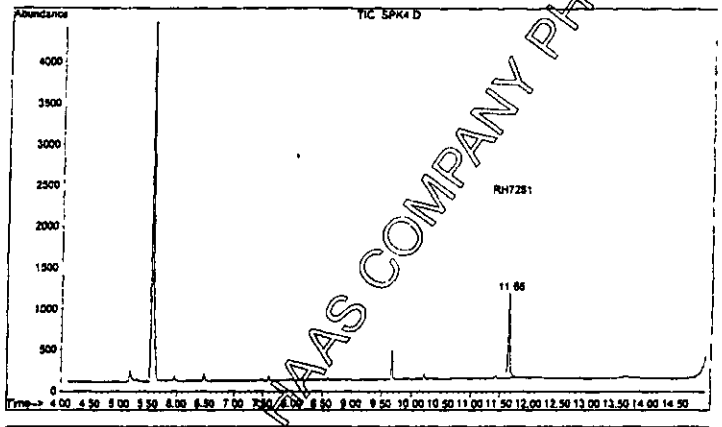


Figure 53 Fortification Zucchini at 0 020 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 1375, Recovery = 78 1 %

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\1\DATA\042699\SPK4 D
Operator
Acquired    : 26 Apr 99  12.18      using AcqMethod 7281SIM
Instrument      5973
Sample Name. spike zucchini 0.05 ppm
Misc Info
Vial Number: 10
```



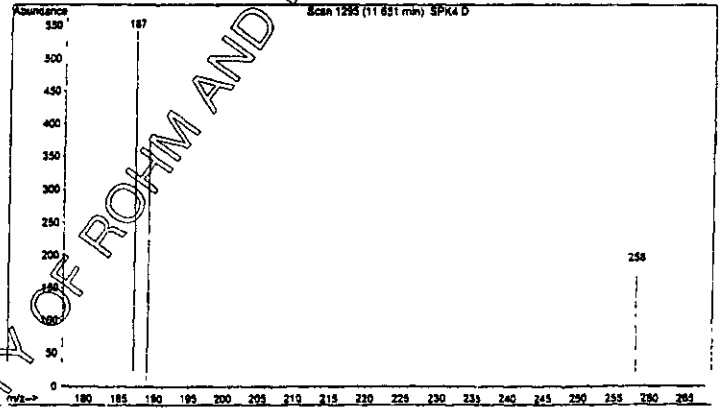
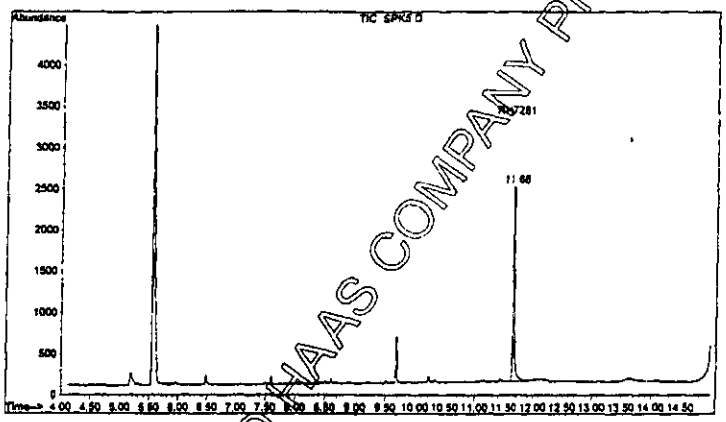


Figure 54 Fortification Zucchini at 0 050 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 3367, Recovery = 76 4%.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

```
File        : C \HPCHEM\1\DATA\042699\SPK5 D
Operator    :
Acquired      26 Apr 99  12:42      using AcqMethod 7281SIM
Instrument :    5973
Sample Name. spike zucchini 0.10ppm
Misc Info
Vial Number  11
```



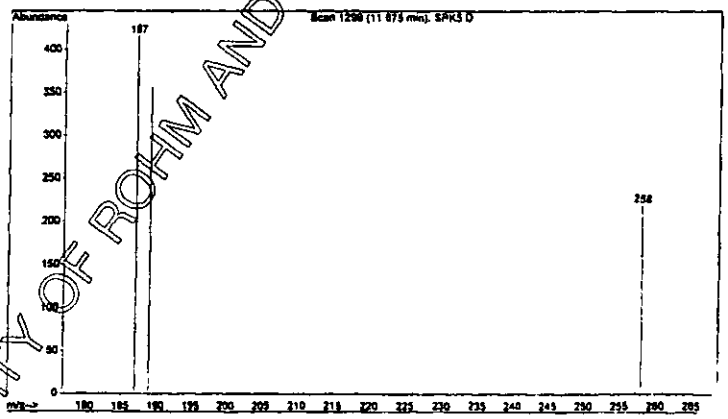


Figure 55. Fortification Zucchini at 0 10 µg/g (GC/MSD, TR 34-99-43)
Peak Area = 7515, Recovery = 85.3%

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Quantitation Report

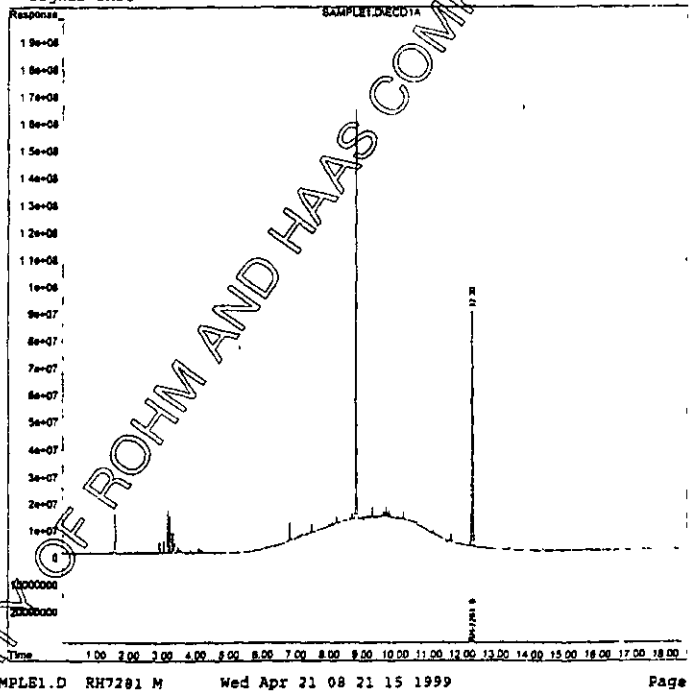
Data File C.\HPCHEM\2\DATA\cucvalid\SAMPLE1 D Vial 10
Acq On 4-20-99 9.37 46 PM Operator IG
Sample metabolism sample 1 Inst GC/ECD
Misc . Multiplier 1.00
Sample Amount 0 00

IntFile EVENTS.E

Quant Time Apr 21 8 21 1999 Quant Results File RH7281.RES

Quant Method C \HPCHEM\2\METHODS\RH7281 M (Chemstation Integrator)
Title 04-20-99
Last Update Wed Apr 21 08 13 12 1999
Response via Multiple Level Calibration
DataAcq Meth RH7281 M

Volume Inj.
Signal Phase
Signal Info



SAMPLE1.D RH7281 M Wed Apr 21 08 21 15 1999 Page 2

Figure 56. Treated Cucumber, Field Sample Replicate 1 (RAR 98-0280-002) RH-7281 = 1 12 µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Quantitation Report

Data File C:\HPCHEM\2\DATA\cucvalid\SAMPLE2 D
Acq On . 4-20-99 10:01 29 PM
Sample metabolism sample 2
Misc

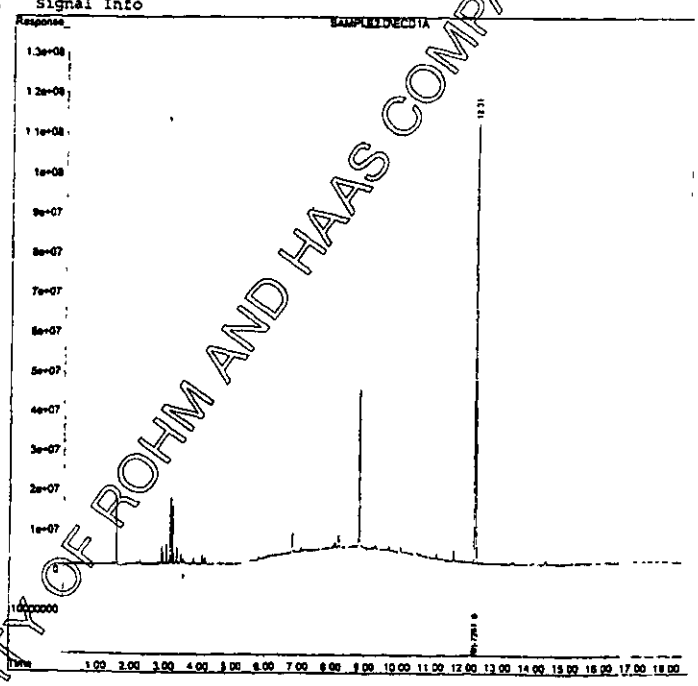
Vial 11
Operator
Inst GC ECD
Multiplr 1.00
Sample Amount 0.00

IntFile . EVENTS E

Quant Time Apr 21 8 21 1999 Quant Results File RH7281.RES

Quant Method . C \HPCHEM\2\METHODS\RH7281 M (Chemstation Integrator)
Title 04-20-99
Last Update Wed Apr 21 08 13.12 1999
Response via . Multiple Level Calibration
DataAcq Meth : RH7281 M

Volume Inj
Signal Phase
Signal Info



SAMPLE2 D RH7281.M Wed Apr 21 08 21 56 1999 Page 2

Figure 57. Treated Cucumber, Field Sample Replicate 2 (RAR 98-0280-002). RH-7281 = 1 44µg/g.

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

Quantitation Report

Data File : C \HPCHEM\2\DATA\cucvalid\SAMPLE3 D
Acq On : 4-20-99 10 25:11 PM
Sample : metabolism sample 3
Misc :

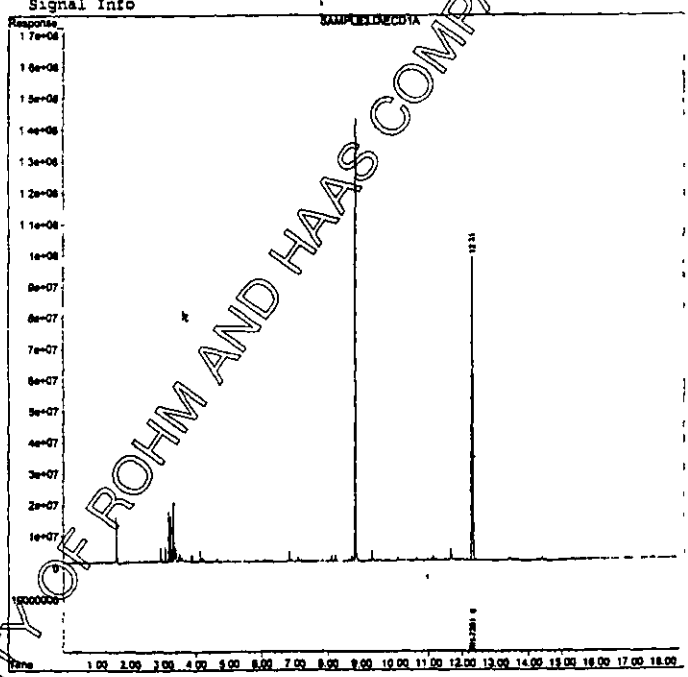
Vial 12
Operator IG
Inst GC \ECD
Multiplr 1 00
Sample Amount 10 00

IntFile . EVENTS.E

Quant Time. Apr 21 8.22 1999 Quant Results File· RH7281 RES

Quant Method : C \HPCHEM\2\METHODS\RH7281 M (Chemstation Integrator)
Title 04-20-99
Last Update . Wed Apr 21 08 13 12 1999
Response via · Multiple Level Calibration
DataAcq Meth : RH7281 M

Volume Inj. :
Signal Phase
Signal Info



SAMPLE3.D RH7281 M . Wed Apr 21 08:22:39 1999 Page 2

Figure 58 Treated Cucumber, Field Sample Replicate 3 (RAR· 98-0280-002)
RH-7281 = 1.28 µg/g

PROPERTY OF ROHM AND HAAS COMPANY PHILADELPHIA

EPA Addendum For Residue Analytical Method
PP# 0F6093
Zoxamide on Tomatoes and Cucumbers

The ACB made minor modifications to the GC-ECD instrument parameters:

GC-ECD - HP 6890 GC, equipped with an ECD Micro.
Injector Temperature - 150°C
Detector Temperature - 300°C
Column Flow - 1 mL/min He
Column: RTX-5 30 m x 0.25 mm id and 0.25 um film thickness.
Make-up gas. 95% Argon/5% Methane at 60 mL/min.

Temperature Program - 80°C for 1 minute, 20°C/min. to 260°C and hold 8 minutes; 30°C/min. to 280°C and hold 1 minute.