Document Cover Page

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STUDY TITLE

Enforcement Residue Analytical Method
for GOAL® Herbicide (oxyfluorfen) in Crop Commodities
with GC/MS Confirmation

DATA REQUIREMENT

Guideline 171-4(c)

STUDY DIRECTOR

D. A. Martin

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STUDY COMPLETED ON

4/29/96

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STUDY PROTOCOL

34P-95-92

LABORATORY PROJECT ID

Rohm and Haas Technical Report No. 34-95-111

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GLP Compliance Statement

The information contained in this report pertains to test method development and, as such, is not required to adhere to 40 CFR 160. The data, however, were compiled from the following:

- GLP compliant residue studies submitted to the EPA,
- additional fortification results for 3 of the above studies received after the submission due date, and,
- a new study conducted to validate the Oxyfluorfen (Goal®) Crop Residue Method, TR 34-94-150 using GC/MS as the confirmatory detection.

This report and work should be considered in compliance with Good Laboratory Practice standards as defined by EPA (40 CFR 160) with the following exceptions:

1. This report is a compilation of multiple studies by several study directors rather than a final report for a single study.
2. Additional fortification data for figs, olives, and pomegranates were obtained after submission of the respective final reports and were authorized by letter by the study director rather than via protocol amendment.

This report is a true and accurate representation of the work performed as attested to in previously submitted reports and by the undersigned.

Dennis A. Martin 4/29/96
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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 Products - Development Research Department and has been found to be a true and accurate reflection of the study performed.

Activity	Date Conducted	Date Reported[1]	QAU[2]
Additional work for submitted reports			
Figs			
Protocol Review	7/10/95	8/18/95 (7/20/95)	CAL
Extraction	7/11/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/7-8/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/18/95	12/18/95 (9/25/95)	CAL
Olives			
Protocol Review	7/7/95	8/18/95 (7/20/95)	CAL
Extraction	7/7/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/8/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/18/95	12/18/95 (9/25/95)	CAL
Pomegranates			
Protocol Review	7/10/95	8/18/95 (7/20/95)	CAL
Extraction	7/7/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/8/95	8/18/95 (8/16/95)	CAL
Raw Data Review	8/18/95	12/18/95 (9/25/95)	CAL
Peach report to be submitted			
PA Trial			
Inspection, harvest	8/17/94	8/18/94	R&H
Field report audit	7/21/95	7/25/95	R&H
Analytical subprotocol review	6/7/95	6/7/95	R&H
Inspections, analysis	7/6, 7/95	7/17/95 (7/12/95)	McK
Data audit, analysis	8/4/95	8/4/95 (8/4/95)	McK
GA Trial			
Field report audit	1/30/96	2/1/96	R&H
Inspections, analysis	11/21,22,28/95	12/11/95 (12/7/95)	McK
Inspections, logs	11/29/95	12/11/95 (12/7/95)	McK
Final report audit	4/1/96	4/1/96	R&H
Current study (34P-95-92)			
protocol review	10/17/95	10/17/95	R&H
protocol review	11/01/95	12/18/95 (11/30/95)	CAL
extraction	11/02/95	12/18/95 (11/30/95)	CAL
extraction	11/09/95	12/18/95 (11/30/95)	CAL
raw data review	12/06/95	2/02/96 (1/10/96)	CAL
Final report audits	3/20/96	3/20/96	R&H
	4/4/96	4/4/96	R&H
	4/26/96	4/26/96	R&H

1 Dates in parentheses pertain to notification of contract laboratory management.

2 CAL= Centre Analytical Laboratories
R&H= Rohm and Haas Company
McK= McKenzie Laboratories

H. E. Aller
QA Officer
Rohm and Haas Company

April 29, 1996
Date

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1. Objective

The original analytical method for crops, TR 34-94-150, was issued December 21, 1994 and later submitted to the EPA as Appendix 8 in MRID 42793300. This enforcement method is a reissue of the original with minor modifications, including the addition of GC/MS confirmatory detection.

2. Summary

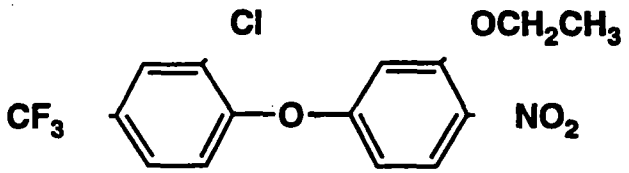
This report outlines the oxyfluorfen (parent only) residue analytical method for crop commodities. Oxyfluorfen is extracted from crops with acetonitrile. The residues are then further purified by liquid-liquid partition, silica gel or Florisil column chromatography and basic alumina solid phase extraction. Quantitation is performed by gas-liquid chromatography on a Restex Rtx-200 or Rtx-50 column with electron capture detection. Confirmatory detection is accomplished through GC/MS.

3. Introduction

Oxyfluorfen (GOAL® Herbicide) is a registered herbicide used on a wide variety of crops. In order to maintain the commercial registration, an analytical residue method for crops is required to generate residue data for the setting and enforcement of tolerances.

4. Experimental Compound

This analytical method is developed for oxyfluorfen (GOAL Herbicide), RH-32915, which has a CAS registry number 42874-03-3. The Chemical Abstracts name is 2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene. Its structure is shown below.



5. Chemicals, Supplies, Solutions

Chemicals and Supplies	Supplier *
Acetonitrile, HPLC Reagent	J. T. Baker
Cotton, sterile and absorbent	Johnson & Johnson
Ethyl Ether, HPLC Reagent	J. T. Baker

Ethyl Acetate, HPLC Reagent	J. T. Baker
Florisil, 60/100 mesh	J. T. Baker
Hexane, HPLC Reagent	J. T. Baker
Methanol, HPLC Reagent	J. T. Baker
Petroleum Ether, Omnisolv	EM Science**
RH-32915 Analytical Standard	Rohm and Haas
Silica Gel, 40-140 mesh	J. T. Baker Catalog # 3404-01
Sodium Chloride	J. T. Baker
Sodium Sulfate, anhydrous granular	Fisher
Supelclean LC-Alumina-B SPE tubes	Supelco Catalog # 5-7085
Toluene, HPLC Reagent	J. T. Baker
Water, Milli-Q	Millipore System

Solutions

Saturated sodium chloride aqueous solution

5/95 (v/v) ethyl ether/petroleum ether

30/70 (v/v) ethyl ether/petroleum ether

50/50 (v/v) ethyl ether/petroleum ether

40/60 (v/v) ethyl ether/petroleum ether

50/50 (v/v) ethyl acetate/hexane

5/95 (v/v) ethyl acetate/hexane

* Equivalent chemicals, equipment and instrumentation can be substituted, when shown to be appropriate.

** EM Science brand is recommended, for details see Section 10.

6. Equipment

Item	Supplier*
Buchner Funnel, porcelain	Coors
Chromatography Columns: Glass 250 mm length, 16 mm I.D.	Ace Glass
Centrifuge Bottles, 250 ml	Fisher
Centrifuge	Beckman
Filter Paper, Whatman #41	Whatman
G10 Gyrotory Shaker	New Brunswick Scientific
Pipets, glass disposable, 10 ml, 5 ml, 1 ml	Fisher
Rotary Evaporator, with dry ice trap	Brinkman
Round Bottom Flask (100, 250, 500 ml, 24/40 ST)	Kimax, Pyrex
Test tubes, 40 ml 12.0 mm I.D.	Kontes
Separatory Funnels, 500 ml	Kimax, Pyrex
Standard laboratory equipment, balance, beakers, etc.	
Visiprep Solid Phase Extraction Vacuum Manifold - 60 ml reservoirs - adapter for sample reservoirs	Supelco Catalog # 5-7044
Volumetric Flasks (100ml)	Pyrex

* Equivalent chemicals, equipment and instrumentation can be substituted, when shown to be appropriate.

7. Method

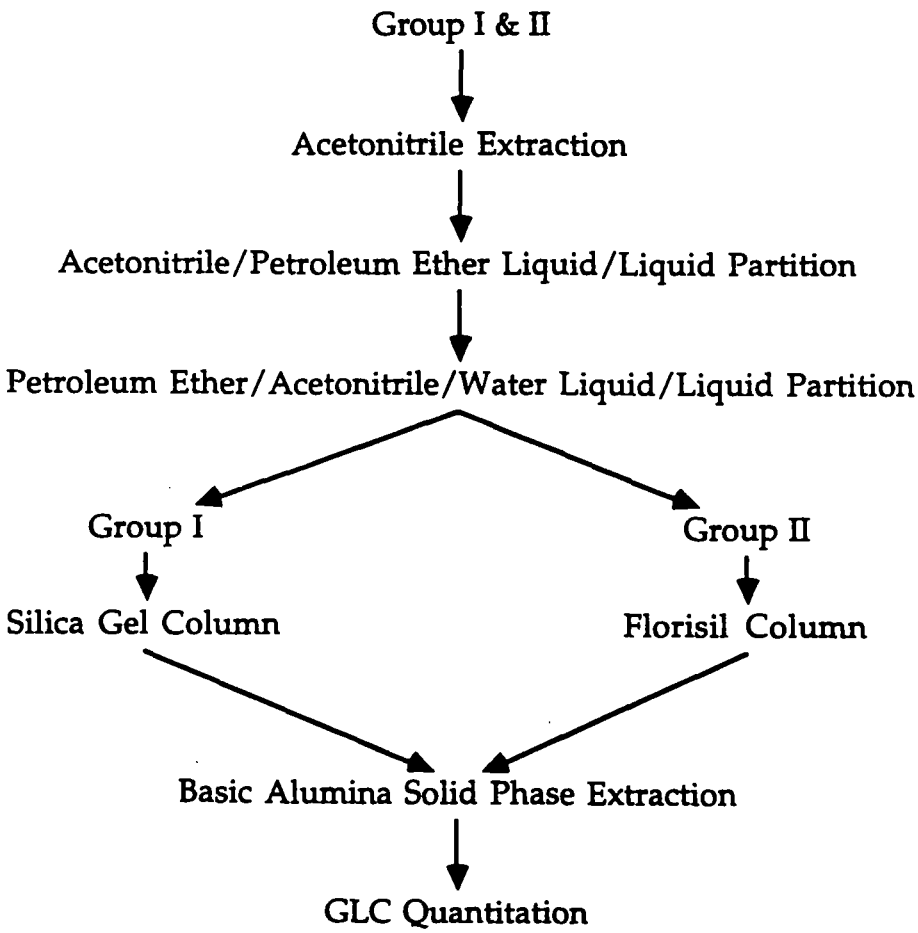
The crop commodities analyzed with this method can be separated into two groups based on the path of analysis. They are designated Group I and Group II for ease in detailing the differences in the methodolgy, specifically during the Column Chromatography. The groups are:

Group I: apple, artichoke, avocado, cherry, fig, grape, kiwi, olive, peach, peanut nutmeat, pomegranate, soybean

Group II: peanut hay, shell, vine

7.1 Flow Diagram

Below is a flow diagram giving a general description of the method. The individual steps are detailed following the diagram.



7.2 Sample Processing

All crop samples are finely and homogenously chopped in preparation for sample extraction. *Apple, artichoke, fig, grape, kiwi, peanut hay, peanut nutmeat, pomegranate,* and *peanut vine* samples are chopped with dry ice in a Hobart food chopper. For *avocado, cherry, peach,* and *olive* samples, the pits are removed prior to processing with dry ice in a Hobart food chopper. Any suitable commercial food chopper can be substituted if a Hobart is not available. All samples with dry ice are left open and allowed to sublime overnight in the freezer. *Peanut shell* and *soybean* samples are processed dry in a Wiley Mill. All samples are maintained frozen prior to analysis.

7.3 Sample Extraction

All the glassware and centrifuge bottles must be pre-washed with methanol twice before any experiment.

For all crops, weigh a homogenized 5.0 g sample of the crop into a 250 ml centrifuge bottle; and, fortify as needed for fresh spikes. Add 100 ml of acetonitrile and then homogenize with a Gyrotory Shaker at 600-800 rpm for 20 min. Centrifuge each extraction mixture at 5000 rpm for 20 minutes at 4°C*. Decant the supernatant into a 500 ml separatory funnel. Add another 100 ml of acetonitrile to the centrifuge bottle containing the crop pellet, then homogenize with the Gyrotory Shaker at 600-800 rpm for another 20 minutes and centrifuge again as described above. Decant the supernatant into the 500 ml separatory funnel with the first acetonitrile extract. Add 200 ml petroleum ether to the 500 ml separatory funnel containing the 200 ml acetonitrile extract. Shake for 20 seconds and vent into a fume hood. Allow layers to separate and drain the acetonitrile (lower) phase into a 500 ml 24/40 ST round bottom flask. Discard the petroleum ether layer to waste.

Evaporate the acetonitrile gently to approximately 50 ml by rotary evaporator at 60°C under diminished presure. Transfer the acetonitrile extract to a new 500 ml separatory funnel for the next step, liquid/liquid partition.

* Option: Filter the samples under suction with a vacuum pump (about 50 mm Hg) through a Buchner funnel with Whatman #41 filter paper which has been prewashed with acetonitrile and dried.

7.4 Liquid/Liquid Partition

For all crops, add 100 ml petroleum ether to the 500 ml round bottom flask which previously contained the acetonitrile extract. Transfer the petroleum ether to the 500 ml separatory funnel containing the acetonitrile extract. Add 10 ml of saturated NaCl solution and 200 ml water to the separatory funnel. Shake vigorously for 2 minutes and vent into a fume hood. Allow the layers to separate and drain the acetonitrile/water (lower) phase and reserve for further partitioning. Then collect the petroleum ether phase in a new 500 ml 24/40 ST round bottom flask. Add another 100 ml petroleum ether to the 500 ml flask that contained the acetonitrile extract, transfer to the separatory funnel and partition the reserved acetonitrile/water phase again, as described above.

Rinse the separatory funnels with methanol, discarding to waste, and allow the funnels to dry. Transfer the petroleum ether fractions to the separatory funnel and then partition with 200 ml water. Shake for one minute, allow layers to separate and drain the water (lower) phase to waste. Collect the petroleum ether layer into a 500 ml 24/40 ST round bottom flask. Evaporate gently to approximately 5 ml of petroleum ether by rotary evaporator at 40°C under diminished pressure. The sample extract is then ready for the next step, column chromatography.

7.5 Column Chromatography

Group I follows the Silica Gel Open Column step. Group II follows the Florisil Open Column step.

Silica Gel Open Column (Group I: apple, artichoke, avocado, cherry, fig, grape, kiwi, olive, peach, peanut nutmeat, pomegranate, soybean)

Activate the silica gel, mesh 40-140, by heating for 24 hours at 200°C. Remove from the oven and store in tightly capped jars in a desiccator. Pack a 250 mm X 16 mm I.D. glass column plugged with cotton with 20 ml of the activated silica gel. Top the column with 10-15 g (approx. 2-3 cm column high) of anhydrous granular sodium sulfate. Precondition the column with 30 ml of petroleum ether.

Add the 5 ml of the remaining petroleum ether extract of the sample, section 6.4, to the column and elute to the top of the silica gel bed. Add 50 ml of petroleum ether to the 500 ml round bottom flask, transfer to the column, and elute to the top of the silica gel bed. Add 20 ml of 95/5 (v/v) petroleum ether/ethyl ether to the 500 ml round bottom flask, transfer to the column, and elute to the top of the silica gel bed. Discard all washes to this point.

Elute the oxyfluorfen by adding 75 ml of 60/40 (v/v) petroleum ether/ethyl ether to the 500 ml round bottom flask, transferring to the column, and eluting to the top of the silica gel bed. Collect the eluate in a 250 ml 24/40 ST round bottom flask. Evaporate to dryness at 45 °C under diminished pressure. Redissolve the residue in 5 ml of hexane. The sample is now ready for the next step, solid phase extraction.

Florisil Open Column (Group II: peanut hay, shell, vine)

Activate the Florisil, mesh 60-100, by heating for 24 hours at 150°C. Remove from the oven and store in tightyly capped jars in a desiccator. Pack a 250 mm X 16 mm I.D. glass column plugged with cotton with 20 ml of the activated Florisil. Top the column with 10-15 g (approx. 2-3 cm column high) of anhydrous sodium sulfate. Precondition the column with 30 ml of petroleum ether.

Add the 5 ml of the remaining petroleum ether extract of the sample, section 6.4, to the column and elute to the top of the Florisil bed. Add 50 ml of petroleum ether to the 500 ml round bottom flask, transfer to the column, and elute to the top of the Florisil bed. Add 20 ml of 70/30 (v/v) petroleum ether/ethyl ether to the 500 ml round bottom flask, transfer to the column, and elute to the top of the Florisil bed. Discard all washes to this point.

Elute the oxyfluorfen residue by adding 50 ml of 50/50 (v/v) petroleum ether/ethyl ether to the 500 ml round bottom flask, transferring to the column, and eluting to the top of the Florisil bed. Collect the eluate in a 250 ml 24/40 ST round bottom flask. Evaporate to dryness at 45 °C under diminished pressure. Redissolve the residue in 5 ml of hexane. The sample is now ready for the next step, solid phase extraction.

7.6 Basic Alumina Solid Phase Extraction (SPE)

Connect a 6 ml Basic Alumina SPE tube with a 60 ml reservoir using an adapter to a Supelco Visiprep Solid Phase Extraction Vacuum manifold or similar apparatus. Precondition the 6 ml Basic Alumina SPE tube with 10 ml ethyl acetate followed by 10 ml hexane. Flow rate through the column should be 2-3 mls per minute. For all crops, add the sample in 5 ml hexane to the column, allow it to elute until the sample is 2 mm above the top of the absorbent bed. Rinse and wash the sample flask 3-4 times with a total 40 ml of hexane, add to the column, and elute as described above. Discard all the hexane eluent at this point.

Elute with 40 ml 50:50 (v/v) ethyl acetate/hexane solution*. Collect this fraction in a 40 ml (12 mm ID) test tube. Transfer the eluate into a 100 ml 24/40 ST round bottom flask. Rinse the tube with hexane several times (5-10 ml) and transfer to the round bottom flask as well. Evaporate to dryness at 45 °C under diminished pressure. Dissolve the sample in 5 ml toluene. The sample is now ready for quantitation by GLC.

*Option: Elute oxyfluorfen with 25 ml 5:95 (v/v) ethyl acetate/hexane solution.

8. Primary Detection Method: GC/ECD Quantitation

8.2. Preparation of Standards

The concentration of standards should be corrected for % purity of analyte.

Prepare a 200 ppm stock solution by weighing 20 mg of RH-32915, oxyfluorfen, into a 100 ml volumetric flask and bringing to volume with toluene. Then, make up a 10 ppm stock solution by adding 5.0 ml of the 200 ppm solution into a 100 ml volumetric flask and bringing to volume with toluene. Standard solutions of RH-32915 in toluene are prepared by serial dilution from the 10 ppm stock solution in the concentration range of 0.005 µg/ml - 1.0 µg/ml.

8.1. Instrumentation

During method development and the residue analyses included in this report a Varian 3500 Capillary Gas Chromatograph equipped with a Varian Model 8100 Auto Sampler and an Electron Capture Detector was used. Data were obtained with an HP 300 Data Acquisition System with Hewlett-Packard Extrachrom Software. Data were processed by Nelson Analytical Software.

Two analysis GLC columns can be used. Column 1 is the primary analytical column, and column 2 is the secondary confirmatory column.

Column 1, Restex Rtx-200:	0.32 mm I.D., 60 m, 1.0 µ film Catalog # 15057
Guard Column:	Restex deactivated, uncoated fused silica gel, 5 m, 0.53 mm I.D., Catalog # 10045
Column Connection:	Supelco Glass Seal Connector. Catalog #2-0479

Gas Flows: Nitrogen (makeup) 40 ml/min
Helium (column) 3.5 ml/min

Injection Volume: 1 µl

Temperatures: Injector 265°C
Detector 270°C
Column 215°C
Initial Hold 1.0 min

Final Temp	Rate (°C/min)	Hold (min)	Total Time(min)
250°C	10.0	17.0	21.5

Electron Capture Detection:	Attenuation	Range
	32	10

Under the conditions mentioned above, the typical retention time is 16.1 min. Conditions may need to be adjusted to achieve the optimum response of the instrument.

Column 2. Restex Rtx-50: 0.32 mm I.D., 60 m, 1.0 µ film
Catalog #10557

Guard Column: Restex deactivated, uncoated fused silica gel, 5 m, 0.53 mm I.D., Catalog # 10045

Column Connection: Supelco Glass Seal Connector. Catalog #2-0479

Gas Flows: Nitrogen (makeup) 40 ml/min
Helium (column) 3.5 ml/min

Injection Volume: 1 µl

Temperatures: Injector 265°C
Detector 270°C

Column 215°C
Initial Hold 1.0 min

Final Temp	Rate (°C/min)	Hold (min)	Total Time (min)
250°C	5.0	25.0	33.0

Electron Capture Detection:	Attenuation	Range
	32	10

Under the conditions mentioned above, the typical retention time is 22.5 min. Conditions may need to be adjusted to achieve the optimum response.

8.3 Quantitation

Standard solutions are prepared in the concentration range of 0.005 µg/ml - 0.15 µg/ml. The standards and samples are injected at the same volume, 1 µl. If necessary, the sample is diluted to an appropriate volume to give a response within the standard curve range. Standard curves are prepared for each analysis day. Each analytical run should have two sets of standards. The standard curve can be constructed using linear or quadratic regression, if needed.

The peak height is measured and the concentration is determined from the standard curve. The component residue concentration is determined as follows: (See example shown in Section 8.4 Fortification)

$$\frac{\text{Final Sample Volume (ml) X Component Concentration (µg/ml)}}{\text{Sample Weight (g)}} = \text{ppm}$$

8.4 Fortification

For samples fortified prior to extraction, with known amounts of RH-32915, measure the peak height, determine the µg/ml from the standard curve, and calculate % recovery from the equation below.

$$\frac{[\text{µg/ml found X Final Sample Volume (ml)}]}{\text{fortification (µg added)}} \times 100 = \% \text{ Recovery}$$

Example (taken from analytical run in Appendix II)

Data file: L26JA6	Sample: 0.01 ppm fortification
Peak Height: 249	Final Volume: 5 ml
Sample Weight: 5 g	µg added: 0.05 µg

µg/ml found= 0.0088 (calculated from curve, $HEIGHT=B0 + B1X + B2X^2$)

B0=75.758, B1=19035, B2=72899, HEIGHT=249

Substituting into the above equation:

$$\frac{[0.0088 \times 5]}{0.05} \times 100 = 88.0\%$$

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9. Confirmatory Detection Method: GC/MS Quantitation

In order to confirm that residues found using the procedure described previously in this report are in fact oxyfluorfen, RH-32915, residues, a confirmatory detection method was established using GC/MS with selected ion monitoring. This method has a Limit of Quantitation of 0.025 ppm. Analytical results are shown in Table 18. Representative chromatograms are shown in Figures 76-96.

The detailed instrument information and conditions is given below:

Instrumentation:	Hewlett-Packard model 5890 Series II GC; model 5972 mass selective detector
MS/Selected Ion Monitoring:	252 molecular weight ion Dwell Time: 100 msec Ionization Potential: 70 eV Electron Multiplier Voltage: 1900-2318 V
Column:	Rtx-50, 0.25 mm i.d. X 30 m, 0.5 µm film thickness
Oven Temps.:	Hold at 100°C for 2 min. 100°-270°C at 10°C/min. Hold for 15 min.
Retention Time:	19.6 minutes
Injector Temp.:	280°C
Detector Temp.:	300°C
Carrier Gas/ Flow Rate:	Helium/2.18 ml/min.
Head Pressure:	20 psi
Injection Mode:	splitless
Injection Liner:	silanized single taper
Injection Purge Delay:	1.0 min.
Septum Purge:	2 ml/min.
Injection Volume:	2 µl

10. Results

The results detailed in this method are covered in various reports and protocols. All reports used are listed in Section 12. Project Information. Many of these reports have been submitted and accepted by the EPA previously. Distinctions in individual results are made on the respective recovery tables, Tables 1-18.

For figs, olives, and pomegranates, there are a few fortifications reported here that were not reported in their respective technical reports, which were already submitted and accepted. Due to the timeline established for submission of those reports, the results were received too late to be included in them (refer to Tables 5, 8, and 11 for identification of data).

Six recoveries have been designated as outliers and are not included in the final calculation of both the average and standard deviation (see Tables 1, 7, 10, 14, 17). These points did not fall within two standard deviations when used in the calculation. It should also be noted that throughout all crop commodities the control samples did not have any detectable residues, that is above 0.003 ppm.

In GC/ECD, the limit of quantitation (LOQ) for all crop commodities except for peanut vine, shell, and hay is 0.01 ppm established in TR 34- 94-150, Appendix 8 in MRID 42793300, and also by fortifications at this level, with a limit of detection of 0.003 ppm. For peanut vine, shell, and hay the limit of quantitation is 0.02 ppm, established by fortifications at this level, with a limit of detection of 0.007 ppm. Table 19 summarizes the LOQ and LOD values, along with average recoveries for each crop.

Tables 1-12 summarize the fortification data for the crop commodities detailed in this method as Group I. This data was obtained with GC/ECD detection using a Rtx-200 column. Average recoveries ranged from 85.4% in apple to 104.9% in soybean.

Tables 13-15 summarize the fortification data for the crop commodities detailed in this method as Group II. This data was also obtained with GC/ECD detection using a Rtx-200 column. Average recoveries were 92.2% in peanut hay, 95.3% in peanut shell, and 81.8% in peanut vine.

Tables 16 and 17 detail the results obtained from grape and soybean, Group I, analyses on the Rtx-50 confirmatory column. Recoveries averaged 92.6% in grapes and 104% in soybean.

In GC/MS, the limit of quantitation for all crop commodities is 0.025 ppm, established by fortifications at this level. The limit of detection is 0.008 ppm.

Table 18 displays the results of analyses of crops using GC/MS as the confirmatory detection method compared to analysis on GC/ECD. Individual recoveries ranged from 81% in peanut nutmeat to 117% in peanut shell.

Table 19 is a summary of the results for all crops detailed in this method, including average recovery, LOQ, and LOD values.

Representative chromatograms are shown in Figures 1-95, including standards, controls and three fortifications (alphabetical within each group). Figures 1-48 show the crop commodities identified as Group I; those from Group II are shown in Figures 49-60. Figures 61-68 are representative of the grape and soybean analyzed on the Rtx-50

confirmatory column. Figures 69-74 are representative chromatograms of standards on the GC/ECD. Figures 75-95 are representative of the GC/MS analysis of designated crop commodities.

NOTE: Representative complete analytical runs, including a run sheet and standard curve for both GC/ECD and GC/MS, can be found in Appendix 2.

11. Discussion

This method is intended to replace the current enforcement method for oxyfluorfen in crop commodities. As detailed in this report, a significant amount of analytical data was obtained in support of this method. This method effectively and consistently extracts and quantifies oxyfluorfen residues. The results show both methods of detection, GC/ECD and GC/MS, to be comparable in the recovery of oxyfluorfen residues. A comparison to the current enforcement method follows.

The extraction is similar to the current enforcement method, as published in PAM vol. II, method II. The techniques are comparable including similar solvents. Although blending is not used in this proposed enforcement method, the procedure using a shaker and centrifuge have proven to be effective in the other GOAL methods described below. Since the procedures are very similar we again conclude that the proposed extraction method provides an efficient mechanism in analyzing GOAL residues in crop commodities.

The extraction methodology found in this method is similar to that in both the "Oxyfluorfen (GOAL®) Meat and Fat Analytical Method" (TR 34-93-72) and the "Oxyfluorfen (GOAL®) Soil Analytical Method" (TR 34-94-47). It was shown to be quite efficient (~90%) at extracting GOAL residues via 14C-labeled matrices in the previously mentioned methods. We conclude that the same outcome is expected of the crop residue samples.

Below are some points to be taken into consideration when performing this method:

- The Rtx-200 and Rtx-50 capillary columns should be conditioned according to the manufacturer's instructions. It was also necessary to prime the column before analysis by making several injections of standards.
- The purity of the petroleum ether can be critical. A self inspection of the solvent being used can be made by the following procedure: rotovap 200 ml of petroleum ether to dryness, then add 5 ml hexane. Inject the hexane extract on the GC with the same conditions as a sample. If the chromatograms give interfering peaks, this batch of petroleum ether cannot be used in the study. Therefore, a substitute for this product from another manufacturer or another batch from the same manufacturer must be used.

- In certain grape matrices, the pellet after the first centrifuge step may adhere very tightly to the side of the centrifuge bottle. If this does occur, it may be necessary to break it apart for the second extraction step to result in optimum performance.

In conclusion, this method is both robust and effective in the extraction of oxyfluorfen residues from crop commodities as written; and, it should be transferrable to other crops as needed.

12. Project Information

Title: Enforcement Residue Analytical Method for GOAL® Herbicide (oxyfluorfen) in Crop Commodities with GC/MS Confirmatory Detection

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100 Independence Mall West, Philadelphia, PA, 19106-2399

Testing Laboratories:

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3048 Research Drive, State College, PA 16801

McKenzie Laboratories
3725 E. Atlanta Ave., Suite One, Phoenix, AZ 85040

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

Study Director: Dennis A. Martin, B.S.

Study Protocol: 34P-95-92, "Method Validation- GC/MS Confirmatory Detection in Oxyfluorfen (Goal®) Crop Residue Analytical Method, TR 34-94-150"

Principal Investigators:

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Archives:

The Rohm and Haas Company, Agricultural Products Development Department Archives at Spring House

Data Handling:

Data system used was the Nelson Analytical, Inc. system with Version 1.2 of the 4400 Chromatography Software as its basis. This software provides the following functions:

- automatic collection and processing of chromatographic data
- detection and integration of peaks for chromatographic standards, samples and fortifications
- calculation and display of summary information for each standard, sample and fortification run
- display results from reports on the Laserjet printer
- editing and insertion of the summary data into IBM System 2000 data base

Report References:

Title	Rohm and Haas TR No.	MRID No.
Rohm and Haas Company Response to EPA CBTS May 12, 1994 Review of Residue Chemistry Data Supporting the Petition For Permanent Tolerances for Oxyfluorfen in Peanuts PP# 3F04229/FAP# 3H5674	34-95-74	Appendix 4 in 42793300
Oxyfluorfen Crop Residue Method	34-95-150	Appendix 8 in 42793300
Oxyfluorfen Residues in Peanut Nutmeat, Vine, Shell, and Hay	34-95-51	Appendix 9 in 42793300
Oxyfluorfen Residues in Apple	34-95-113	43794001
Oxyfluorfen Residues in Avocado	34-95-115	43794002
Oxyfluorfen Residues in Fig	34-95-116	43794003
Oxyfluorfen Residues in Pomegranate	34-95-117	43794004
Oxyfluorfen Residues in Kiwi	34-95-118	43794005
Oxyfluorfen Residues in Olive	34-95-119	43794006
Oxyfluorfen Residues in Artichoke	34-95-120	43794007
Oxyfluorfen Residues in Cherries	34-95-121	43794008

Oxyfluorfen Residues in Peach	34-95-114	N/A*
Oxyfluorfen Residues in Grape	34-95-104	N/A*
Oxyfluorfen Residues in Non-Dormant Grape (non-CA trials); Supplemental to TR 34-95-104	34-95-173	N/A*

* N/A means that these reports were not submitted to the EPA as of the study completion date of this report.

Notebook References:

QZ04	Rohm and Haas No: 060662
QZ05	Rohm and Haas No: 061114
QZ06	Rohm and Haas No: 062196
QZ07	Rohm and Haas No: 062755
QZ08	Rohm and Haas No: 063646
KRK15	Rohm and Haas No: 064107

Reviewed and Edited By: Juan Chen, Rohm and Haas Company

Table 1. Apple Oxyfluorfen Fortification Summary [1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0129-001	0.01	80.9
94-0130-001	0.01	81.1
94-0130-001	0.01	85.2
94-0130-001	0.01	88.8
95-1408-003	0.01	88.5
94-0152-001	0.01	76.5
94-0130-001	0.02	80.0
94-0130-001	0.05	90.8
94-0129-001	0.10	82.3
94-0130-001	0.10	89.7
94-0130-001	0.10	93.4
94-0152-001	0.10	40.2 [2]
95-1408-003	0.10	89.3
95-1408-003	0.50	91.7
94-0129-001	0.50	89.7
94-0130-001	0.50	83.5
94-0152-001	0.50	74.2
	Average n=16	85.4
	Standard Deviation	5.7

[1] All data reported in this table is taken from MRID 43794001.

[2] Value outside 2 x SD, therefore not included in statistics.

Table 2. Artichoke Oxyfluorfen Fortification Summary[1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0060-001	0.01	82.1
94-0061-001	0.01	99.0
94-0060-001	0.01	78.2
94-0060-001	0.01	68.3
94-0060-001	0.01	75.1
94-0060-001	0.02	104.0
94-0060-001	0.05	97.6
94-0060-001	0.10	104.0
94-0061-001	0.10	127.0
94-0060-001	0.50	99.4
94-0061-001	0.50	99.4
	Average n=11	94.0
	Standard Deviation	16.7

[1] All data reported in this table is taken from MRID 43794007.

Table 3. Avocado Oxyfluorfen Fortification Summary[1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0141-001	0.01	51.4
94-0141-001	0.01	52.3
94-0141-001	0.01	71.7
94-0141-001	0.01	105.0
94-0141-001	0.01	108.0
94-0141-001	0.02	109.0
94-0141-001	0.02	111.0
94-0141-001	0.05	112.0
94-0141-001	0.10	100.0
94-0141-001	0.10	104.0
94-0141-001	0.50	92.8
94-0141-001	0.50	110.0
	Average n=12	93.9
	Standard Deviation	22.6

[1] All data reported in this table is taken from MRID 43794002.

Table 4. Cherry Oxyfluorfen Fortification Summary[1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
92-0041-002	0.01	73.8
92-0041-002	0.01	75.1
92-0041-002	0.01	76.1
92-0041-002	0.01	72.5
92-0042-002	0.01	75.9
92-0041-002	0.02	96.5
92-0041-002	0.05	109.0
92-0041-002	0.10	90.4
92-0042-002	0.10	101.0
92-0041-002	0.50	87.8
92-0042-002	0.50	93.6
	Average n=11	86.5
	Standard Deviation	12.6

[1] All data reported in this table is taken from MRID 43794008.

Table 5. Fig Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0142-001	0.01 [1]	90.1
94-0142-001	0.01	88.0
94-0142-001	0.01	86.6
94-0142-001	0.01	84.1
94-0142-001	0.01	80.6
94-0142-001	0.02	103.0
94-0142-001	0.02	99.5
94-0142-001	0.05	97.8
94-0142-001	0.10	88.1
94-0142-001	0.1 [1]	98.5
94-0142-001	0.50	93.6
94-0142-001	0.5 [1]	88.8
	Average n=12	91.6
	Standard Deviation	6.9

[1] This data is taken from MRID 43794003.

All other data collected under 34P-95-30A.

Table 6. Grape Oxyfluorfen Fortification Summary[1]

Variety	RAR/ Sample No.	Fortification Level, ppm	% Recovery
Thompson	93-0012-001	0.01	70.5
	93-0012-001	0.01	71.8
	93-0012-001	0.01	74.7
	93-0012-001	0.01 [1]	90.7
	93-0012-001	0.01 [1]	91.8
	93-0012-001	0.01 [1]	85.6
	93-0012-001	0.01 [1]	84.4
	93-0012-001	0.05	76.8
	93-0012-001	0.10	81.3
	93-0012-001	0.1 [1]	78.1
	93-0012-001	0.1 [1]	84.4
	93-0012-001	0.1 [1]	78.1
	93-0012-001	0.1 [1]	85.5
	93-0012-001	0.50	83.6
	93-0012-001	0.50	88.0
	93-0012-001	0.5 [1]	87.8
	93-0012-001	0.5 [1]	85.5
	93-0012-001	0.5 [1]	91.1
Concord	92-0089-005	0.01 [1]	109.0
	92-0089-005	0.01 [1]	95.8
	92-0089-005	0.1 [1]	107.0
	92-0089-005	0.1 [1]	77.0
	92-0089-005	0.5 [1]	104.0
	92-0089-005	0.5 [1]	108.0

Data continued on the next page.

[1] This data is taken from Appendix 8 in MRID 42793300.

All other data on this page is taken from TR 34-95-104, under protocol 34P-95-65A, MRID not available yet.

Table 6. Grape Oxyfluorfen Fortification Summary[1]
(continued)

Variety	RAR/ Sample No.	Fortification Level, ppm		% Recovery
Concord	92-0089-005	0.01		88.8
	92-0089-005	0.10		114.0
Cabernet	92-0100-005	0.01		81.0
		0.01		86.7
		0.01		69.6
		0.02		88.0
		0.02		96.0
		0.10		106.0
Niagra	92-0072-005	0.01		86.9
		0.01		88.0
		0.01		93.1
		0.02		90.5
		0.02		108.0
		0.05		106.0
		Average	38	89.3
		Standard Deviation		11.6

[1] All other data on this page is taken from TR 34-95-173, under protocol 34P-95-65A, MRID not available yet.

Table 7. Kiwi Oxyfluorfen Fortification Summary[1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0146-001	0.01[2]	64
94-0146-001	0.01	76.4
94-0146-001	0.01	86.5
94-0146-001	0.01	87.4
94-0146-001	0.01	93.8
94-0146-001	0.02	97.5
94-0146-001	0.02	101.0
94-0146-001	0.05	103.0
94-0146-001	0.05	104.0
94-0146-001	0.10	96.9
94-0146-001	0.50	84.2
94-0146-001	0.50	91.2
	Average n=11	92.9
	Standard Deviation	8.7

[1] All data reported in this table is taken from MRID 43794005.

[2] Value outside 2 x SD, therefore not included in statistics.

Table 8. Olive Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0172-001	0.01 [1]	91.5
94-0172-001	0.01	81.5
94-0172-001	0.01	75.1
94-0172-001	0.01	74.6
94-0172-001	0.01	82.4
94-0172-001	0.02	107.0
94-0172-001	0.02	106.0
94-0172-001	0.05	98.6
94-0172-001	0.10	74.5
94-0172-001	0.1 [1]	105.0
94-0172-001	0.50	91.6
94-0172-001	0.5 [1]	93.8
	Average n=12	90.1
	Standard Deviation	12.4

[1] This data is taken from MRID 43794006.

All other data collected under 34P-95-33A.

Table 9. Peach Oxyfluorfen Fortification Summary [1]

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0117-001	0.01	70.0
94-0117-001	0.01	85.0
94-0117-001	0.01	86.1
94-0117-001	0.01	90.9
94-0117-001	0.01	92.9
94-0117-001	0.01 [2]	102.0
95-0196-003	0.01	88.3
94-0117-001	0.02	73.0
94-0117-001	0.02	87.5
94-0117-001	0.02	91.5
94-0117-001	0.05	78.2
94-0117-001	0.05	87.4
94-0117-001	0.05	93.4
94-0117-001	0.05 [2]	103.0
95-0196-003	0.05	89.8
94-0117-001	0.10	72.1
94-0117-001	0.10	86.5
94-0117-001	0.10	93.7
94-0117-001	0.10 [2]	88.7
94-0117-001	0.50	81.7
94-0117-001	0.50	88.5
94-0117-001	0.50	91.9
94-0117-001	0.50 [2]	75.2
95-0196-003	0.10	83.2
	Average n=24	86.7
	Standard Deviation	8.5

[1] All data reported is taken from TR 34-95-114 (MRID not yet available), under protocols 34P-95-35A, 34P-95-51A, except those noted as below.

[2] This data was not collected under the protocols noted above or reported in TR 34-95-114. It was collected as an evaluation of the method.

Table 10. Peanut Nutmeat Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
93-0142-005	0.01[1]	115.0
93-0142-005	0.01[1]	131.0[2]
93-0142-005	0.01[3]	79.0
93-0142-005	0.01[3]	86.0
93-0142-005	0.01[3]	96.3
93-0142-005	0.01[3]	84.8
94-0176-010	0.01[1]	105.0
93-0142-005	0.02[3]	102.0
93-0142-005	0.02[3]	95.5
93-0142-005	0.02[3]	79.5
93-0142-005	0.02[3]	84.5
93-0142-005	0.05[3]	113.0
93-0142-005	0.1[3]	76.4
93-0157-004	0.1[1]	70.1
94-0176-010	0.1[1]	80.6
93-0142-005	0.5[3]	90.6
93-0157-004	0.5[1]	99.4
94-0176-010	0.5[1]	86.6
	Average n=17	90.8
	Standard Deviation	12.9

[1] Data reported taken from Appendix 9 in MRID 42793300.

[2] Value outside 2 x SD, therefore not included in statistics.

[3] Data reported taken from Appendix 4 in MRID 42793300.

Table 11. Pomegranate Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
94-0143-001	0.01 [1]	76.4
94-0143-001	0.01	87.2
94-0143-001	0.01	80.1
94-0143-001	0.01	81.9
94-0143-001	0.01	70.6
94-0143-001	0.02	103.0
94-0143-001	0.02	109.0
94-0143-001	0.05	107.0
94-0143-001	0.10	92.7
94-0143-001	0.1 [1]	100.0
94-0143-001	0.50	104.0
94-0143-001	0.5 [1]	84.0
	Average n=12	91.3
	Standard Deviation	13.0

[1] This data is taken from MRID 43794004.

All other data collected under 34P-95-31A.

Table 12. Soybean Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
92-0107-014	0.01	108.0
92-0107-014	0.01	119.0
94-0136-001	0.01	119.0
94-0136-001	0.01	108.0
92-0107-014	0.10	98.5
92-0107-014	0.10	100.0
94-0136-001	0.10	86.3
94-0136-001	0.10	106.0
92-0107-014	0.50	121.0
92-0107-014	0.50	108.0
94-0136-001	0.50	91.6
94-0136-001	0.50	93.2
	Average n=12	104.9
	Standard Deviation	11.3

[1] This data is taken from Appendix 8 in MRID 42793300.

Table 13. Peanut Hay Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
93-0142-003	0.02[1]	87.4
93-0142-003	0.02[2]	97.5
93-0142-003	0.02[2]	96.5
93-0157-002	0.02[1]	111.0
93-0157-002	0.02[2]	80.5
93-0157-002	0.05[2]	97.0
93-0157-002	0.05[2]	92.8
93-0142-003	0.05[2]	99.0
93-0142-003	0.1[1]	82.0
93-0157-002	0.5[1]	78.0
	Average n=10	92.2
	Standard Deviation	10.2

[1] Data reported taken from Appendix 9 in MRID 42793300.

[2] Data reported taken from Appendix 4 in MRID 42793300.

Table 14. Peanut Shell Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
93-0142-005	0.02 [1]	102.0
93-0142-005	0.02 [3]	98.5
93-0157-004	0.02 [1]	65.5 [2]
93-0157-004	0.02 [3]	89.5
93-0157-004	0.05 [3]	95.2
93-0142-005	0.02 [3]	94.5
93-0142-005	0.05 [3]	98.0
93-0142-005	0.05 [3]	94.0
93-0142-005	0.1 [1]	89.6
93-0157-004	0.1 [3]	86.4
93-0157-004	0.5 [1]	105.0
	Average n=10	95.3
	Standard Deviation	5.8

[1] Data reported taken from Appendix 9 in MRID 42793300.

[2] Value outside 2 x SD, therefore not included in statistics.

[3] Data reported taken from Appendix 4 in MRID 42793300.

Table 15. Peanut Vine Oxyfluorfen Fortification Summary

RAR/ Sample No.	Fortification Level, ppm	% Recovery
93-0142-001	0.02 [2]	87.5
93-0142-001	0.02 [2]	82.5
93-0142-001	0.02 [1]	82.1
93-0142-001	0.02 [1]	87.6
93-0142-001	0.05 [2]	87.4
93-0142-001	0.05 [2]	95.2
93-0142-001	0.05 [2]	98.0
93-0142-001	0.1 [1]	73.2
93-0142-001	0.1 [1]	64.6
93-0142-001	0.5 [1]	68.2
93-0142-001	0.5 [1]	74.0
	Average n=11	81.8
	Standard Deviation	10.8

[1] Data reported taken from Appendix 9 in MRID 42793300.

[2] Data reported taken from Appendix 4 in MRID 42793300.

Table 16. Grape Oxyfluorfen Fortification Summary [1]
Rtx-50 Confirmatory Column

Variety	RAR/ Sample No.	Fortification Level, ppm		% Recovery
Thompson	93-0012-001	0.01		100.0
	93-0012-001	0.01		77.9
	93-0012-001	0.10		75.3
	93-0012-001	0.10		97.3
	93-0012-001	0.50		101.0
	93-0012-001	0.50		95.2
Concord	92-0089-005	0.01		92.5
	92-0089-005	0.01		84.2
	92-0089-005	0.10		97.8
	92-0089-005	0.10		83.9
	92-0089-005	0.50		110.0
	92-0089-005	0.50		96.5
		Average	n=12	92.6
		Standard Deviation		10.3

[1] This data is taken from Appendix 8 in MRID 42793300.

Table 17. Soybean Oxyfluorfen Fortification Summary
Rtx-50 Confirmatory Column

RAR/ Sample No.	Fortification Level, ppm	% Recovery
92-0107-014	0.01	54 *
92-0107-014	0.01	50 *
94-0136-001	0.01	106.0
94-0136-001	0.01	113.0
92-0107-014	0.10	98.7
92-0107-014	0.10	90.8
94-0136-001	0.10	100.0
94-0136-001	0.10	124.0
92-0107-014	0.50	110.0
92-0107-014	0.50	97.7
94-0136-001	0.50	104.0
94-0136-001	0.50	95.6
	Average n=10	104.0
	Standard Deviation	9.7

[1] This data is taken from Appendix 8 in MRID 42793300.

* Value outside 2 X SD, therefore not included in statistics.

Table 18. Oxyfluorfen GC/MS Confirmatory Data Summary [1]

Sample Type	RAR/ Sample No.	Fortification Level, ppm	% Recovery GC/MS	% Recovery ECD
grape	92-0100-005	0.025	93.0	99.0
grape	92-0100-005	0.05	86.0	82.9
grape	92-0100-005	0.125	108.0	91.2
		Average	95.7	91.0
peach	94-0117-001	0.025	85.0	91.4
peach	94-0117-001	0.05	82.0	79.4
peach	94-0117-001	0.125	102.0	89.6
		Average	89.7	86.8
peanut nutmeat	93-0157-004	0.025	109.0	109.0
peanut nutmeat	93-0157-004	0.05	81.0	163.0
peanut nutmeat	93-0157-004	0.125	87.0	116.0
		Average	92.3	129.3
peanut shell	93-0157-004	0.025	104.0	104.0
peanut shell	93-0157-004	0.05	117.0	107.0
peanut shell	93-0157-004	0.125	103.0	106.0
		Average	108.0	105.7

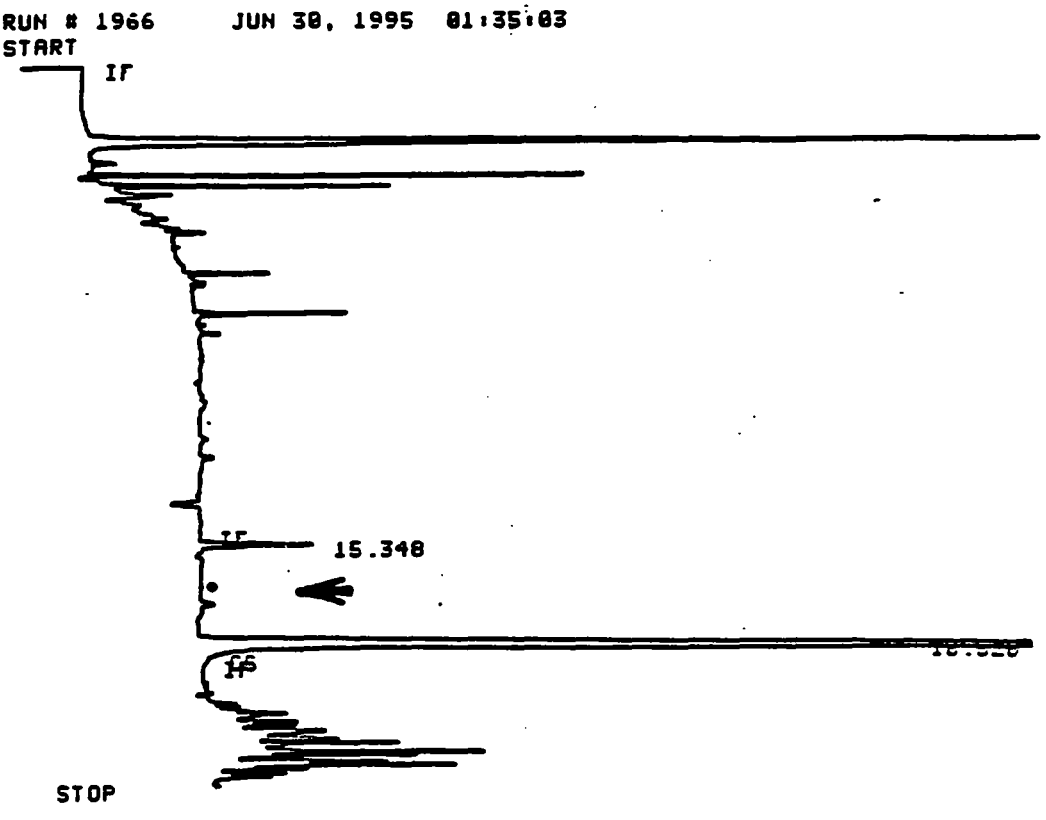
[1] This data was collected and reviewed under protocol 34-95-92.
LOQ=0.025 ppm
LOD=0.008 ppm

Table 19. Oxyfluorfen Data Summary by Crop

Crop	Group[1]	Detection	Average Recovery	LOQ	LOD
apple	I	GC/ECD	85.4	0.01 ppm	0.003 ppm
artichoke	I	GC/ECD	94	0.01 ppm	0.003 ppm
avocado	I	GC/ECD	93.9	0.01 ppm	0.003 ppm
cherry	I	GC/ECD	86.5	0.01 ppm	0.003 ppm
fig	I	GC/ECD	91.6	0.01 ppm	0.003 ppm
grape	I	GC/ECD	89.3	0.01 ppm	0.003 ppm
kiwi	I	GC/ECD	92.9	0.01 ppm	0.003 ppm
olive	I	GC/ECD	90.1	0.01 ppm	0.003 ppm
peach	I	GC/ECD	86.7	0.01 ppm	0.003 ppm
peanut nutmeat	I	GC/ECD	90.9	0.01 ppm	0.003 ppm
pomegranate	I	GC/ECD	91.3	0.01 ppm	0.003 ppm
soybean	I	GC/ECD	104.9	0.01 ppm	0.003 ppm
peanut hay	II	GC/ECD	92.2	0.02 ppm	0.007 ppm
peanut shell	II	GC/ECD	95.3	0.02 ppm	0.007 ppm
peanut vine	II	GC/ECD	81.8	0.02 ppm	0.007 ppm

[1] Refers to section 7 crop grouping for analyis.

Figure 1. Apple Control
95-1408-003



RUN# 1966 JUN 30, 1995 01:35:03

APPLE

SAMPLE NAME: 95-1408 SAMPLE# 23 Control

METHOD NAME: M:GOALEXT.MET

G FINAL WT 5 VOL INJ ul ML FINAL VOL 5 DIL FACTOR 1 MG INJ 1

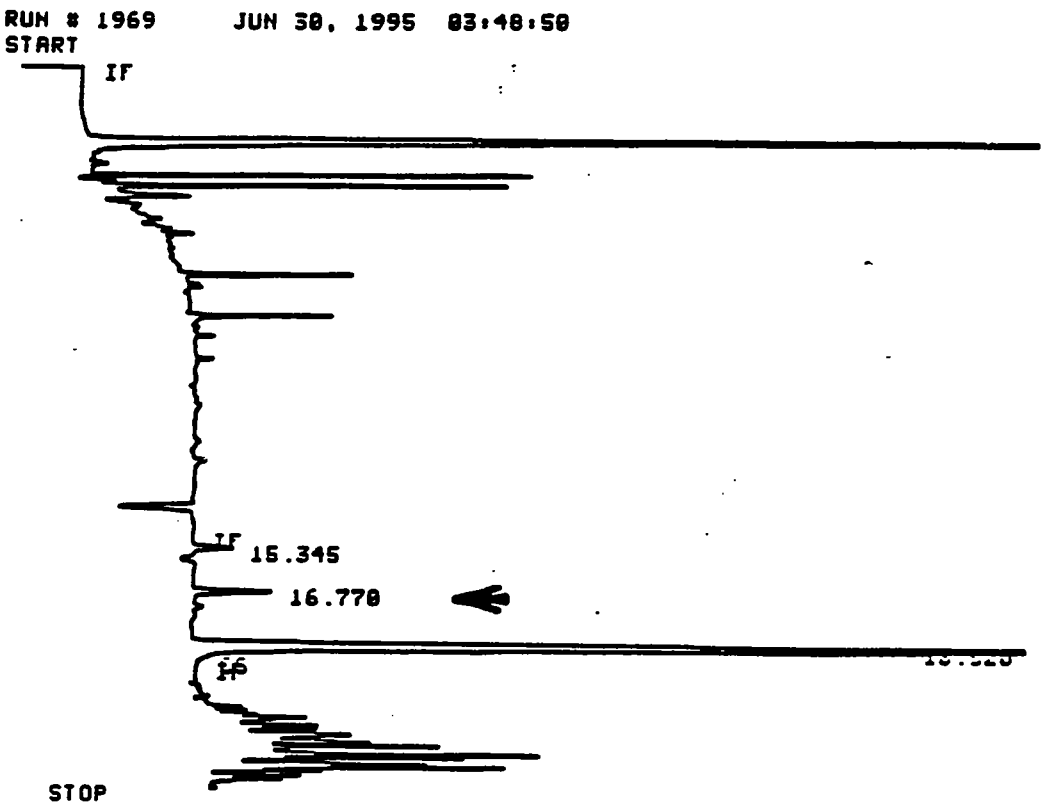
HEIGHTX

RT	HEIGHT	TYPE	WIDTH	HEIGHTX
15.348	3803	BB	.117	8.14573
18.526	42884	PB	.196	91.85424

TOTAL HEIGHT= 46687

MUL FACTOR=1.0000E+00

Figure 2. Apple Fortification 0.01 PPM
95-1408-003



RUN# 1969 JUN 30, 1995 03:48:50

APPLE fortification

SAMPLE NAME: 0.01ppm SAMPLE# 26 95-1408

METHOD NAME: M:GOALEXT.MET

G FINAL WT 5 VOL INJ 1ml ML FINAL VOL 5 DIL FACTOR 1 MG INJ 1

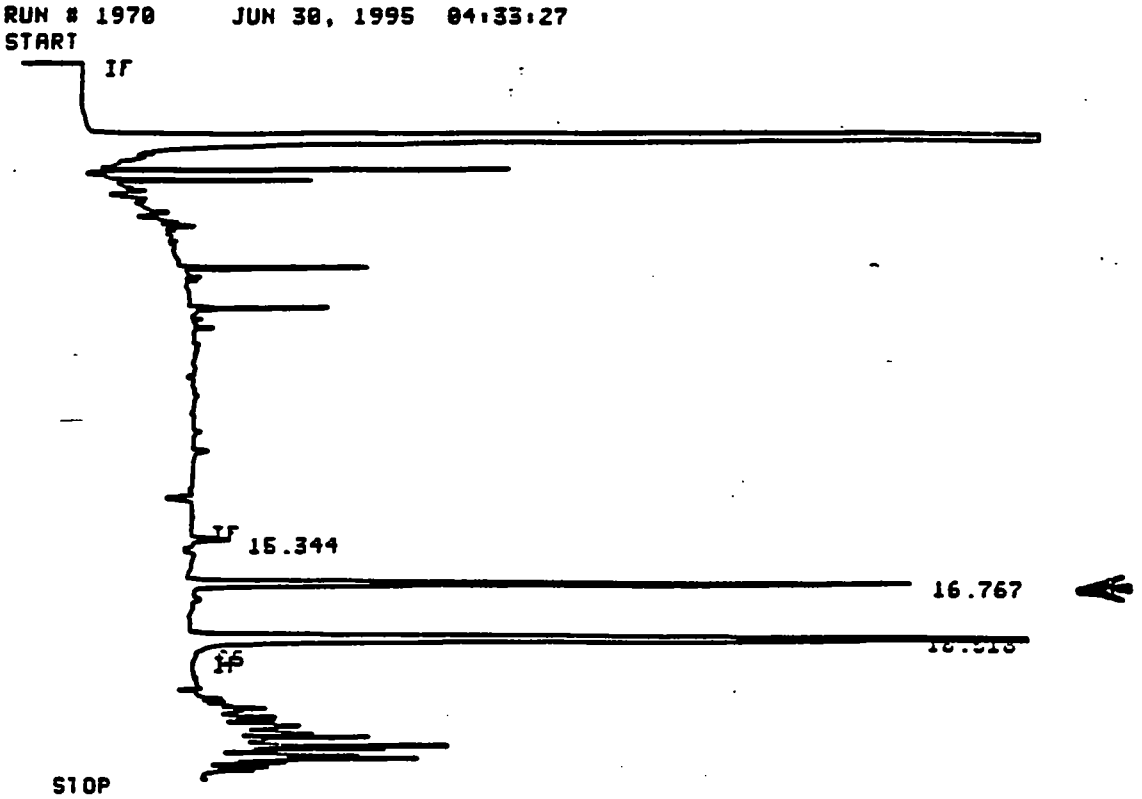
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
15.345	1441	BB	.158	3.66331
16.770	2657	BB	.123	6.75465
18.520	35238	PB	.187	89.58205

TOTAL HEIGHT= 39336

MUL FACTOR=1.0000E+00

Figure 3. Apple Fortification 0.10 PPM
95-1408-003



RUN# 1970 JUN 30, 1995 04:33:27

APPLE fortification

SAMPLE NAME: 0.1ppm SAMPLE# 27 95-1408

METHOD NAME: M:GOALEXT.MET

G FINAL WT 5 VOL INJ 1ml ML FINAL VOL 5 DIL FACTOR 1 MG INJ 1

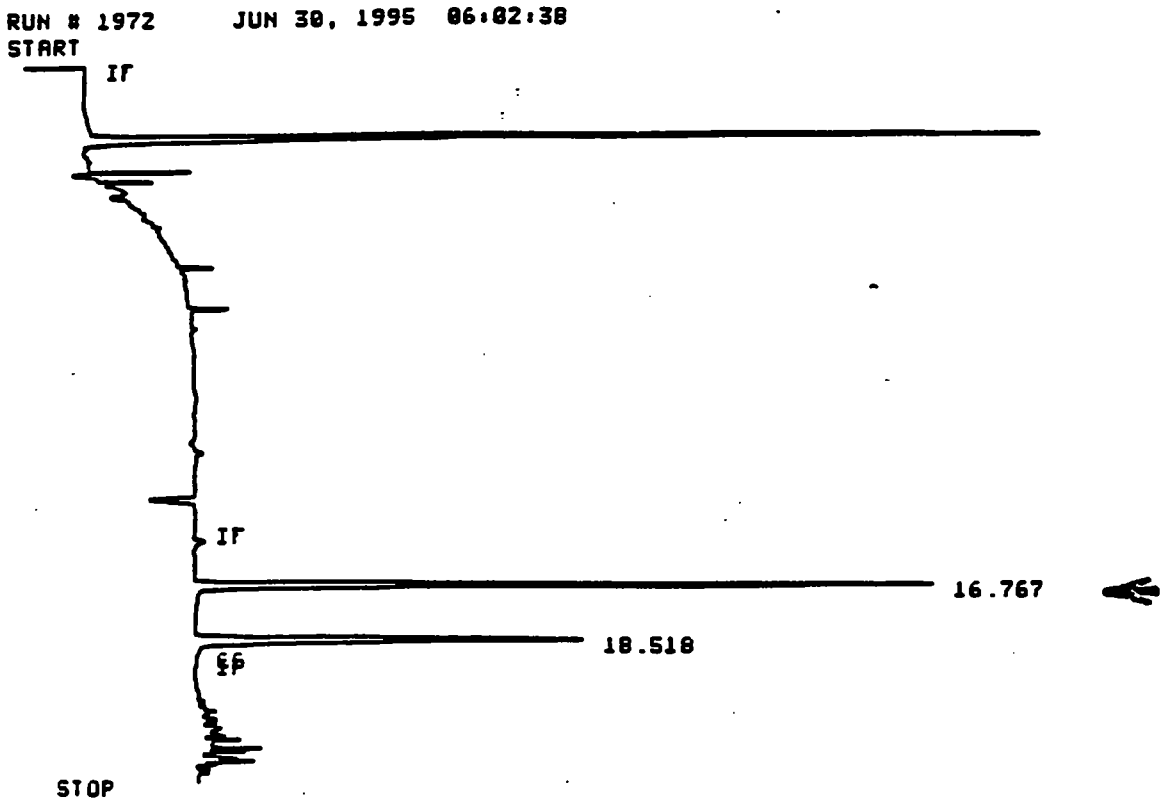
HEIGHTx

RT	HEIGHT	TYPE	WIDTH	HEIGHTx
15.344	1374	BP	.147	2.28315
16.767	24114	PB	.124	40.06979
18.518	34692	PB	.185	57.64707

TOTAL HEIGHT= 60180

MUL FACTOR=1.0000E+00

Figure 4. Apple Fortification 0.50 PPM
95-1408-003



RUN# 1972 JUN 30, 1995 06:02:38

APPLE
SAMPLE NAME: fortification 0.5 ppm SAMPLE# 29 95-1408
METHOD NAME: M:GOALEXT.MET
G FINAL WT 5 VOL INJ lml ML FINAL VOL 5 DIL FACTOR 5 MG INJ 0.2

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
16.767	24710	PB	.124	65.70934
18.518	12895	PB	.164	34.29066

TOTAL HEIGHT= 37605
MUL FACTOR=1.0000E+00

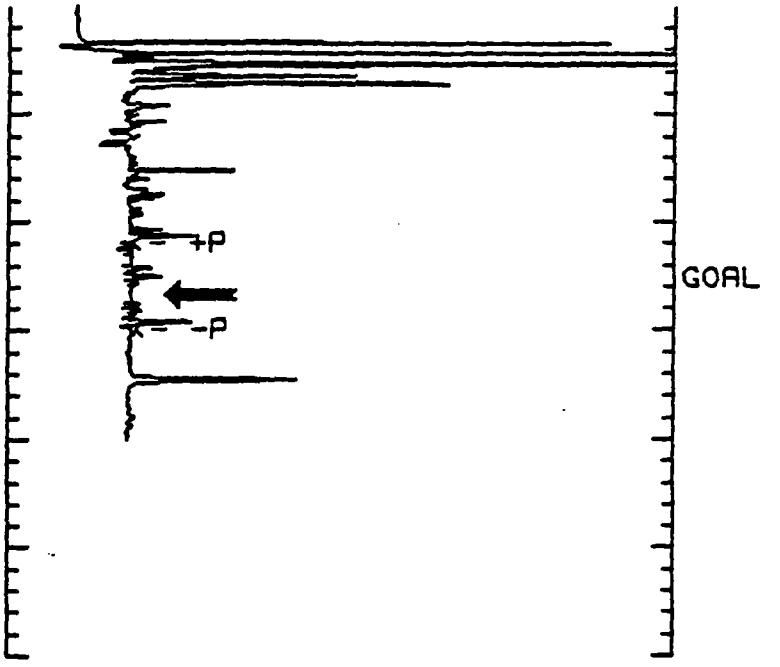
Figure 5. Artichoke Control
94-0060-001

Data file: L0688 RAR number: 94-0060
Method file: GOAL9a Sample No: 00121
Type: SAMPLE Component: ARTICHOKE

Sample Name: NA Cal. Curve: 07/06/95
Date: 6 Jul 1995 19:12 Method: GOAL9a Analyst : SS
Interface: 711 Cycle#: 5 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min He Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.46	GOAL	0.000E+00	.000E+00	5.00	5.00

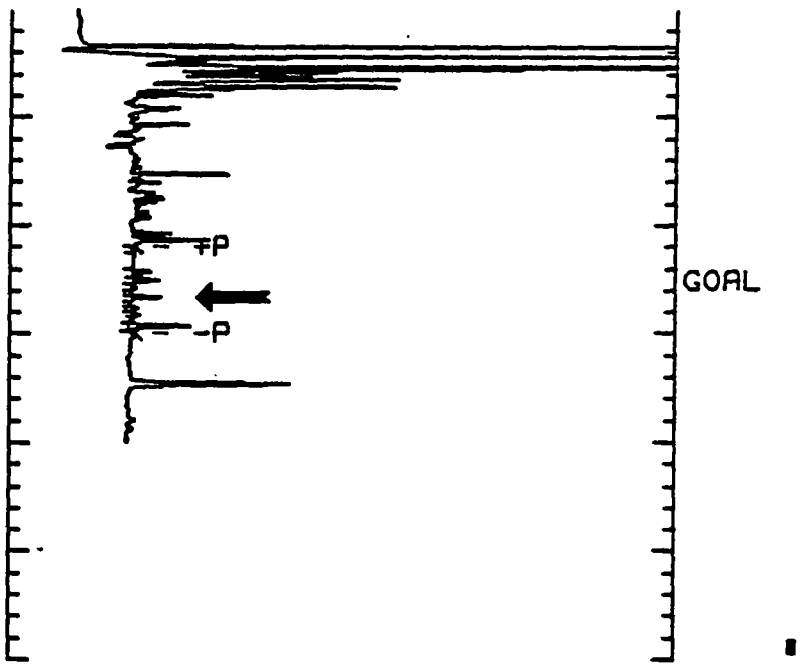
Figure 6. Artichoke Fortification 0.01 PPM
94-0060-001

Data file: L0688 RAR number: 94-0060
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: ARTICHOKE

Sample Name: NA Cal. Curve: 07/06/95
Date: 6 Jul 1995 20:26 Method: GOALVm Analyst : ES SZ
Interface: 711 Cycle#: 8 Channel#: A

Instrument: VARIAN 3600,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 20 minutes
Plot range: 50 millivolts (-.24 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	2.770E+08	.124E+03	5.00	5.00	0.050

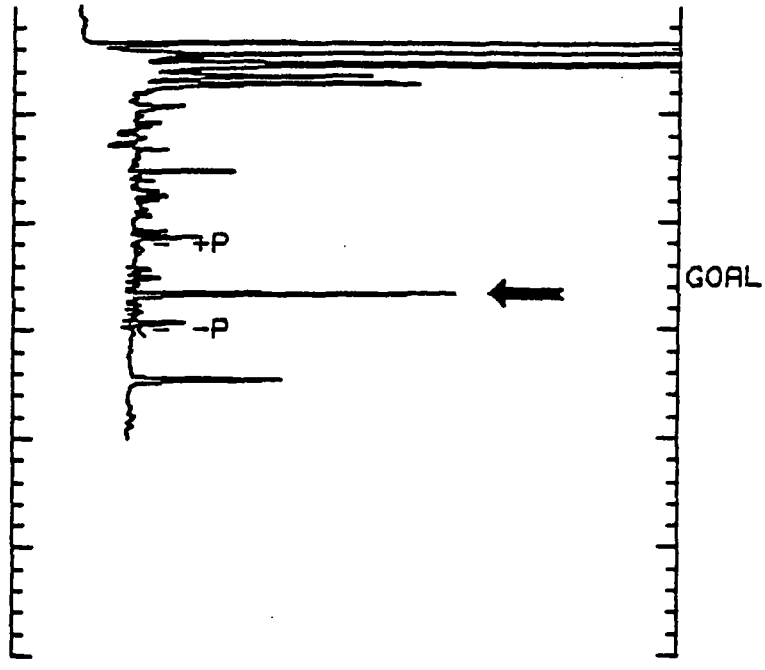
Figure 7. Artichoke Fortification 0.10 PPM
94-0060-001

Data file: L0689 RAR number: 94-0060
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: ARTICHOKE

Sample Name: NA Cal. Curve: 07/06/95
Date: 6 Jul 1995 20:53 Method: GOALVm Analyst : SS SZ
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.27 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	2.960E+01	.241E+04	5.00	5.00	0.500

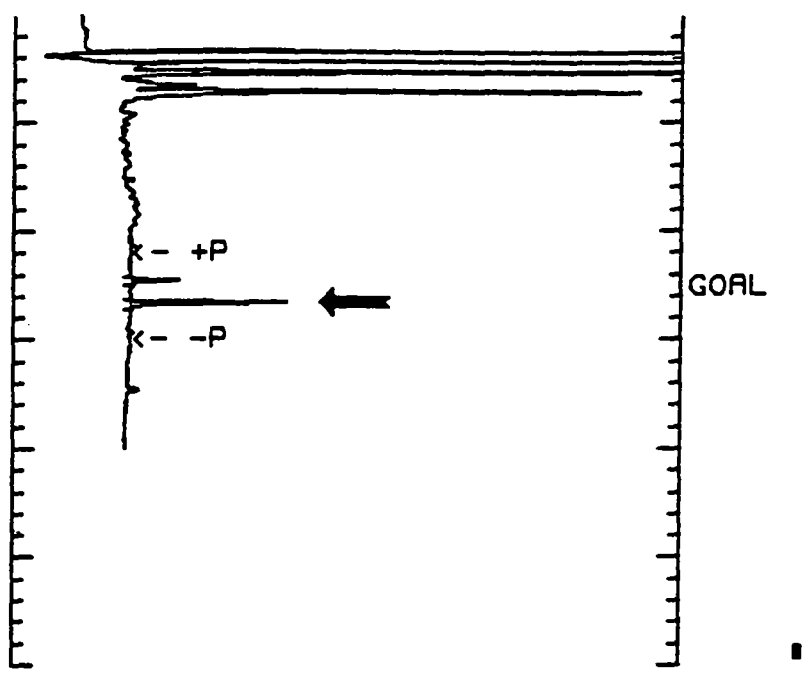
Figure 8. Artichoke Fortification 0.50 PPM 94-0060-001

Data file: L06810 RAR number: 94-0060
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: ARTICHOKE

Sample Name: NA Cal. Curve: 07/06/95
Date: 6 Jul 1995 21:19 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 18 Channel#: A

Instrument: VARIAN 3600,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-15
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: SCD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.32	GOAL	1.460E+01	.117E+04	50.0	5.00	2.50

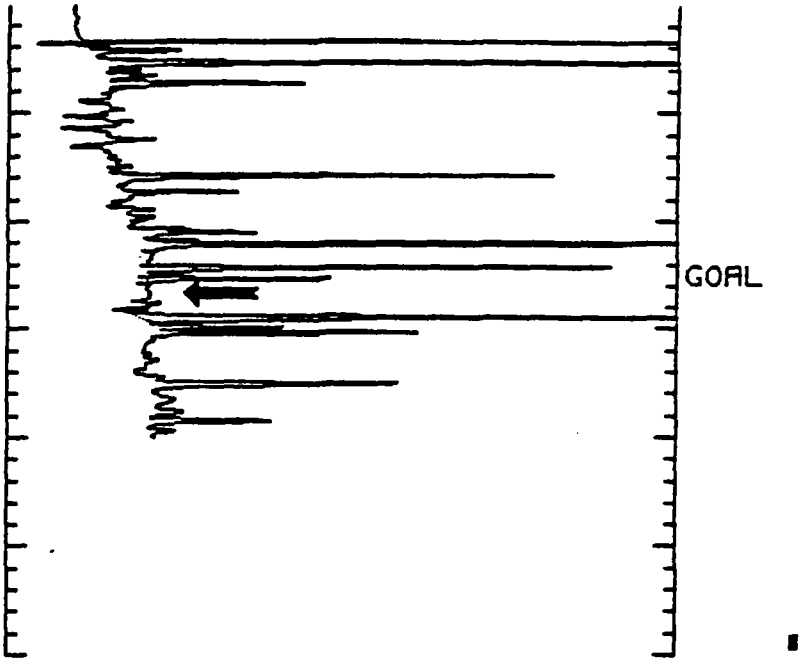
Figure 9. Avocado Control
94-0141-001

Data file: L1985 RAR number: 94-0141
Method file: GOALVn Sample No: 001
Type: SAMPLE Component: AVOCADO

Sample Name: NA Cal. Curve: 07/20/95
Date: 20 Jul 1995 23:35 Method: GOALVn Analyst : SF
Interface: 711 Cycle#: 5 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.8ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.24 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.31	GOAL	0.000E+00	.000E+00	5.00	5.00

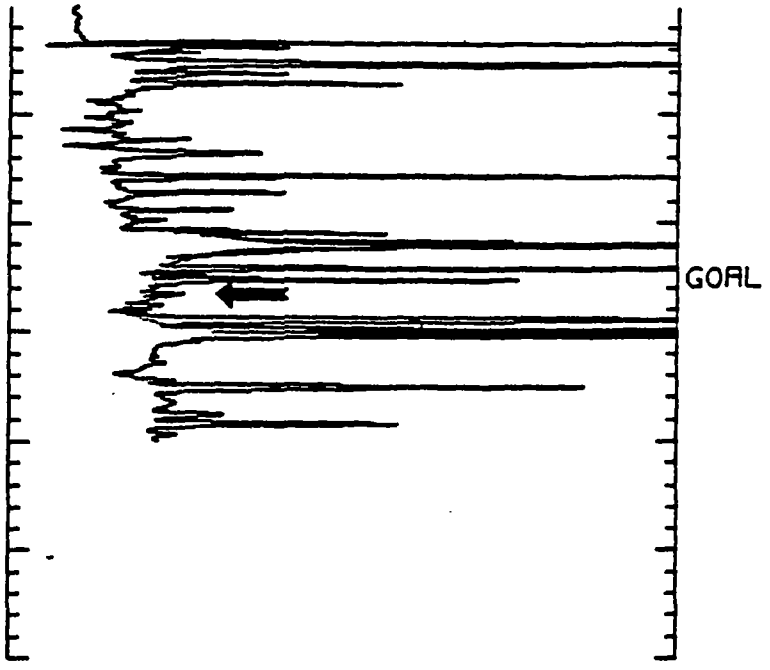
Figure 10. Avocado Fortification 0.01 PPM 94-0141-001

Data file: L1989 RAR number: 94-0141
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: AVOCADO

Sample Name: NA Cal. Curve: 07/20/95
Date: 21 Jul 1995 01:16 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.5 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.31	GOAL	3.119E+00	.246E+03	5.00	5.00	0.050

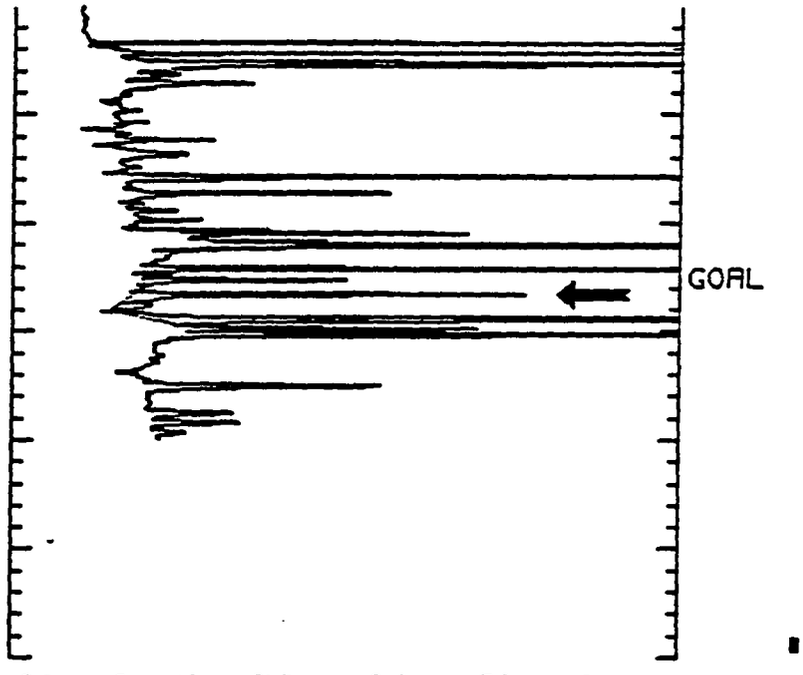
Figure 11. Avocado Fortification 0.10 PPM 94-0141-001

Data file: L11RA9 RAR number: 94-0141
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: AVOCADO

Sample Name: NA Cal. Curve: 07/13/95
Date: 13 Jul 1995 16:14 Method: GOALVm Analyst : SS SZ
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.31	GOAL	4.240E+01	.297E+04	5.00	5.00	0.500

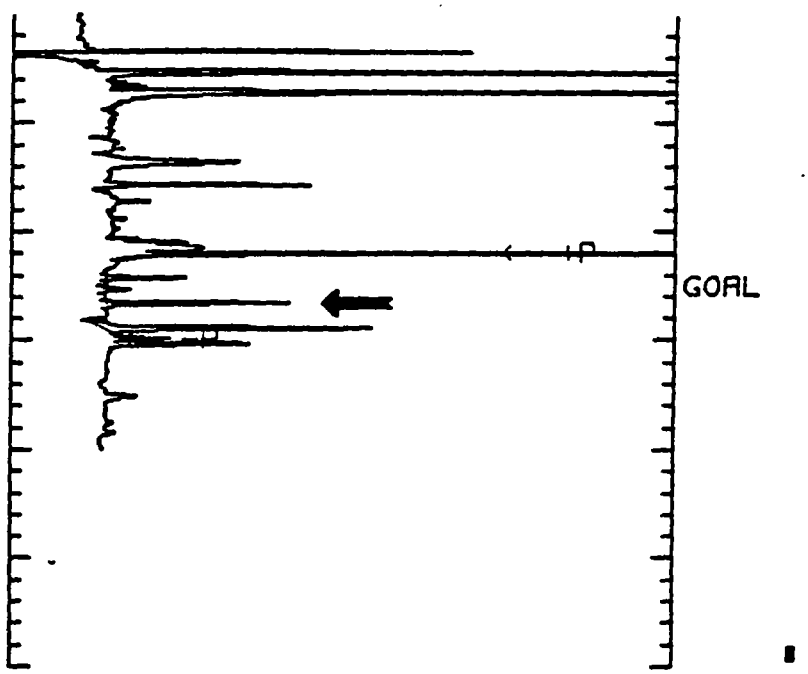
Figure 12. Avocado Fortification 0.50 PPM 94-0141-001

Data file: L19816 RAR number: 94-0141
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: AVOCADO

Sample Name: NA Cal. Curve: 07/20/95
Date: 21 Jul 1995 04:13 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 16 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.19 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.31	GOAL	1.670E+01	.137E+04	50.0	5.00	2.50

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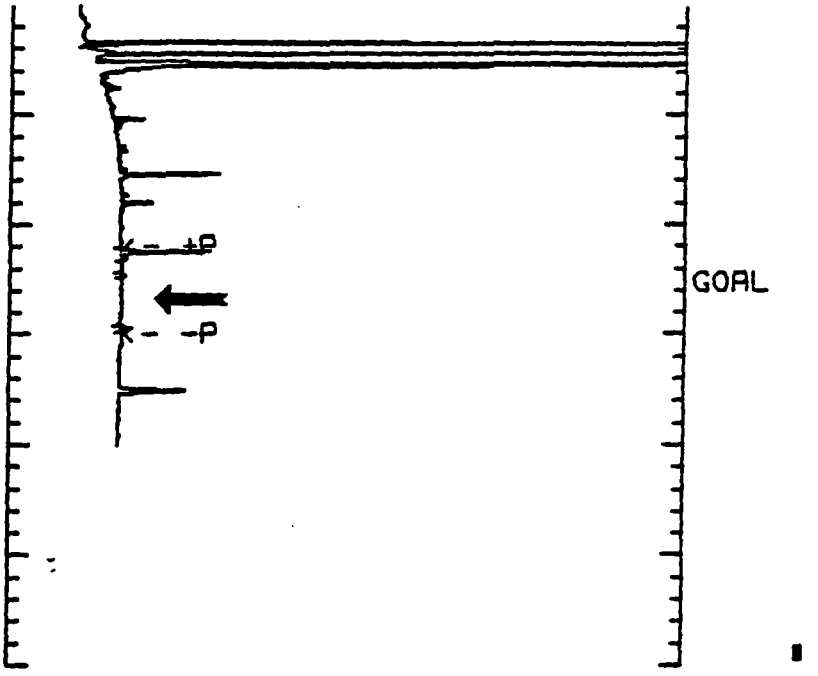
Figure 13. Cherry Control
92-0041-002

Data file: LG586 RAR number: 92-0041
Method file: GOALVm Sample No: 0028
Type: SAMPLE Component: CHERRY

Sample Name: NA Cal. Curve: 07/05/95
Date: 5 Jul 1995 19:21 Method: GOALVm Analyst : SS
Interface: 711 Cycle#: 6 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.29 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.46	GOAL	0.000E+00	.000E+00	5.00	5.00

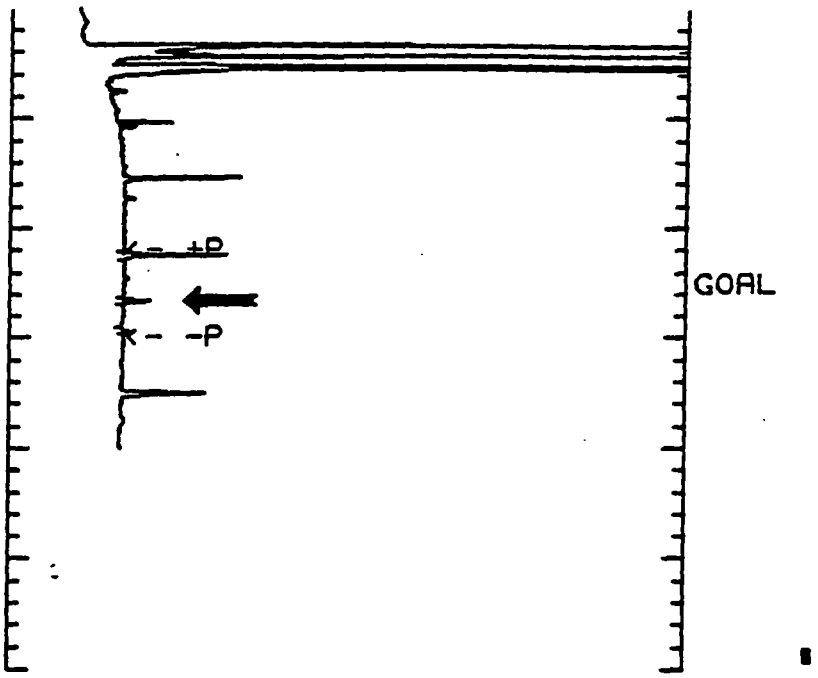
Figure 14. Cherry Fortification 0.01 PPM
92-0041-002

Data file: L8588 RAR number: 92-0041
Method file: GOALVm Sample No: 002
Type: FORTIFICATION Component: CHERRY

Sample Name: NA Cal. Curve: 07/08/95
Date: 8 Jul 1995 19:40 Method: GOALVm Analyst : SS SZ
Interface: 711 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.34	GOAL	2.460E+00	.192E+03	5.00	5.00	0.050

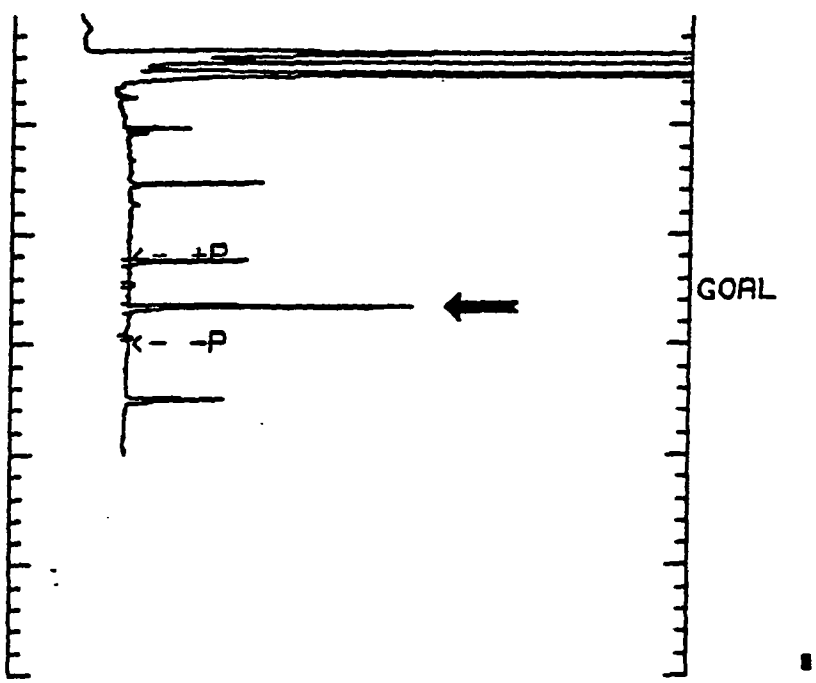
Figure 15. Cherry Fortification 0.10 PPM
92-0041-002

Data file: L0589 RAR number: 92-0041
Method file: GOALVa Sample No: 002
Type: FORTIFICATION Component: CHERRY

Sample Name: NA Cal. Curve: 07/05/95
Date: 5 Jul 1995 19:56 Method: GOALVa Analyst : SE
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	3.610E+01	.388E+04	5.00	5.00	0.500

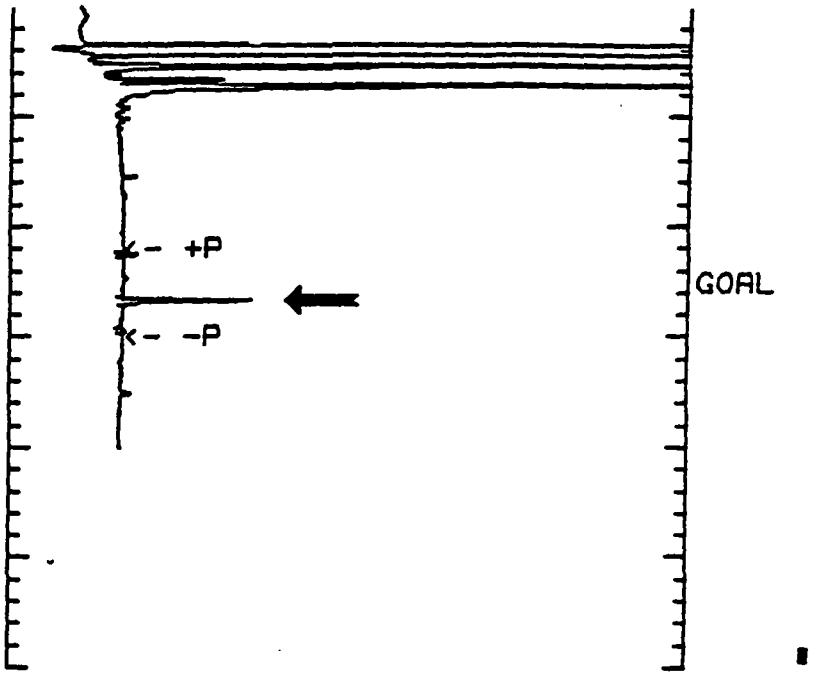
Figure 16. Cherry Fortification 0.50 PPM
92-0041-002

Data file: L05810 RAR number: 92-0041
Method file: GOALVm Sample No: 002
Type: FORTIFICATION Components: CHERRY

Sample Name: NA Cal. Curve: 07/05/95
Date: 5 Jul 1995 20:24 Method: GOALVm Analyst : SS SZ
Interface: 711 Cycle#: 10 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.32 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.24	GOAL	1.200E+01	.945E+03	50.0	5.00	2.50

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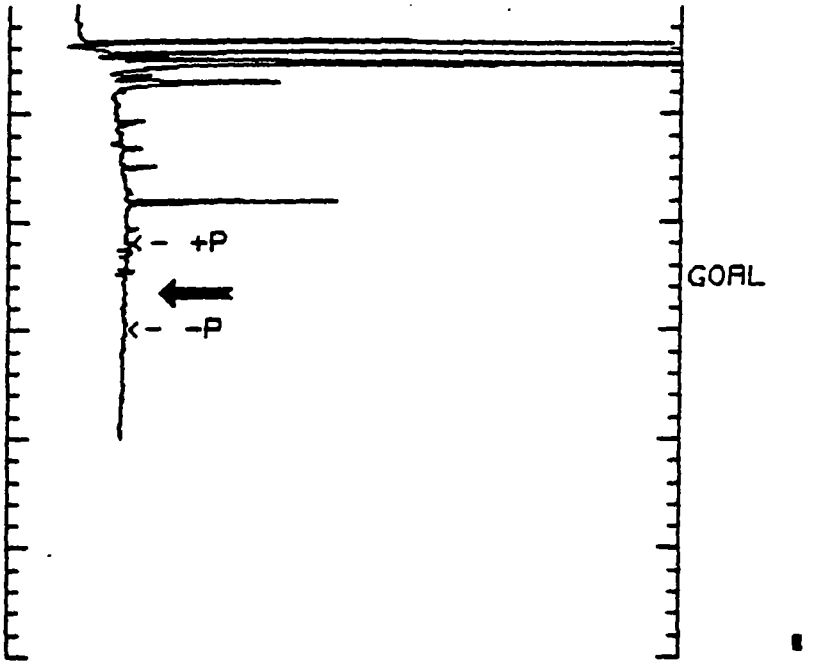
Figure 17. Fig Control
94-0142-001

Data file: L11SA13 RAR number: 94-0142
Method file: GOALVm Sample No: 0012
Type: SAMPLE Component: FIG

Sample Name: NA Cal. Curve: 07/13/95
Date: 13 Jul 1995 17:54 Method: GOALVm Analyst : SB SZ
Interface: 711 Cycle#: 13 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.27 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.31	GOAL	0.0000E+00	.000E+00	5.00	5.00

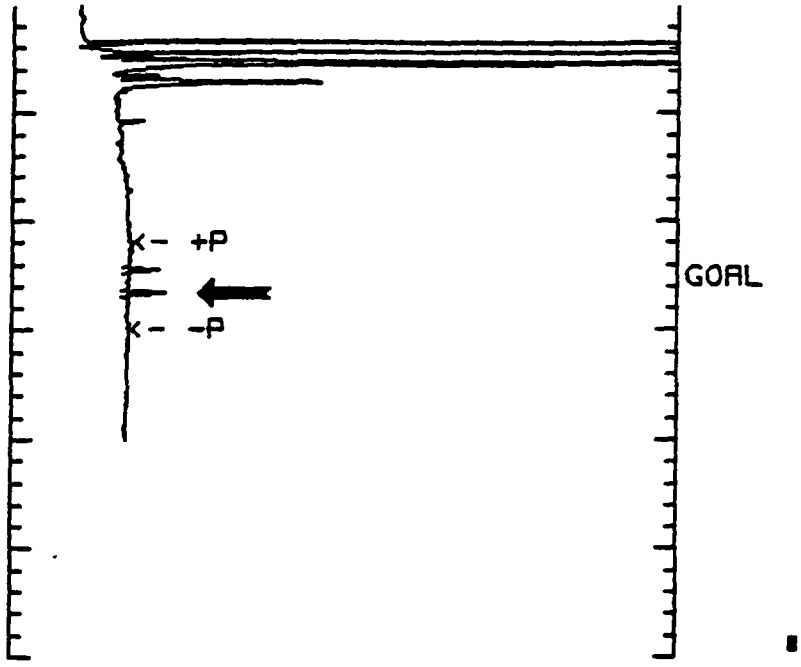
Figure 18. Fig Fortification 0.01 PPM
94-0142-001

Data file: L115A16 RAR number: 94-0142
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/12/95
Date: 12 Jul 1995 19:10 Method: GOALVm Analyst : SS-SE
Interface: 711 Cycle#: 16 Channel#: A

Instrument: VARIAN 3500.
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.32	GOAL	3.2368+00	.2778+03	5.00	5.00	0.050

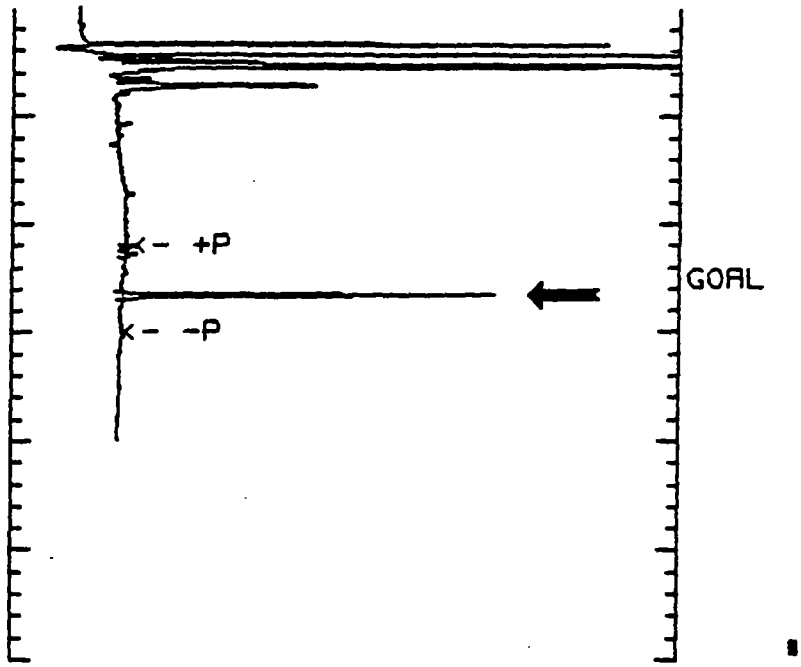
Figure 19. Fig Fortification 0.10 PPM
94-0142-001

Data file: L11SA17 RAR number: 94-0142
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/13/95
Date: 12 Jul 1995 19:35 Method: GOALVm Analyst : SS/JZ
Interface: 711 Cycle#: 17 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.32	GOAL	3.260E+01	.276E+04	5.00	5.00	0.500

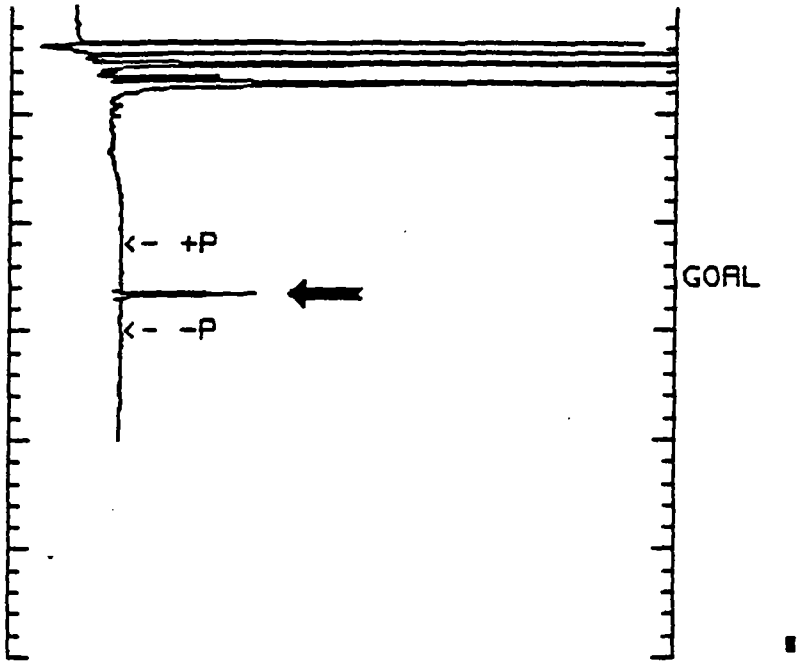
Figure 20. Fig Fortification 0.50 PPM 94-0142-001

Data file: L11SA18 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/13/95
Date: 13 Jul 1995 20:00 Method: GOALVa Analyst : SE SZ
Interface: 711 Cycle#: 18 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.37 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.32	GOAL	1.250E+01	.102E+04	50.0	5.00	2.50

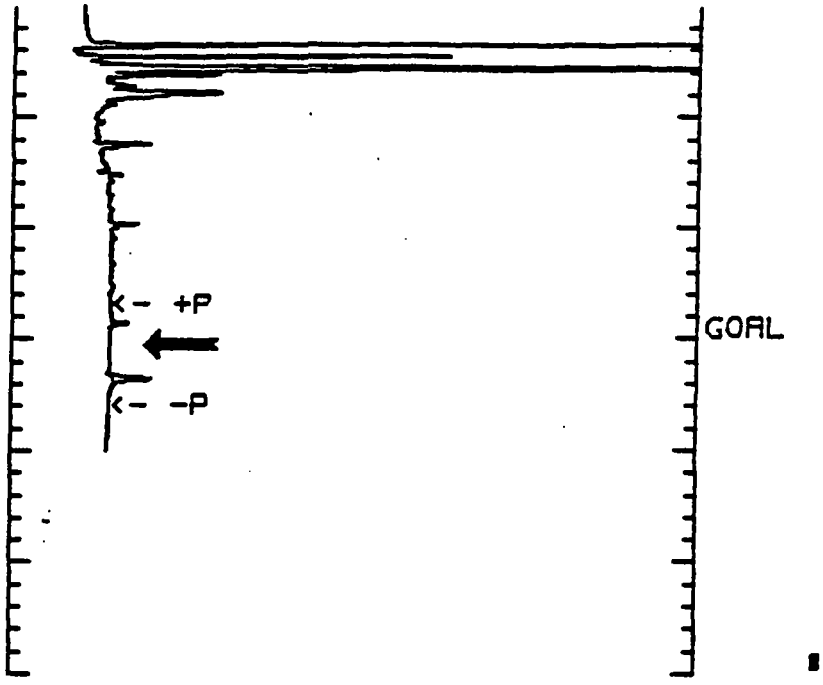
Figure 21. Grape Control
93-0012-001

Data file: J05J6 RAR number: 93-0012
Method file: GOALVm Sample No: 001
Type: SAMPLE Component: THOMPSON

Sample Name: NA Cal. Curve: 01/05/95
Date: 5 Jan 1995 21:29 Method: GOALVm Analyst : SZ SZ
Interface: 711 Cycle#: 6 Channel#: A

Instrument: VARIAN 3300,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 360 millivolts (31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	* Samp Wt.
15.26	GOAL	0.000E+00	.000E+00	5.00	5.00

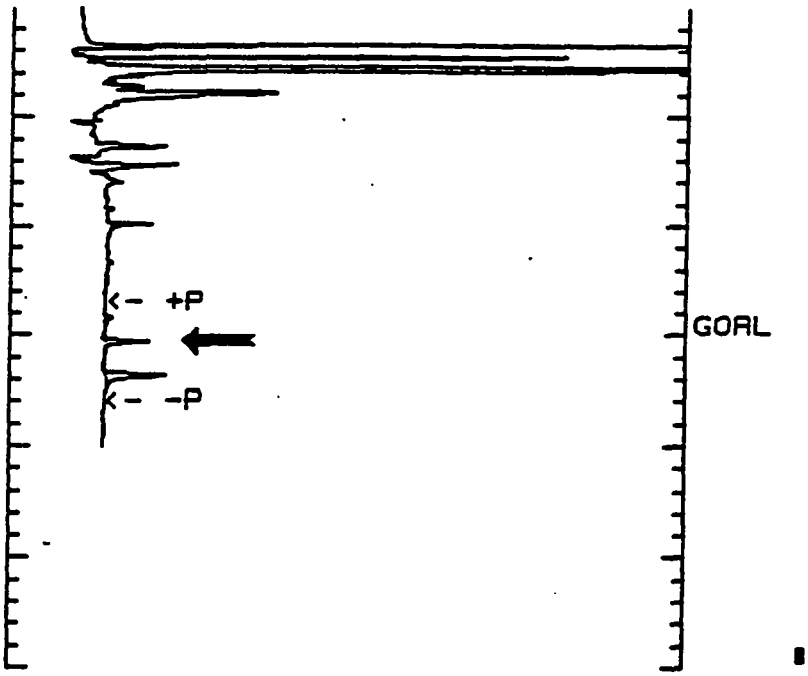
Figure 22. Grape Fortification 0.01 PPM
93-0012-001

Data file: J1337 RAR number: 93-0012
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: THOMPSON

Sample Name: NA Cal. Curve: 01/14/95
Date: 14 Jan 1995 17:49 Method: GOALVm Analyst : SZ
Interface: 715 Cycle#: 7 Channel#: A

Instrument: VARIAN 3300,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 360 millivolts (31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.30	GOAL	3.610E+01	.231E+04	5.00	5.00	0.050

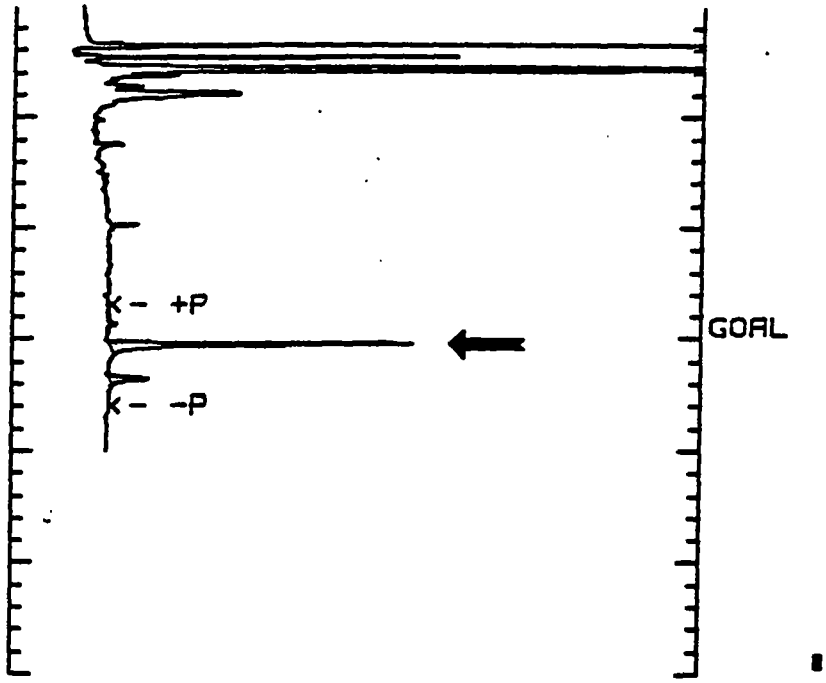
Figure 23. Grape Fortification 0.10 PPM
93-0012-001

Data file: J05J8 RAR number: 93-0012
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: THOMPSON

Sample Name: NA Cal. Curve: 01/05/95
Date: 5 Jan 1995 22:42 Method: GOALVm Analyst : SZ SZ
Interface: 711 Cycle#: 8 Channel#: A

Instrument: VARIAN 3300,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog. Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 360 millivolts (31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.25	GOAL	2.780E+02	.160E+05	5.00	5.00	0.500

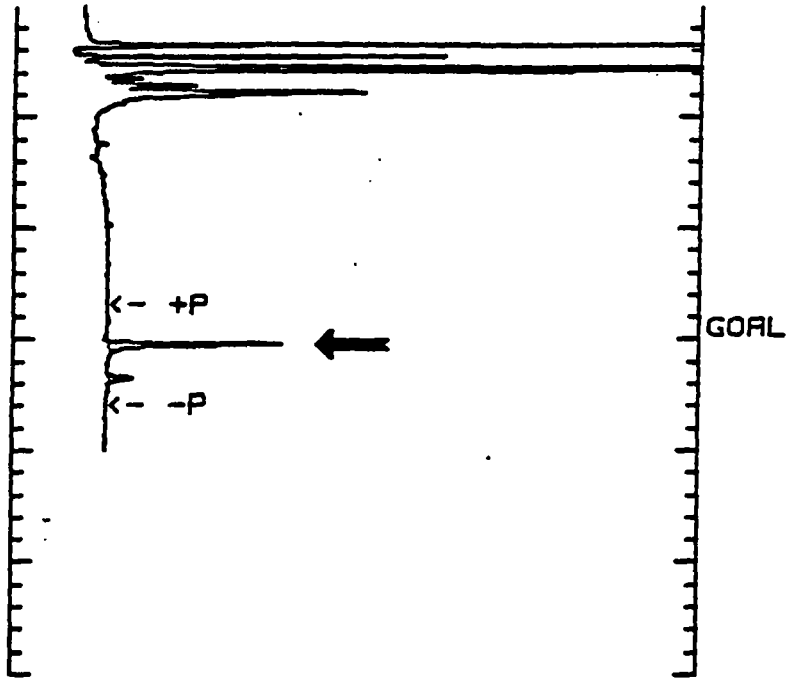
Figure 24. Grape Fortification 0.50 PPM 93-0012-001

Data file: J05J9 RAR number: 93-0012
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: THOMPSON

Sample Name: NA Cal. Curve: 01/05/95
Date: 5 Jan 1995 23:19 Method: GOALVm Analyst : SZ
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 360 millivolts (31 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	* Samp Wt.	ug Added
15.25	GOAL	1.590E+02	.902E+04	50.0	5.00	2.50

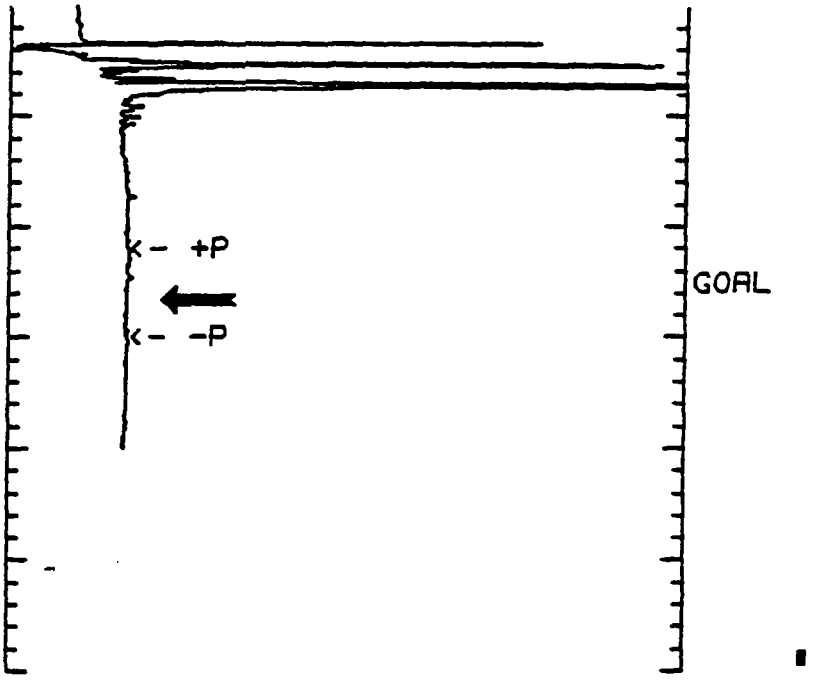
Figure 25. Kiwi Control
94-0146-001

Data file: L26JAS SAR number: 94-0146
Method file: GOALVa Sample No: 001
Type: SAMPLE Component: KIWI

Sample Name: NA Cal. Curve: 07/22/95
Date: 22 Jul 1995 10:28 Method: GOALVa Analyst : JB
Interface: 711 Cycle#: 5 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.33	GOAL	0.0000E+00	.0000E+00	5.00	5.00

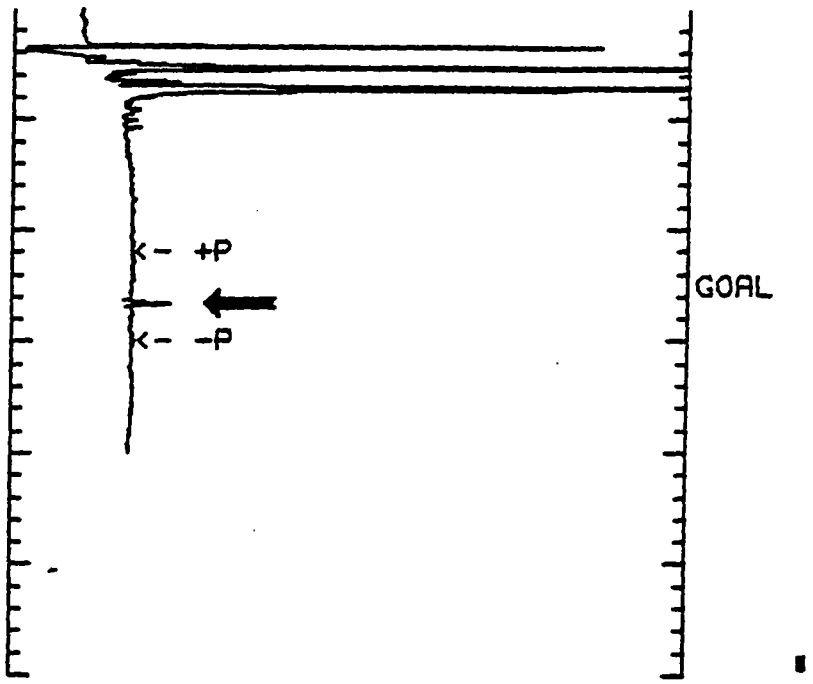
Figure 26. Kiwi Fortification 0.01 PPM
94-0146-001

Data file: L20JA6 RAR number: 94-0146
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: KIWI

Sample Name: NA Cal. Curve: 07/22/95
Date: 22 Jul 1995 18:54 Method: GOALVa Analyst : JB
Interface: 711 Cycle#: 6 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 1DC End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	3.390E+08	.281E+03	5.00	5.00	0.050

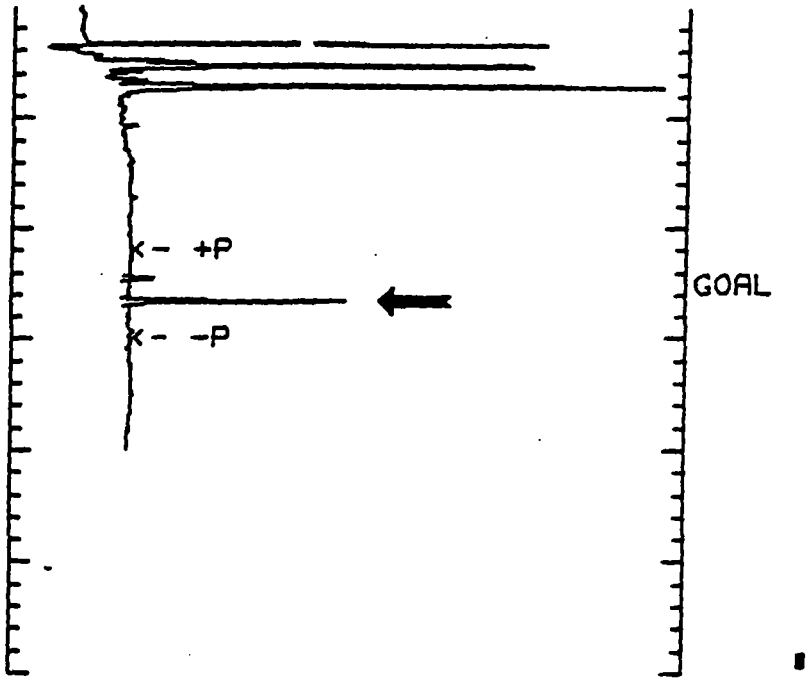
Figure 27. Kiwi Fortification 0.10 PPM 94-0146-001

Data file: L30JA7 RAR number: 94-0146
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: KIWI

Sample Name: NA Cal. Curve: 07/22/95
Date: 23 Jul 1995 11:19 Method: GOALVa Analyst : JB
Interface: 711 Cycle#: 7 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.8ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.26 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	1.8608+01	.1598+04	5.00	5.00	0.250

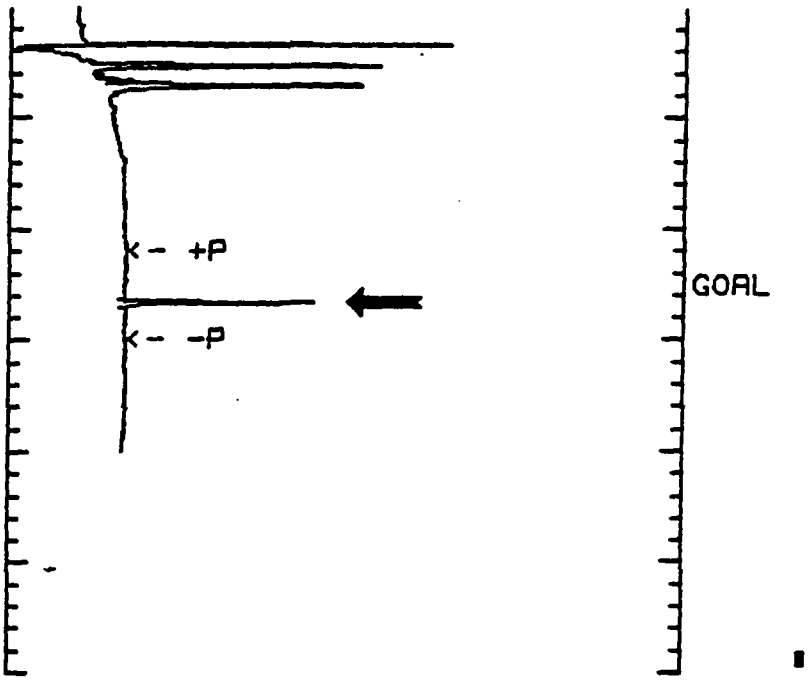
Figure 28. Kiwi Fortification 0.50 PPM
94-0146-001

Data file: L10XA8 R&R number: 94-0146
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: KIWI

Sample Name: NA Cal. Curve: 07/22/95
Date: 22 Jul 1995 11:44 Method: GOALVa Analyst : JB
Interface: 711 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.24 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	1.660E+01	.138E+04	50.0	5.00	2.50

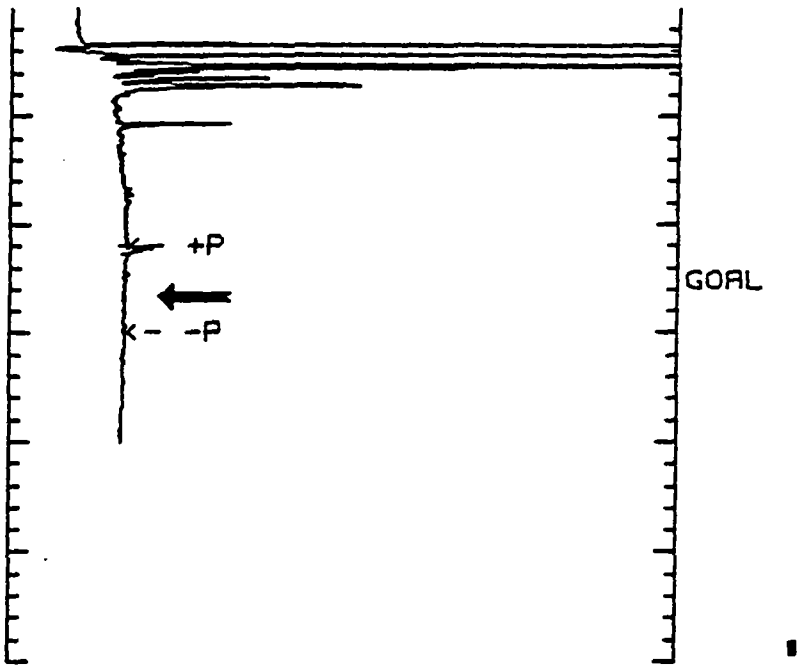
Figure 29. Olive Control
94-0172-001

Data file: L07513 RAR number: 94-0172
Method file: GOALVa Sample No: 0013
Type: SAMPLE Component: OLIVE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 20:52 Method: GOALVa Analyst : SS SZ
Interface: 711 Cycle#: 13 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.25 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.35	GOAL	0.000E+00	.000E+00	5.00	5.00

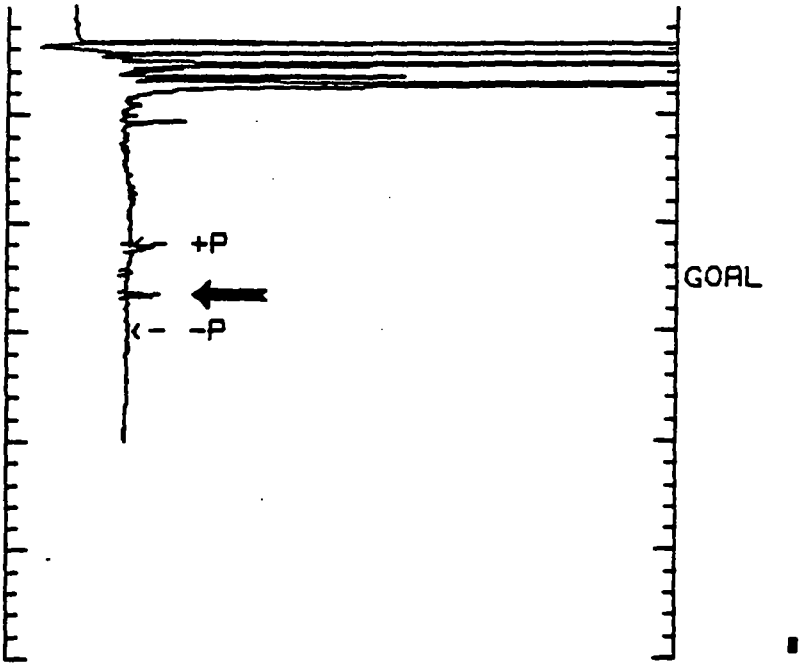
Figure 30. Olive Fortification 0.01 PPM
94-0172-001

Data file: L07816 RAR number: 94-0172
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: OLIVE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 22:07 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 16 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.3 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	3.040E+00	.249E+03	5.00	5.00	0.050

TR 34 95 111

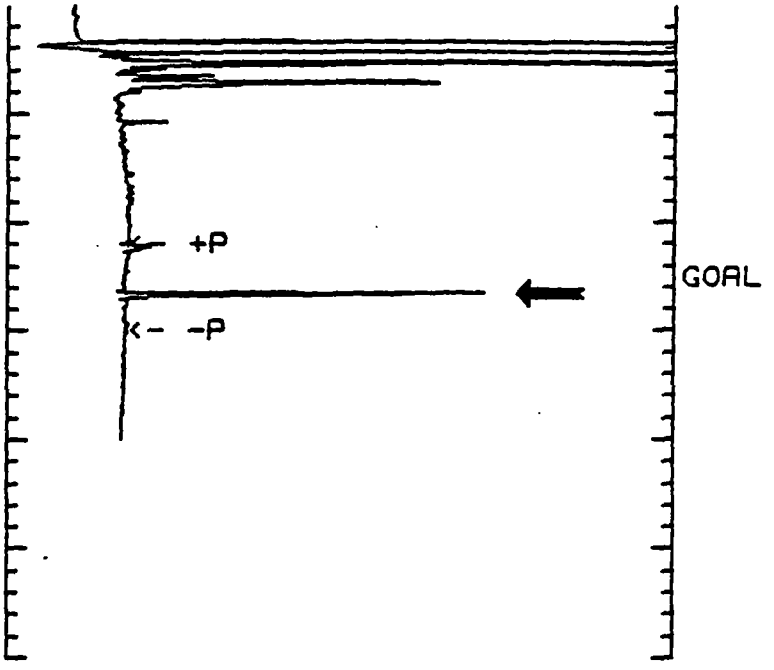
Figure 31. Olive Fortification 0.10 PPM
94-0172-001

Data file: L07817 RAR number: 94-0172
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: OLIVE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 22:32 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 17 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 230 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.28 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	3.180E+01	.265E+04	5.00	5.00	0.500

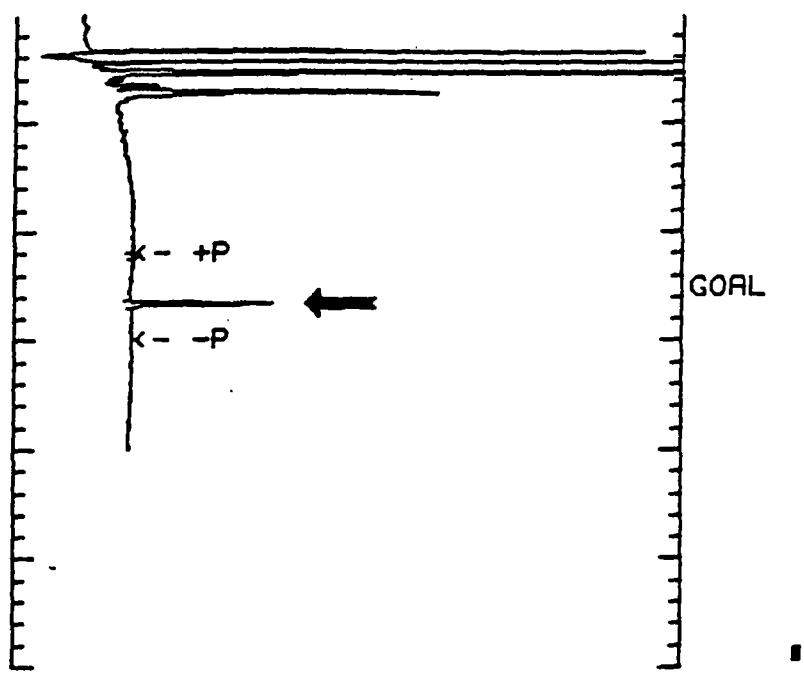
Figure 32. Olive Fortification 0.50 PPM
94-0172-001

Data file: L07816 RAR number: 94-0172
Method file: GOALVn Sample No: 001
Type: FORTIFICATION Component: OLIVE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 22:58 Method: GOALVn Analyst : RS
Interface: 711 Cycle0: 18 Channel: A

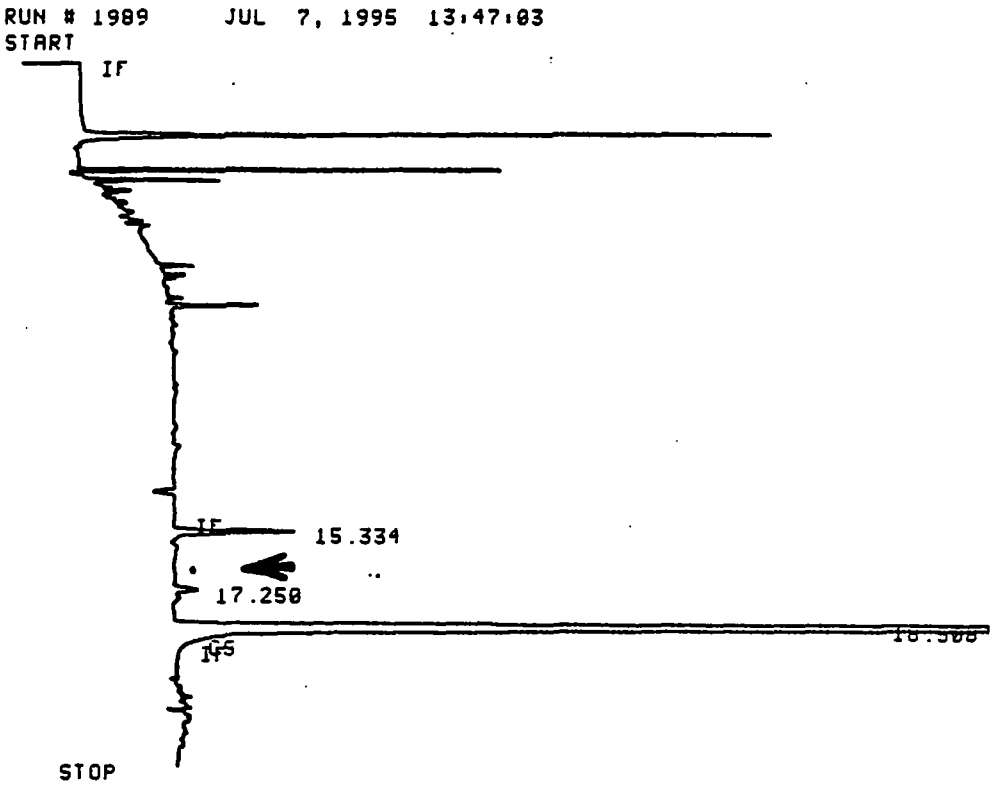
Instrument: VARIAN 3600,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.25 uv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	1.320E+01	.186E+04	50.0	5.00	2.50

Figure 33. Peach Control
94-0117-001



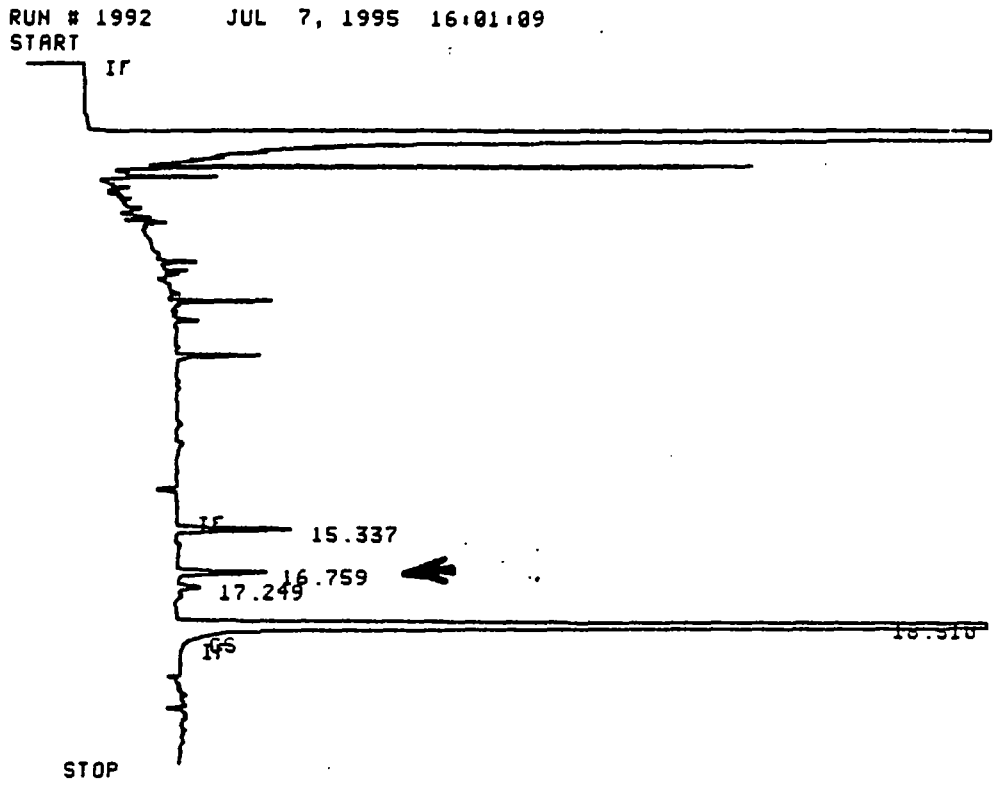
RUN# 1989 JUL 7, 1995 13:47:03
FRUIT
SAMPLE NAME: 94-0117-001 SAMPLE# 6 CONTROL
METHOD NAME: M:GOALEXT.MET
G FINAL WT____ VOL INJ____ ML FINAL VOL____ DIL FACTOR____ MG INJ____

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
15.334	4164	BB	.118	7.55470
17.250	812	PV	.143	1.47320
18.508	50142	PB	.199	90.97210

TOTAL HEIGHT= 55118
MUL FACTOR=1.0000E+00

Figure 34. Peach Fortification 0.01 PPM
94-0117-001



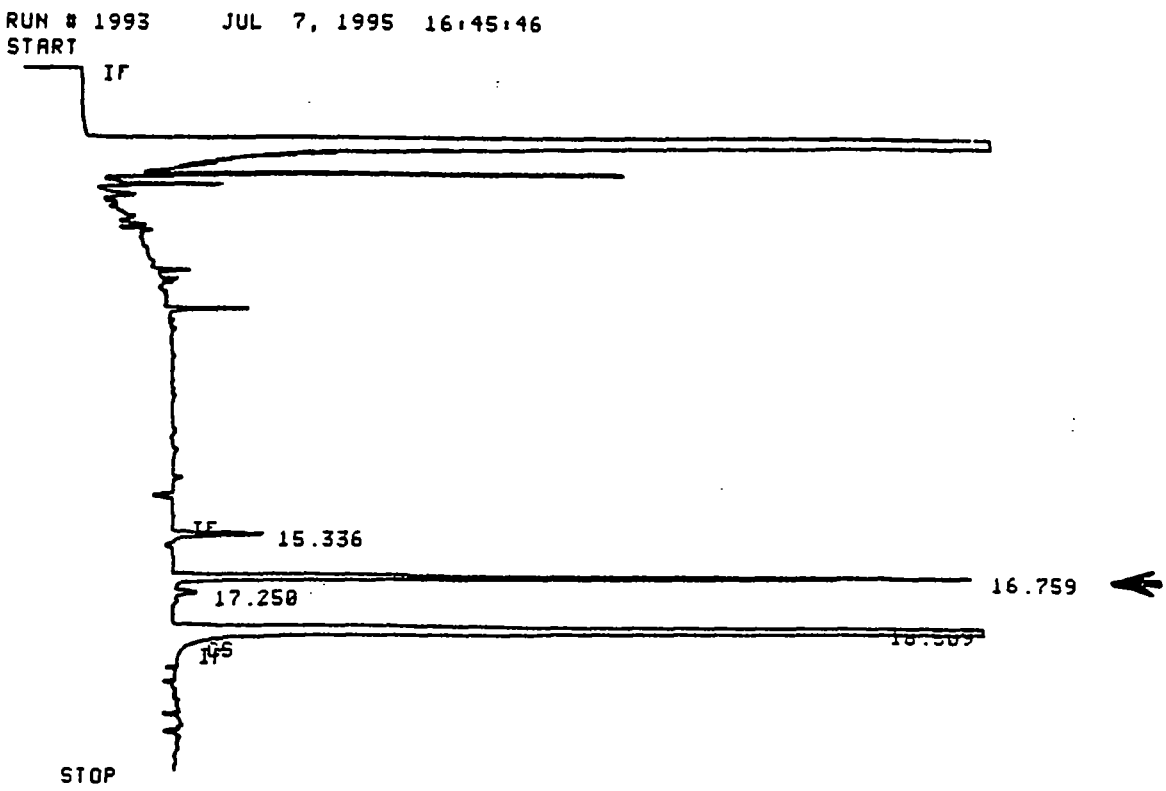
RUN# 1992 JUL 7, 1995 16:01:09
FRUIT Fortification
SAMPLE NAME: 0.01ppm SAMPLE# 9 94-0117-001
METHOD NAME: M:GOALEXT.MET
G FINAL WT 5 VOL INJ 1 ML FINAL VOL 5 DIL FACTOR 1 MG INJ 1

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
15.337	4040	BB	.115	7.08685
16.759	3154	PB	.122	5.53265
17.249	765	PB	.129	1.34194
18.510	49048	PB	.199	86.03853

TOTAL HEIGHT= 57007
MUL FACTOR=1.0000E+00

Figure 35. Peach Fortification 0.10 PPM
94-0117-001



RUN# 1993 JUL 7, 1995 16:45:46

FRUIT Fortification

SAMPLE NAME: 0.10ppm SAMPLE# 10 94-0117-001

METHOD NAME: M:GOALEXT.MET

G FINAL WT 5 VOL INJ 1ul ML FINAL VOL 5 DIL FACTOR 1 MG INJ 1

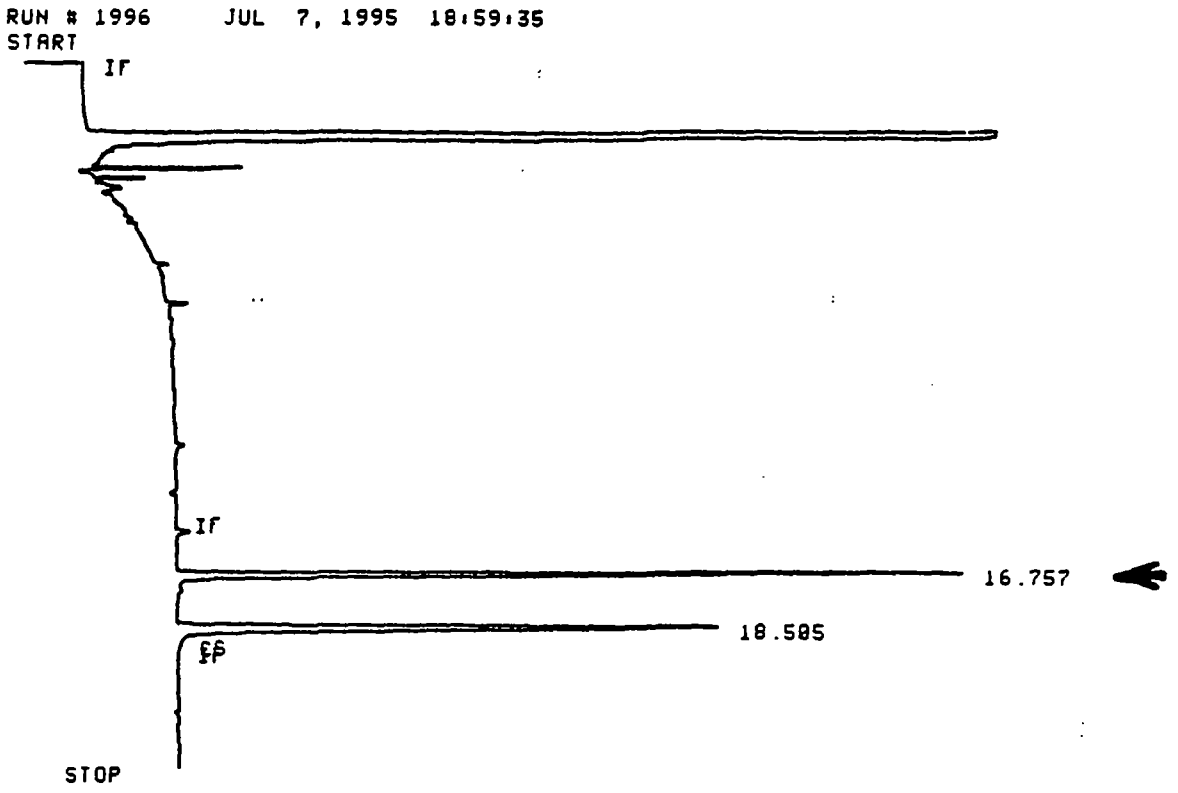
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
15.336	3362	BB	.133	4.00271
16.759	28139	PB	.125	33.50160
17.250	751	PB	.122	.89412
18.509	51741	PB	.203	61.60157

TOTAL HEIGHT= 83993

MUL FACTOR=1.0000E+00

Figure 36. Peach Fortification 0.50 PPM
94-0117-001



RUN# 1996 JUL 7, 1995 18:59:35

FRUIT Fortification

SAMPLE NAME: 0.5 ppm SAMPLE# 13 94-0117-001

METHOD NAME: M:GOALEXT.MET

G FINAL WT 5 VOL INJ 1ML ML FINAL VOL 5 DIL FACTOR 5 MG INJ 0.2

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
16.757	27553	PB	.124	59.21939
18.505	18974	PB	.173	40.78062

TOTAL HEIGHT= 46527

MUL FACTOR=1.0000E+00

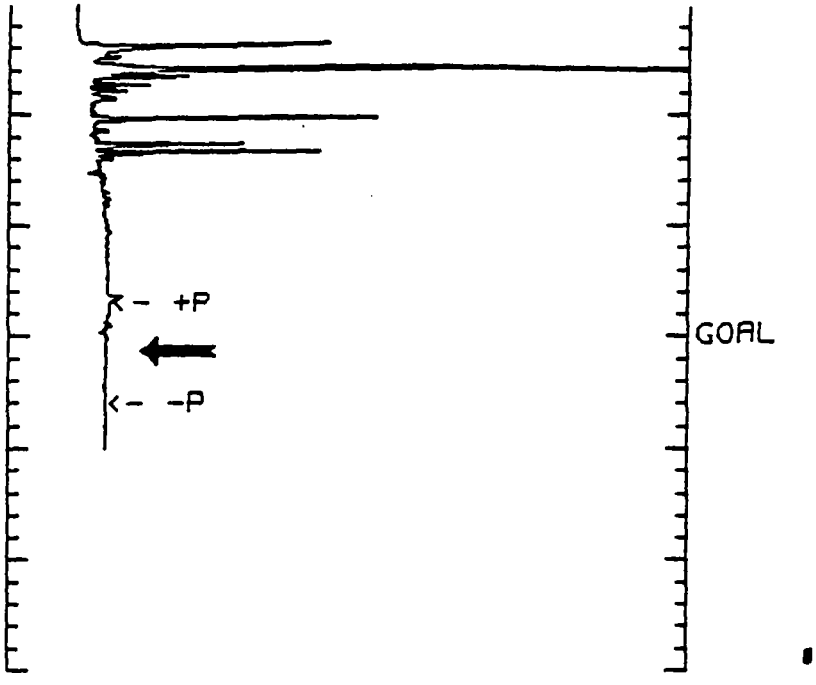
Figure 37. Peanut Nutmeat Control 94-0176-010

Data file: T1086 RAR number: 94-0176
Method file: GOALVm Sample No: 010
Type: SAMPLE Component: NUT

Sample Name: NA Cal. Curve: 05/16/95
Date: 16 May 1995 16:23 Method: GOALVm Analyst : SS
Interface: 707 Cycle#: 6 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
15.63	GOAL	0.000E+00	.000E+00	5.00	5.00

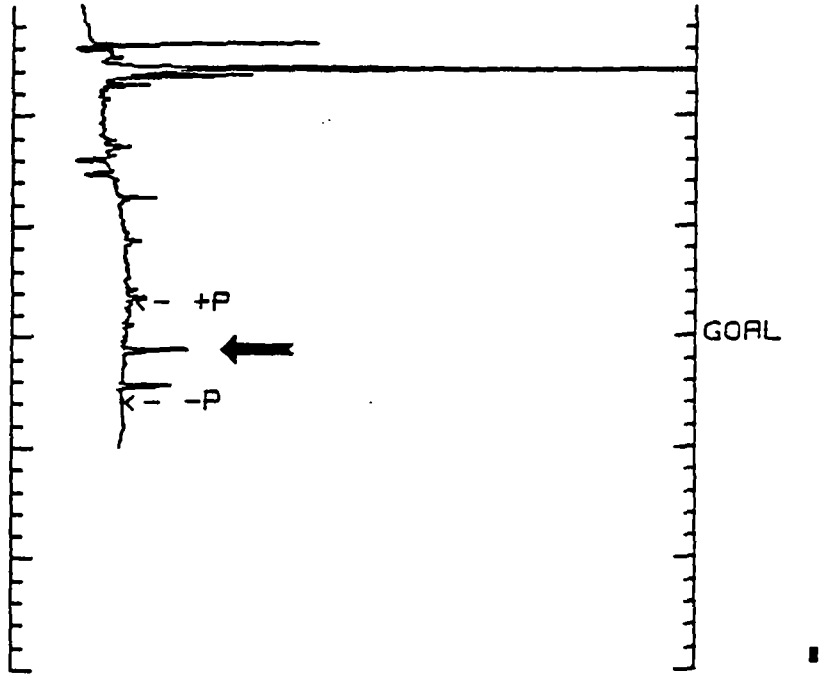
Figure 38. Peanut Nutmeat Fortification 0.01 PPM 93-0142-005

Data file: Y048A9 RAR number: 93-0142
Method file: GOALVm Sample No: 005
Type: FORTIFICATION Component: NUT

Sample Name: NA Cal. Curve: 05/12/95
Date: 12 May 1995 19:29 Method: GOALVm Analyst : SZ
Interface: 707 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: SCD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.62	GOAL	8.520E+01	.680E+04	5.00	5.00	0.050

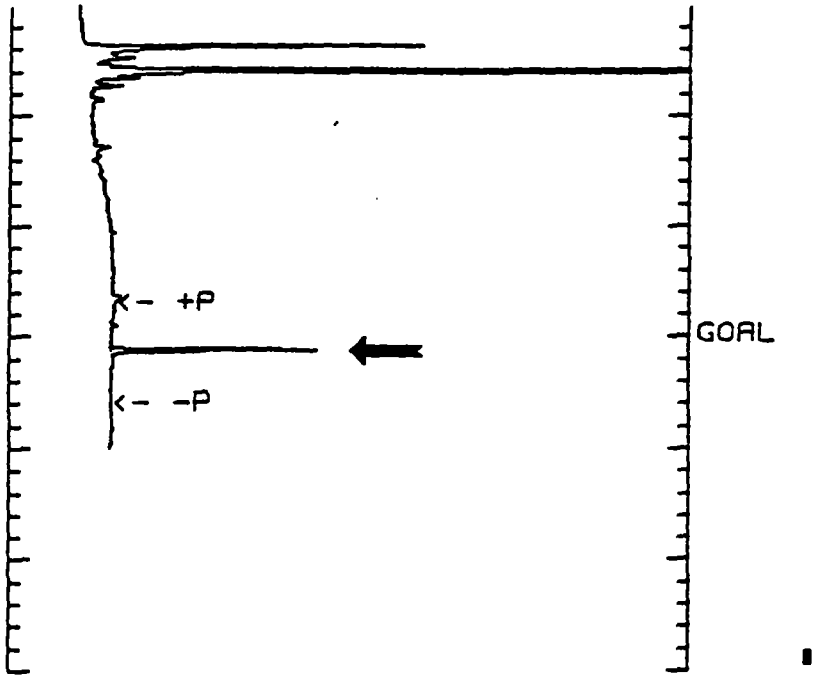
Figure 39. Peanut Nutmeat Fortification 0.10 PPM 94-0176-010

Data file: Y1038 RAR number: 94-0176
Method file: GOALVm Sample No: 010
Type: FORTIFICATION Component: NUT

Sample Name: NA Cal. Curve: 05/16/95
Date: 16 May 1995 17:37 Method: GOALVm Analyst : SS
Interface: 707 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.63	GOAL	2.790E+02	.224E+05	10.0	5.00	0.500

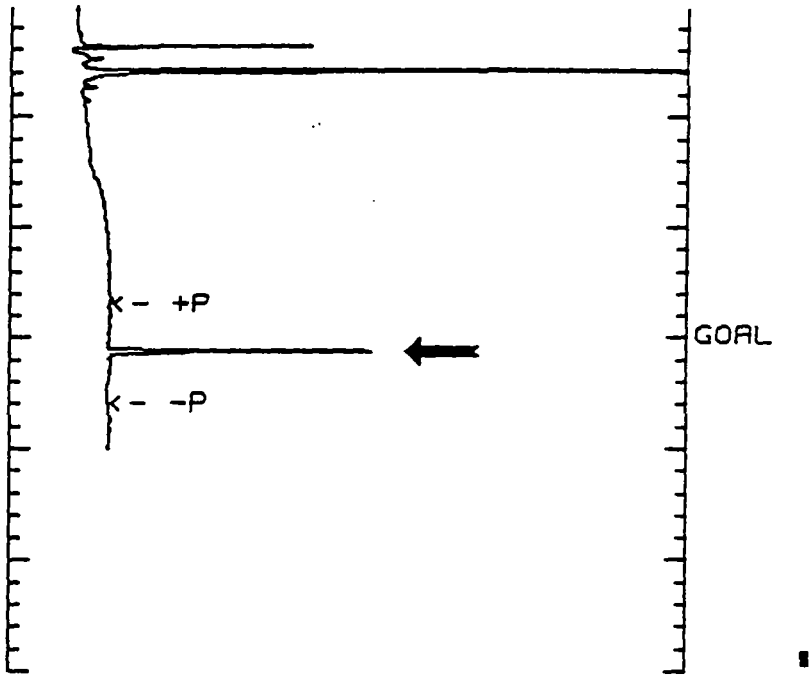
Figure 40. Peanut Nutmeat Fortification 0.50 PPM 93-0157-004

Data file: Y048A11 RAR number: 93-0157
Method file: GOALVa Sample No: 004
Type: FORTIFICATION Component: NUT

Sample Name: NA Cal. Curve: 05/12/95
Date: 13 May 1995 20:43 Method: GOALVa Analyst : SS
Interface: 707 Cycles: 11 Channels: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.61	GOAL	1.570E+02	.287E+05	50.0	5.00	2.50

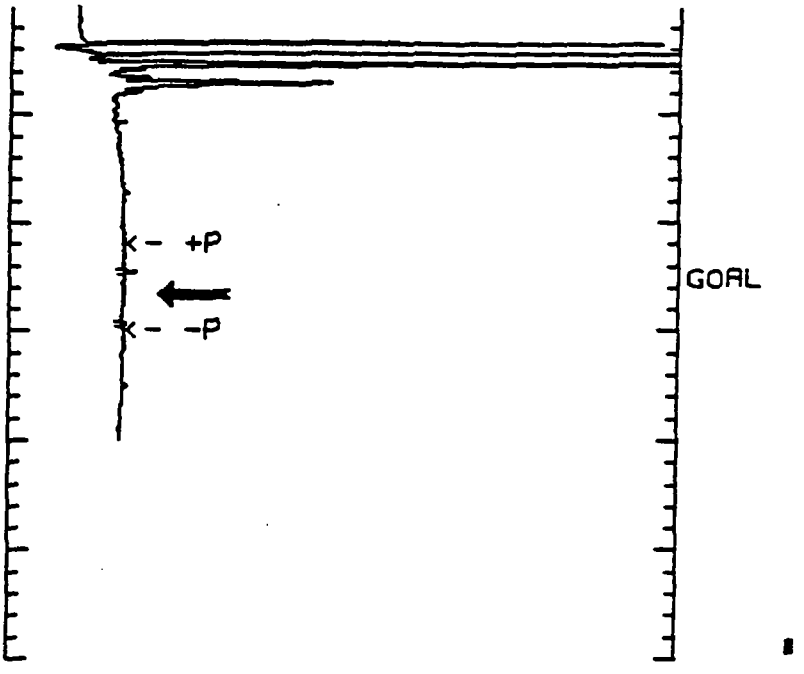
Figure 41. Pomegranate Control
94-0143-001

Data file: L0785 RAR number: 94-0143
Method file: GOALVa Sample No: 0013
Type: SAMPLE Component: POMEGRANATE

Sample Name: MA Cal. Curve: 07/07/95
Date: 7 Jul 1995 17:31 Method: GOALVa Analyst : SS
Interface: 711 Cycle: 5 Channels: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): MA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min Split Ratio: MA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.28 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.35	GOAL	0.000E+00	.000E+00	5.00	5.00

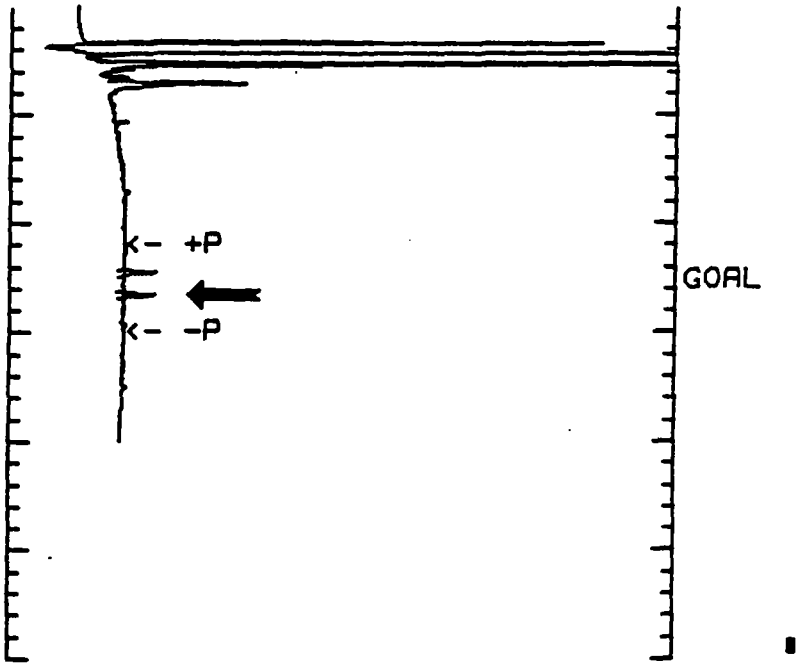
Figure 42. Pomegranate Fortification 0.01 PPM 94-0143-001

Data file: L0788 RAR number: 94-0143
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: POMEGRANATE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 18:46 Method: GOALVa Analyst : SS
Interface: 711 Cycle#: 8 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.26 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	2.660E+00	.220E+03	5.00	5.00	.0.050

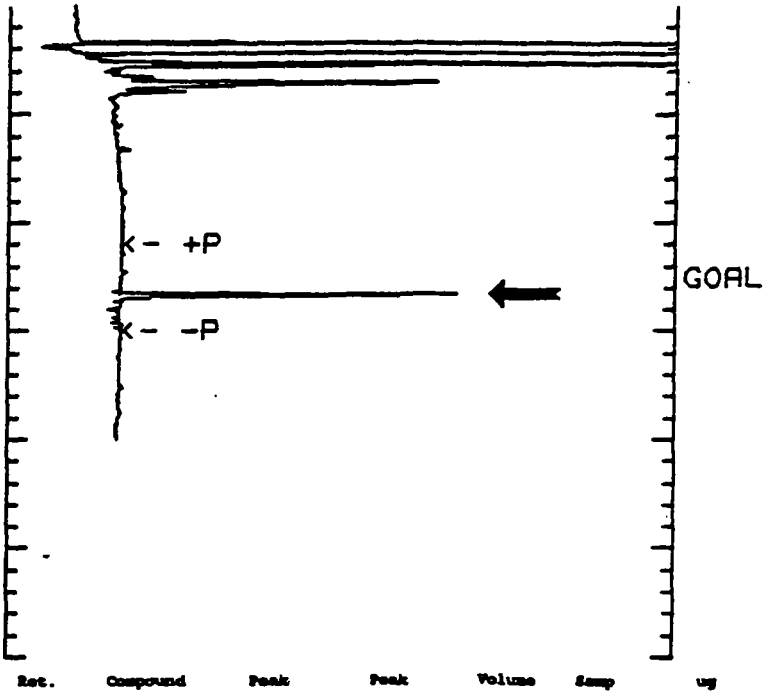
Figure 43. Pomegranate Fortification 0.10 PPM 94-0143-001

Data file: L0789 RAR number: 94-0143
Method file: GOALVn Sample No: 001
Type: FORTIFICATION Component: POMEGRANATE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 19:11 Method: GOALVn Analyst : SS
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.27 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	3.0308+01	.2508+04	5.00	5.00	0.500

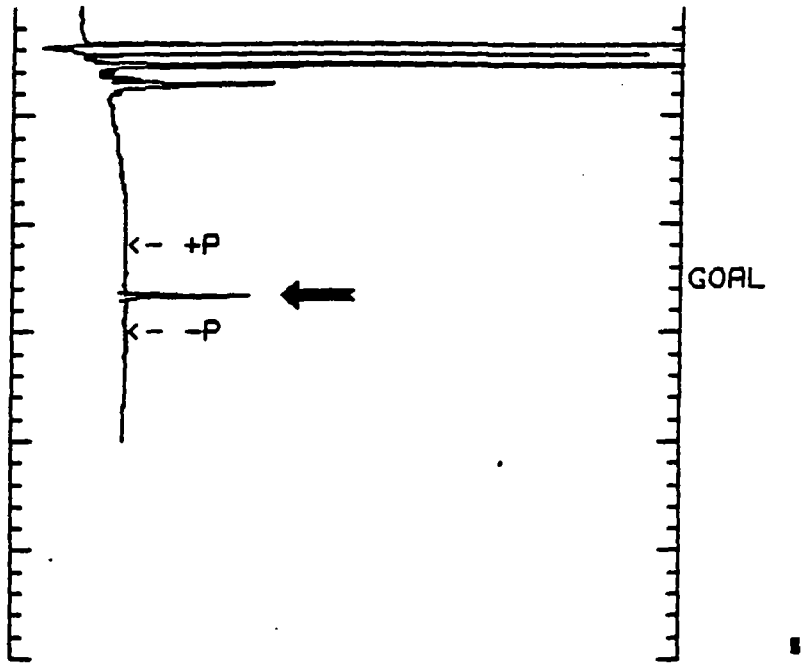
Figure 44. Pomegranate Fortification 0.50 PPM 94-0143-001

Data file: L07S10 RAR number: 94-0143
Method file: GOALVs Sample No: 001
Type: FORTIFICATION Component: POMEGRANATE

Sample Name: NA Cal. Curve: 07/07/95
Date: 7 Jul 1995 19:37 Method: GOALVs Analyst : SS
Interface: 711 Cycle#: 10 Channel#: A

Instrument: VARIAN 3500.
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.27 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.33	GOAL	1.180E+01	.945E+03	50.0	5.00	2.50

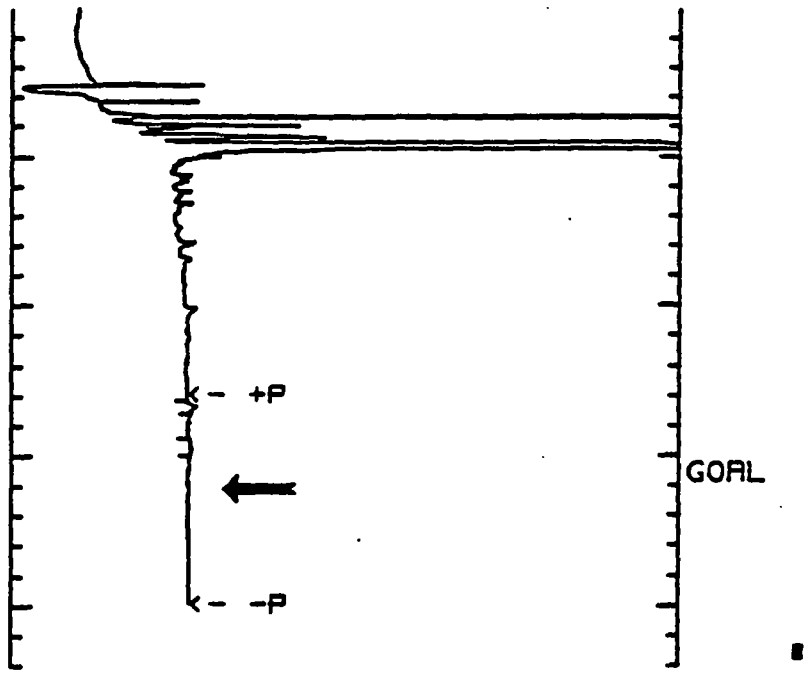
Figure 45. Soybean Control
92-0107-014

Data file: X0009949 RAR number: 92-0107
Method file: MGOALP1m Sample No: 014
Type: SAMPLE Component: SOYBEAN

Sample Name: SOYBEAN Cal. Curve: 11-9-94
Date: 9 Nov 1994 12:27 Method: MGOALP1m Operator: DAN
Interface: 711 Cycle#: 9 Channel#: A

Instrument: VARIAN 3300
Column: Rtx-200 Column Length: 60 Meters
Start Temp-Time (deg-min): 215 Ramp Hold (deg-min): 17
Program Rate (deg/min): 10 End Time-Temp (deg-min): 250
Prog Slope (# or Linear): Inj Port Temp: 265
Flowrate/Gas: 3.5 Split Ratio:
Det 1-Type & Temp: Det 2-Type & Temp: ECD/270

Plot times: 0 to 22 minutes
Plot range: 50 millivolts (-.038 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
16.10	GOAL	0.000E+00	.000E+00	5.00	5.00

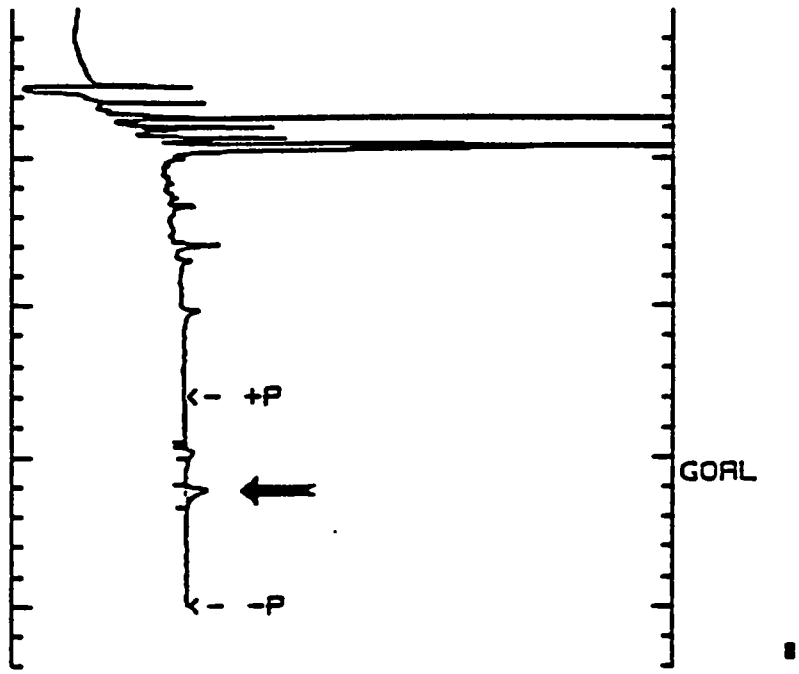
Figure 46. Soybean Fortification 0.01 PPM 92-0107-014

Data file: XN099410 RAR number: 92-0107
Method file: MGOALP1m Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: SOYBEAN Cal. Curve: 11-9-94
Date: 9 Nov 1994 12:52 Method: MGOALP1m Operator: DAN
Interface: 711 Cycle#: 10 Channel#: A

Instrument: VARIAN 3300
Column: Rtx-200 Column Length: 60 Meters
Start Temp-Time (deg-min): 215 Ramp Hold (deg-min): 17
Program Rate (deg/min): 10 End Time-Temp (deg-min): 250
Prog Slope (# or Linear): Inj Port Temp: 265
Flowrate/Gas: 3.5 Split Ratio:
Det 1-Type & Temp: Det 2-Type & Temp: ECD/270

Plot time: 0 to 22 minutes
Plot range: 50 millivolts (-.092 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
16.09	GOAL	5.260E+00	.164E+03	5.00	5.00	0.050

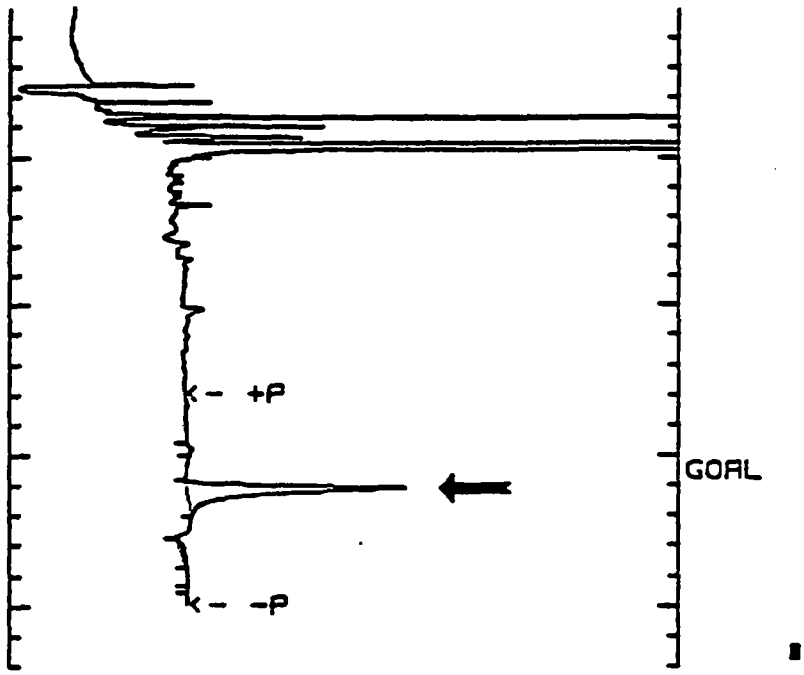
Figure 47. Soybean Fortification 0.10 PPM 92-0107-014

Data file: XH099412 RAR number: 92-0107
Method file: MOOALP1m Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: SOYBEAN Cal. Curve: 11-9-94
Date: 9 Nov 1994 13:42 Method: MOOALP1m Operator: DAN
Interface: 711 Cycle#: 12 Channel#: A

Instrument: VARIAN 3300
Column: Rtx-200 Column Length: 60 Meters
Start Temp-Time (deg-min): 215 Temp Hold (deg-min): 17
Program Rate (deg/min): 10 End Time-Temp (deg-min): 250
Prog Slope (0 or Linear): Inj Port Temp: 265
Flowrate/Gas: 3.5 Split Ratio:
Det 1-Type & Temp: Det 2-Type & Temp: ECD/270

Plot time: 0 to 22 minutes
Plot range: 50 millivolts (.001 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
16.08	GOAL	5.410E+01	.160E+04	5.00	5.00	0.500

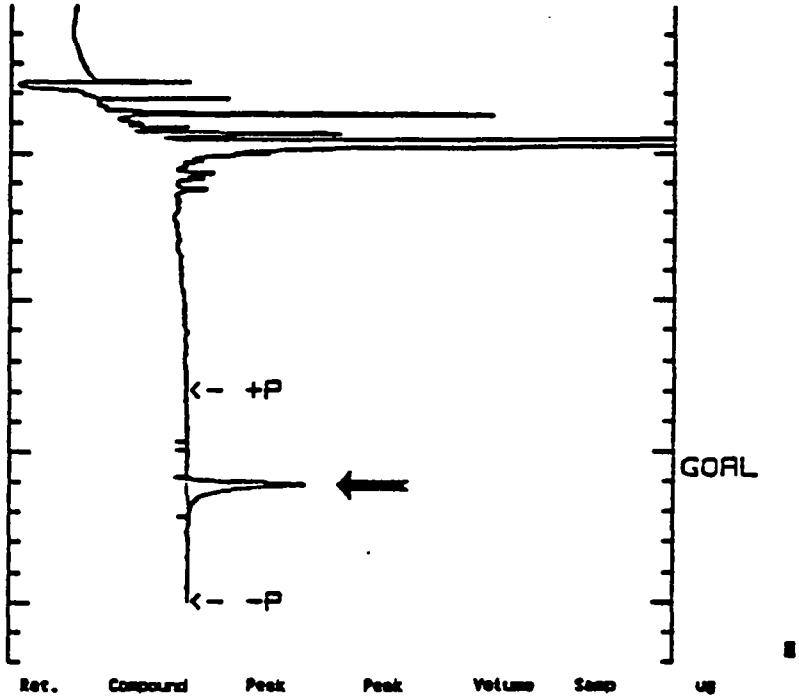
Figure 48. Soybean Fortification 0.50 PPM 92-0107-014

Data file: XE099414 RAR number: 92-0107
Method file: HGOALP1m Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: SOYBEAN Cal. Curve: 11-9-96
Date: 9 Nov 1996 16:32 Method: HGOALP1m Operator: DAN
Interface: 711 Cycle#: 14 Channel#: A

Instrument: VARIAN 3500
Column: Rtx-200 Column Length: 60 Meters
Start Temp-Time (deg-min): 215 Ramp Hold (deg-min): 17
Program Rate (deg/min): 10 End Time-Temp (deg-min): 250
Prog Slope (# or Linear): Inj Port Temp: 285
Flowrate/Gas: 3.5 Split Ratio:
Det 1-Type & Temp: Det 2-Type & Temp: ECD/270

Plot time: 0 to 22 minutes
Plot range: 50 millivolts (-.038 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
16.09	GOAL	2.890E+01	.897E+03	50.0	5.00	2.50

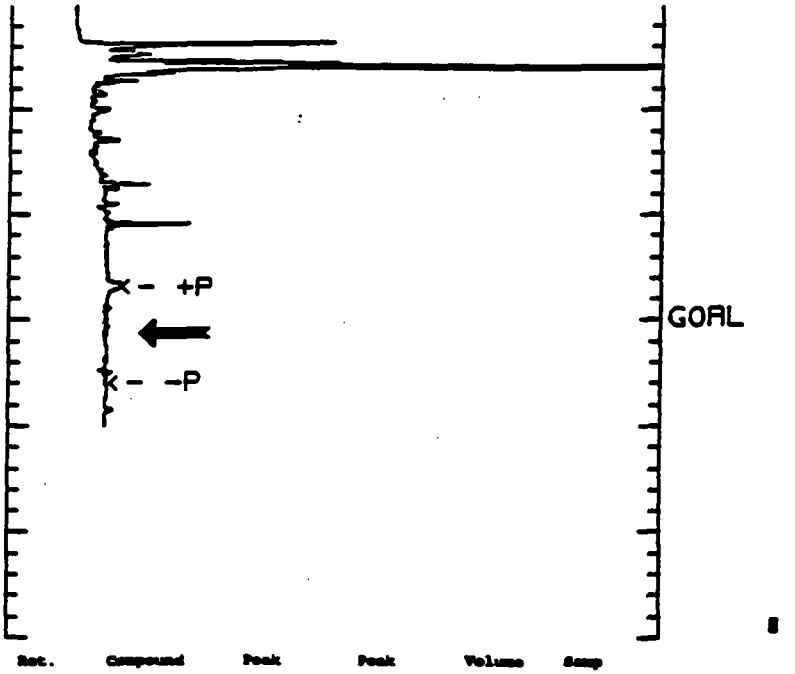
Figure 49. Peanut Hay Control
93-0142-003

Data file: T1187 RAR number: 93-0142
Method file: GOALVa Sample No: 003
Type: SAMPLE Component: HAY

Sample Name: HA Cal. Curve: 05/16/95
Date: 16 May 1995 11:37 Method: GOALVa Analyst : SS
Interface: 707 Cycle#: 7 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): HA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: HA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: HA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
15.63	GOAL	1.8782e+00	.1468e+03	10.0	5.00

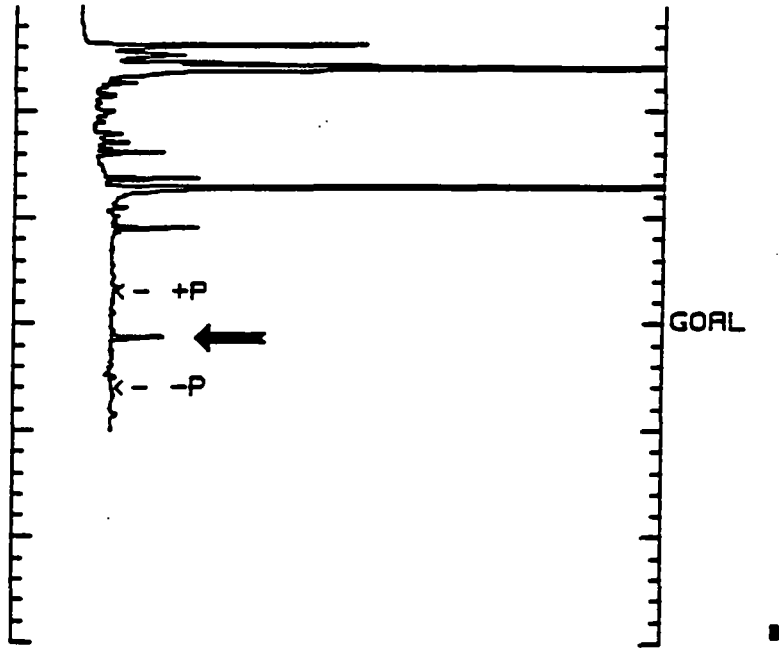
Figure 50. Peanut Hay Fortification 0.02 PPM 93-0157-002

Data file: T12S12 RAR number: 93-0157
Method file: GOALVm Sample No: 002
Type: FORTIFICATION Component: HAY

Sample Name: NA Cal. Curve: 05/16/95
Date: 16 May 1995 14:41 Method: GOALVm Analyst : SS
Interface: 787 Cycle8: 12 Channel8: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 784 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.61	GOAL	7.766E+01	.596E+04	10.0	5.00	0/100

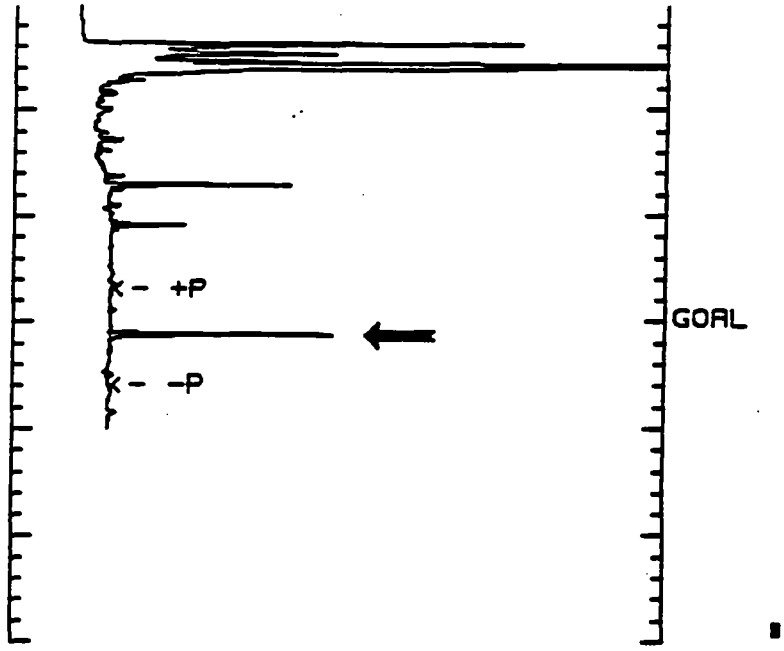
Figure 51. Peanut Hay Fortification 0.10 PPM 93-0142-003

Data file: T12E11 RAR number: 93-0142
Method file: GOALVa Sample No: 003
Type: FORTIFICATION Component: HAY

Sample Name: HA Cal. Curve: 05/16/95
Date: 16 May 1995 14:04 Method: GOALVa Analyst : SS
Interface: 707 Cycles: 11 Channels: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): HA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: HA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: HA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.61	GOAL	3.170E+02	.256E+05	10.0	5.00	0.500

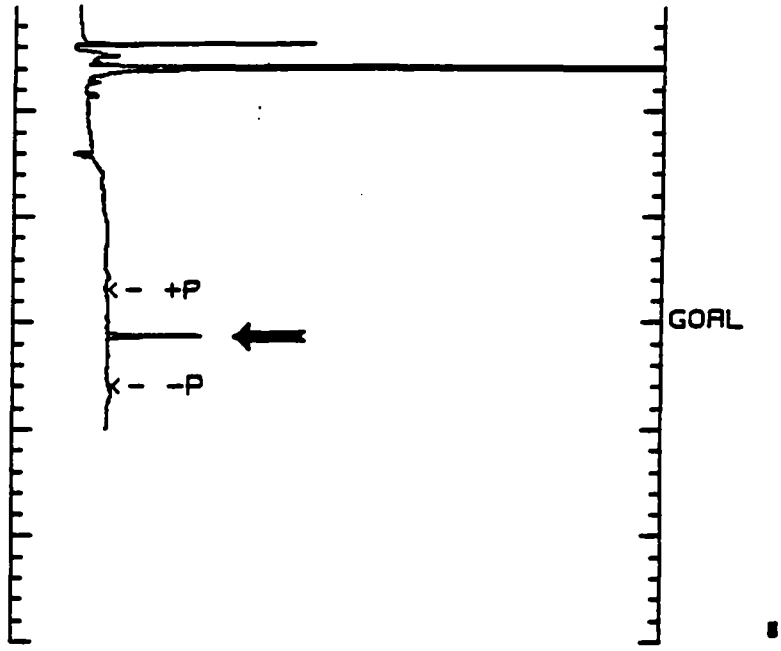
Figure 52. Peanut Hay Fortification 0.50 PPM 93-0157-002

Data file: T12813 RAR number: 93-0157
Method file: GOALVa Sample No: 002
Type: FORTIFICATION Component: HAY

Sample Name: HA Cal. Curve: 05/16/95
Date: 16 May 1995 15:16 Method: GOALVa Analyst : SS
Interface: 707 Cycle0: 13 Channel0: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 8C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): HA Inj Port Temp: 265
Flowrate/Gas: 3.8ml/min. Split Ratio: HA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: HA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.62	GOAL	1.388E+02	.188E+05	100	5.00	2.50

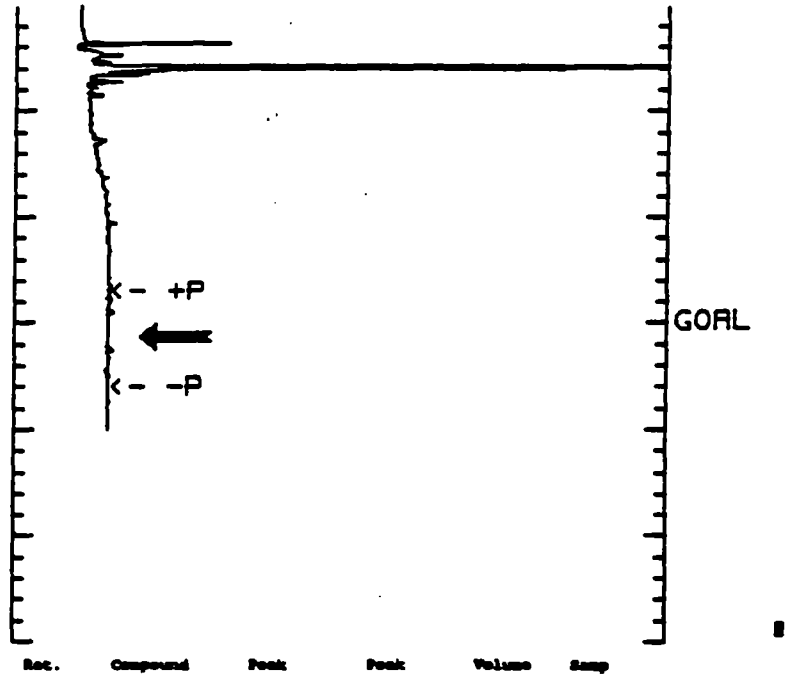
Figure 53. Peanut Shell Control
93-0157-004

Data file: T1SS6 RAR number: 93-0157
Method file: GOALVm Sample No: 004
Type: SAMPLE Component: SHELL

Sample Name: NA Cal. Curve: 05/17/95
Date: 17 May 1995 13:12 Method: GOALVm Analyst : SZ
Interface: 707 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 230 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: SCD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
15.64	GOAL	0.000E+00	.000E+00	10.0	5.00

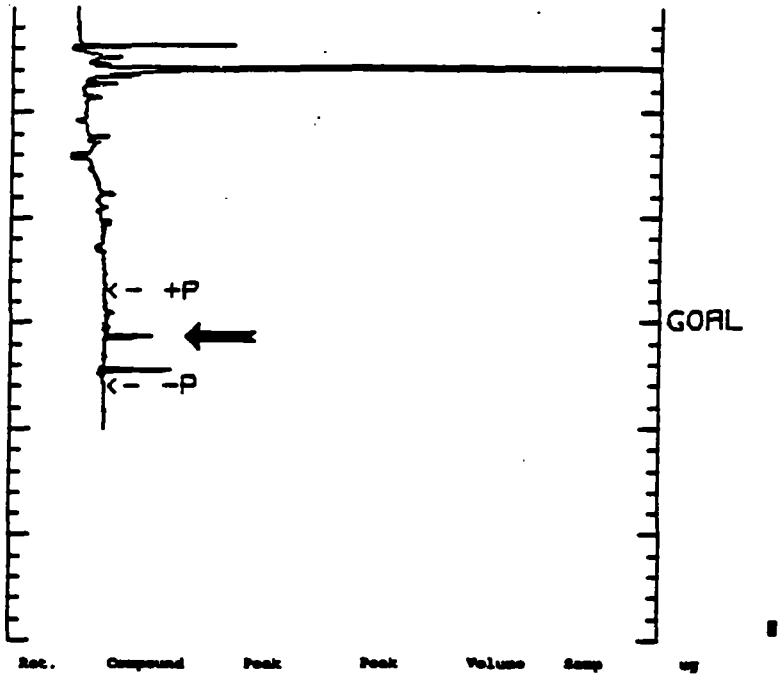
Figure 54. Peanut Shell Fortification 0.02 PPM 93-0142-005

Data file: Y18810 RAR number: 93-0142
Method file: GOALVa Sample No: 005
Type: FORTIFICATION Component: SHELL

Sample Name: NA Cal. Curve: 05/17/95
Date: 17 May 1995 14:26 Method: GOALVa Analyst : SE
Interface: 787 Cycles: 10 Channels: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 1.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.64	GOAL	6.990E+01	.347E+04	10.0	5.00	0.100

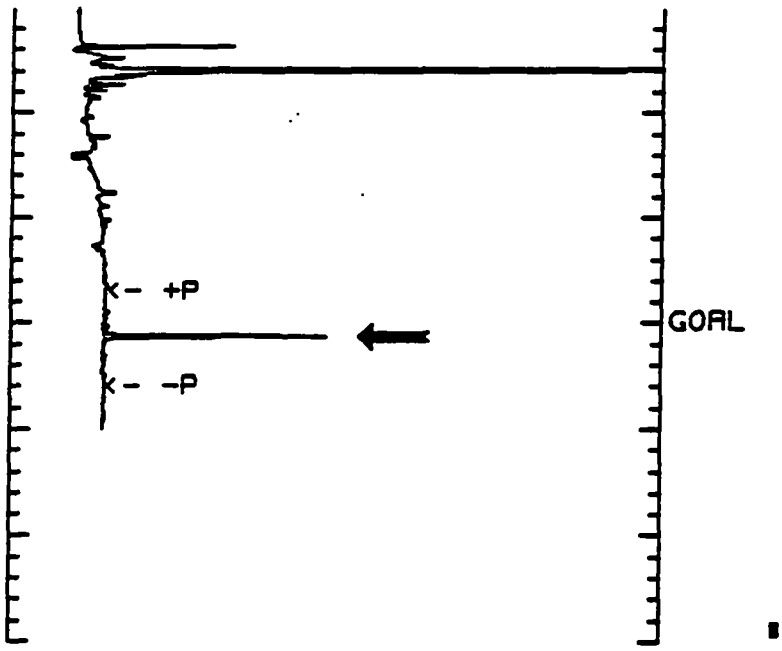
Figure 55. Peanut Shell Fortification 0.10 PPM 93-0142-005

Data file: T15S11 RAR number: 93-0142
Method file: GOALVa Sample No: 005
Type: FORTIFICATION Component: SHELL

Sample Name: NA Cal. Curve: 05/17/93
Date: 17 May 1993 15:03 Method: GOALVa Analyst : SS
Interface: 787 Cycle#: 11 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.63	GOAL	3.1908e+02	.2542e+01	10.0	5.00	0.500

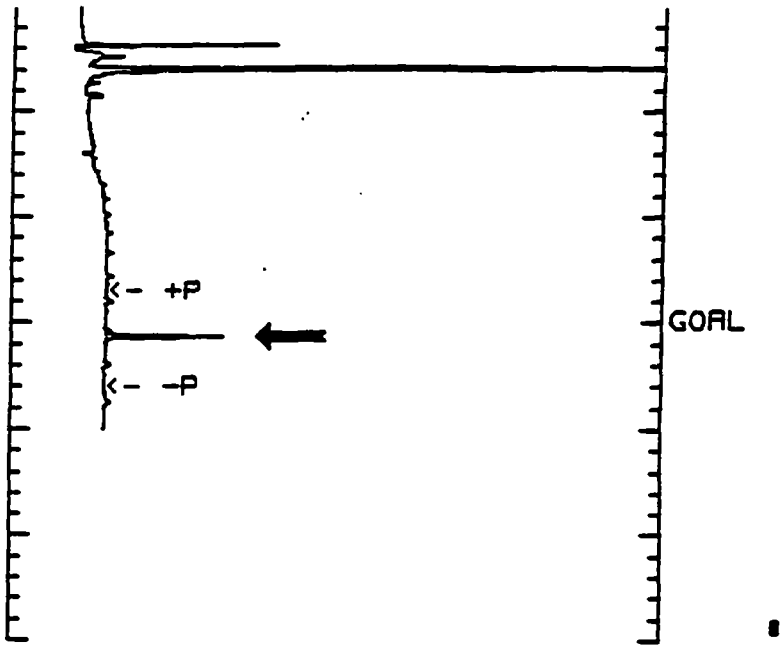
Figure 56. Peanut Shell Fortification 0.50 PPM 93-0157-004

Data file: Y15813 RAR number: 93-0157
Method file: GOALVa Sample No: 004
Type: FORTIFICATION Component: SHELL

Sample Name: NA Cal. Curve: 05/17/95
Date: 17 May 1995 16:17 Method: GOALVa Analyst : SS
Interface: 707 Cycle#: 13 Channel#: A

Instrument: VARIAN 3600,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.64	GOAL	1.740E+02	.135E+05	100	5.00	2.50

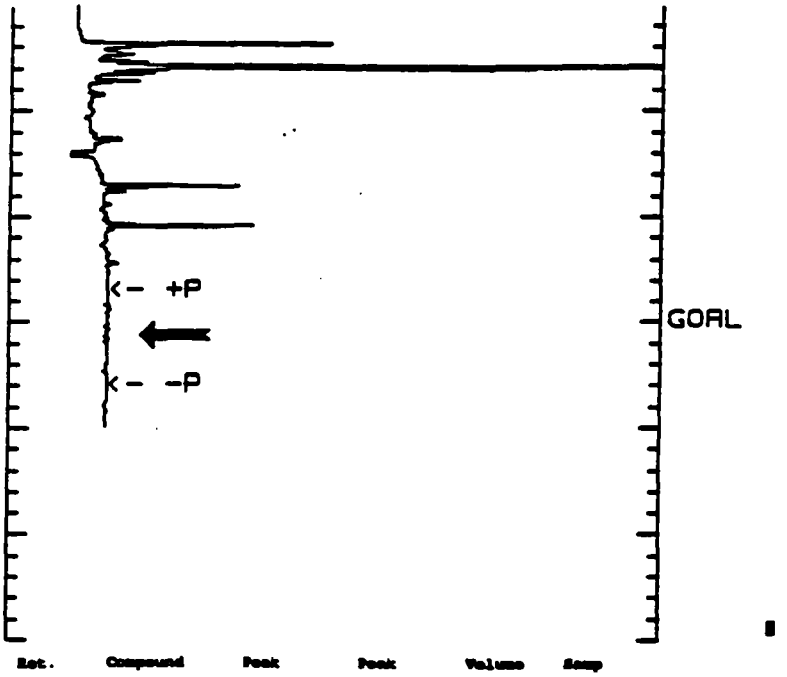
Figure 57. Peanut Vine Control
93-0142-001

Data file: T1157 RAR number: 93-0142
Method file: GOALVn Sample No: 061
Type: SAMPLE Component: VINE

Sample Name: NA Cal. Curve: 05/15/95
Date: 15 May 1995 11:17 Method: GOALVn Analyst : JB
Interface: 787 Cycles: 7 Channels: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 780 millivolts (78 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Amnt Wt.
15.62	GOAL	0.000E+00	.000E+00	10.0	0.00

TR 34 95 111

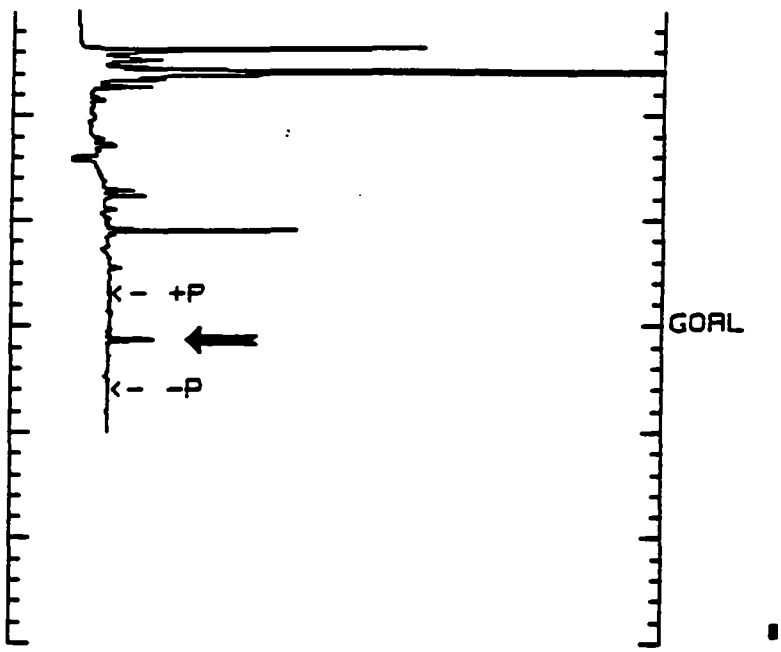
Figure 58. Peanut Vine Fortification 0.02 PPM 93-0142-001

Data file: T11514 RAR number: 93-0142
Method file: GOALVn Sample No: 001
Type: FORTIFICATION Component: VINE

Sample Name: NA Cal. Curve: 05/15/95
Date: 15 May 1995 15:33 Method: GOALVn Analyst : JB
Interface: 707 Cycle0: 14 Channel0: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.63	GOAL	6.5502e+01	.5075e+04	10.0	5.00	0.100

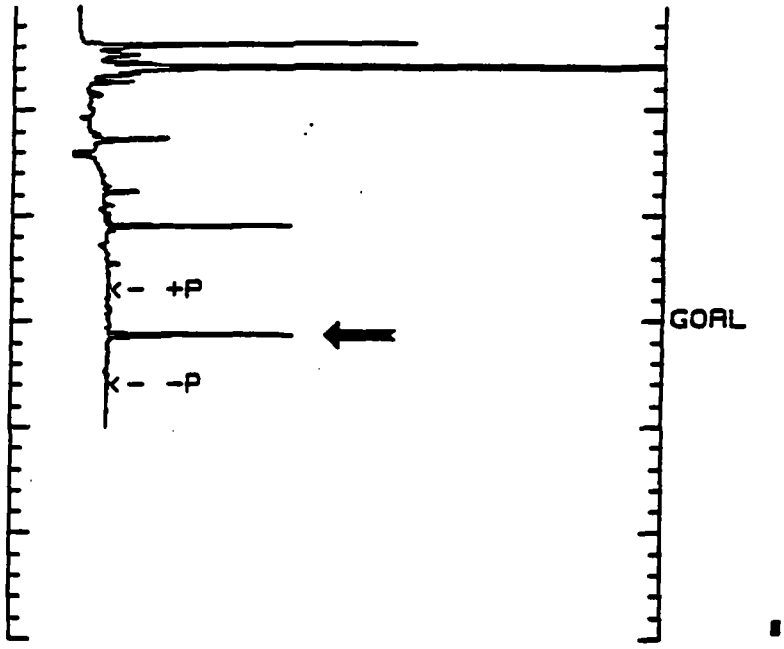
Figure 59. Peanut Vine Fortification 0.10 PPM 93-0142-001

Data file: T11S15 RAR Number: 93-0142
Method file: GOALVn Sample No: 001
Type: FORTIFICATION Component: VINE

Sample Name: NA Cal. Curve: 05/15/95
Date: 15 May 1995 16:10 Method: GOALVn Analyst : JS
Interface: 707 Cycle#: 15 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.62	GOAL	2.5608e+02	.2128e+05	10.0	5.00	0.500

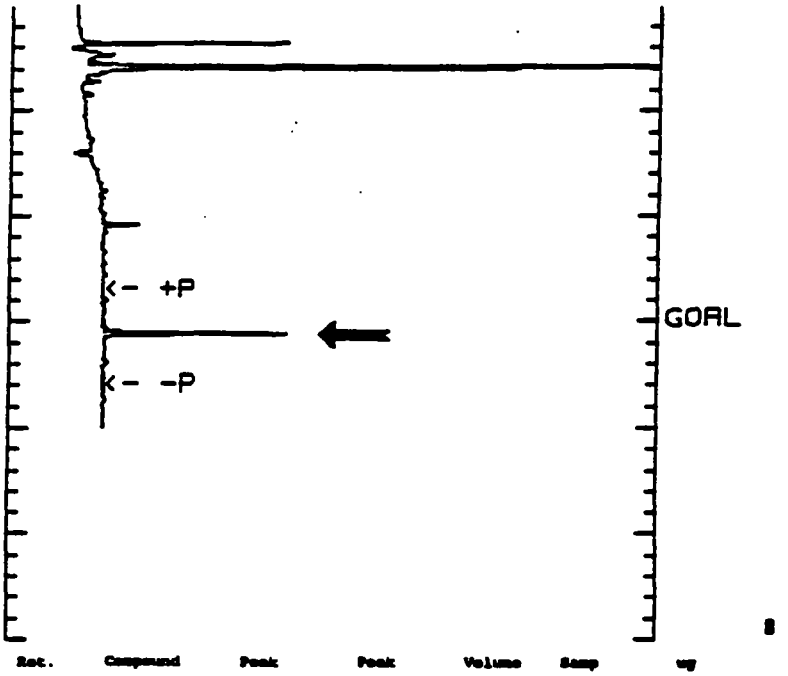
Figure 60. Peanut Vine Fortification 0.50 PPM 93-0142-001

Data file: Y11818 RAR number: 93-0142
Method file: GOALVn Sample No: 001
Type: FORTIFICATION Component: VINE

Sample Name: NA Cal. Curve: 05/15/95
Date: 15 May 1995 18:00 Method: GOALVn Analyst : JB
Interface: 707 Cycle#: 18 Channel#: A

Instrument: VARIAN 3500.
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (S or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min. Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 750 millivolts (70 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
15.62	GOAL	2.6708e+02	.2125e+05	50.0	5.00	2.50

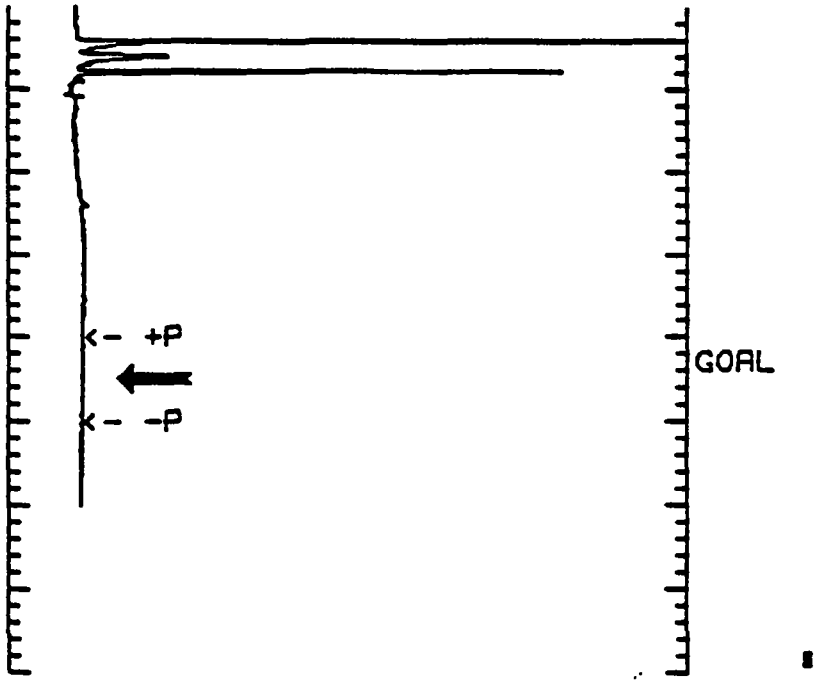
Figure 61. Grape Control (Rtx-50 confirmatory)

Data file: N155A8 RAR number: 93-0012
Method file: GOAL50Vm Sample No: 001 GRAPE
Type: SAMPLE Component: FRUIT

Sample Name: NA Cal. Curve: 11/18/94
Date: 19 Nov 1994 05:25 Method: GOAL50Vm Analyst : SZ
Interface: 707 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
22.50	GOAL	0.000E+00	.000E+00	5.00	5.00

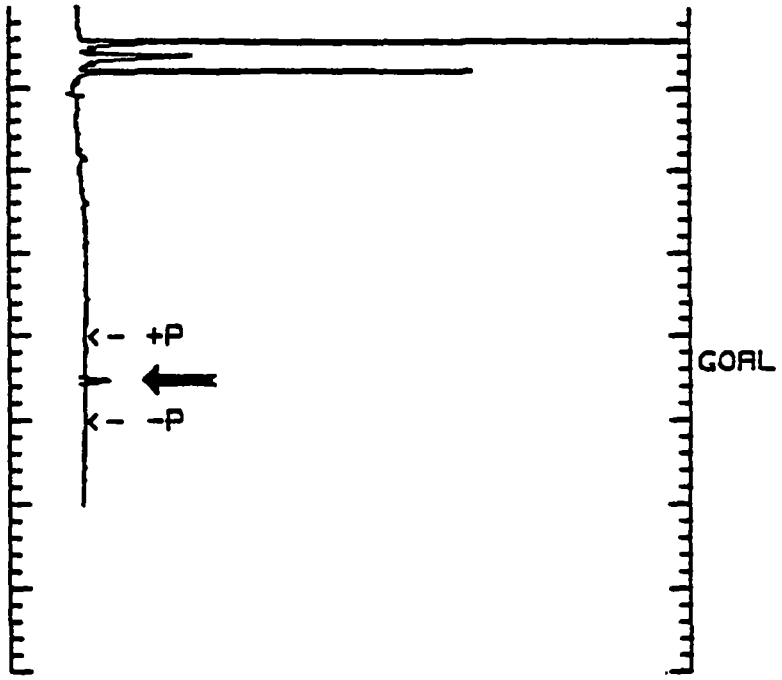
Figure 62. Grape Fortification 0.01 PPM (Rtx-50 confirmatory)

Data file: B155A11 RAR number: 93-0012
Method file: GOAL50Vm Sample No: 001 SPK.5
Type: FORTIFICATION Component: FRUIT

Sample Name: NA Cal. Curve: 11/18/94
Date: 19 Nov 1994 05:13 Method: GOAL50Vm Analyst : SZ
Interface: 707 Cycle#: 11 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 230 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.1 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
22.54	GOAL	2.760E+00	.139E+03	5.00	5.00	0.050

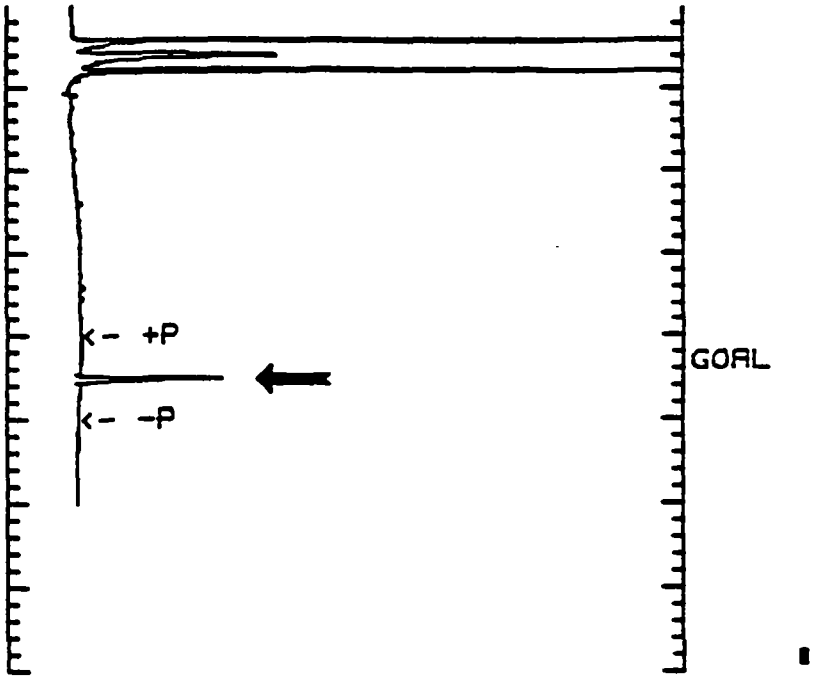
Figure 63. Grape Fortification 0.10 PPM (Rtx-50 confirmatory)

Data file: N15SA12 RAR number: 93-0012
Method file: GOALSOVm Sample No: 001 SPK-C
Type: FORTIFICATION Component: FRUIT

Sample Name: NA Cal. Curve: 11/18/94
Date: 19 Nov 1994 05:50 Method: GOALSOVm Analyst : SZ SZ
Interface: 707 Cycle#: 12 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
22.54	GOAL	1.770E+01	.844E+03	5.00	5.00	0.500

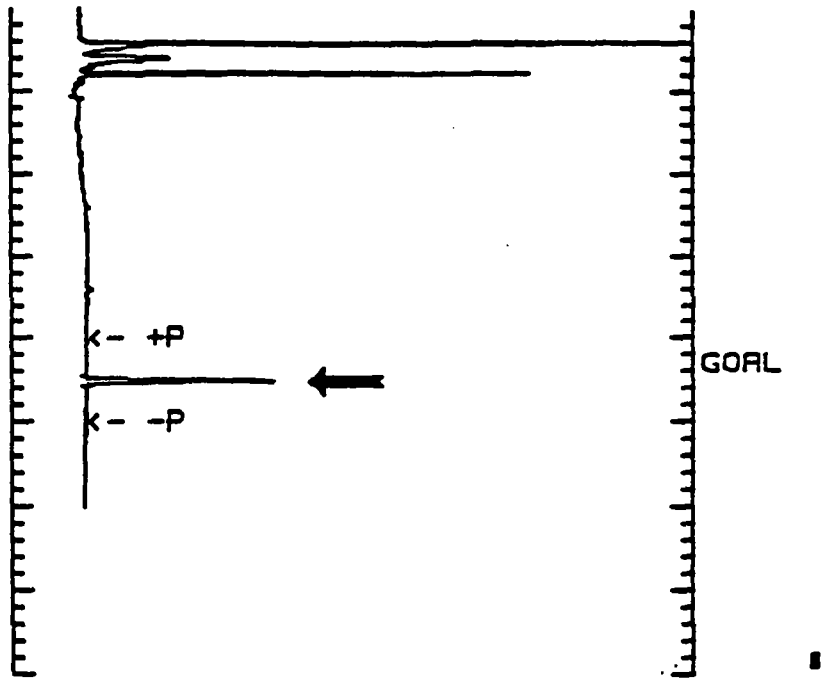
Figure 64. Grape Fortification 0.50 PPM (Rtx-50 confirmatory)

Data file: N15SA14 RAR number: 93-0012
Method file: GOAL50Vm Sample No: 001 PKE
Type: FORTIFICATION Component: FRUIT

Sample Name: NA Cal. Curve: 11/18/94
Date: 19 Nov 1994 07:03 Method: GOAL50Vm Analyst : SZ SZ
Interface: 707 Cycle#: 14 Channel#: A

Instrument: VARIAN 3300, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265 C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.1 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
22.53	GOAL	2.300E+01	.110E+06	25.0	5.00	2.50

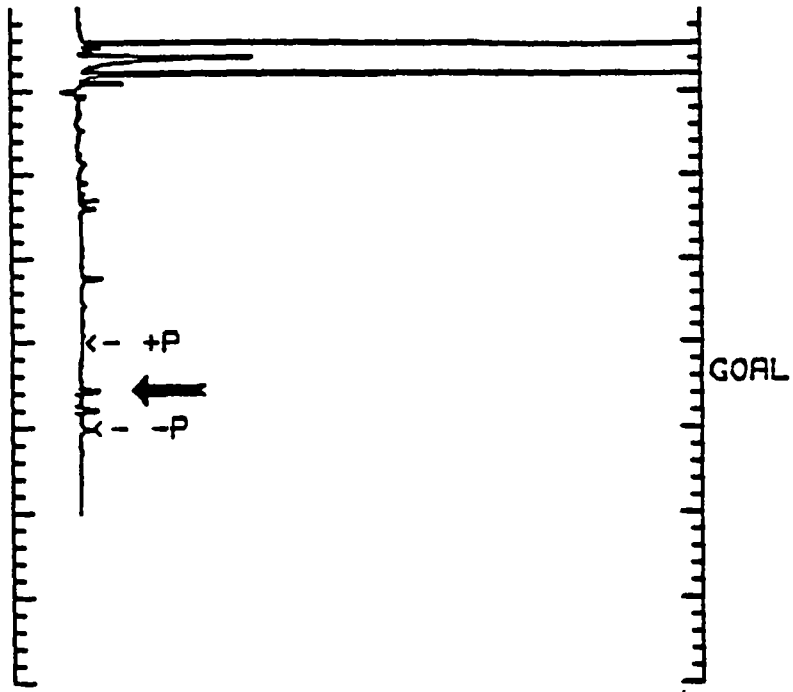
Figure 65. Soybean Control (Rtx-50 confirmatory)

Data file: H0859 RAR number: 92-0107
Method file: GOALSOVm Sample No: 014
Type: SAMPLE Component: SOYBEAN

Sample Name: NA Cal. Curve: 11/21/94
Date: 21 Nov 1994 16:54 Method: GOALSOVm Analyst : SZ ϕ Z
Interface: 707 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
22.78	GOAL	1.730E+00	.955E+02	5.00	5.00

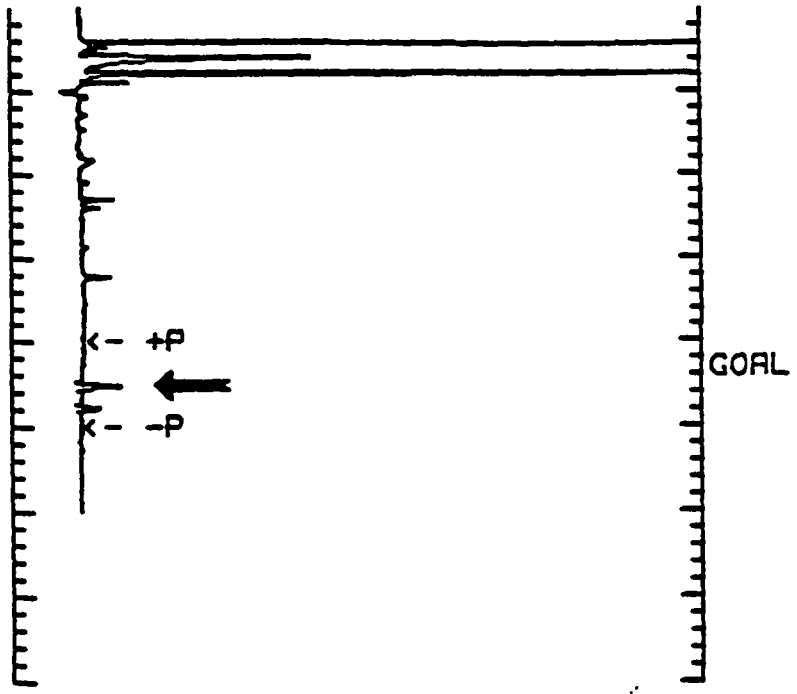
Figure 66. Soybean Fortification 0.01 PPM (Rtx-50 confirmatory)

Data file: M08511 RAR number: 92-0107
Method file: GOALSOVm Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: NA Cal. Curve: 11/21/94
Date: 21 Nov 1994 18:08 Method: GOALSOVm Analyst : SZ
Interface: 707 Cycle#: 11 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 230 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C.
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
22.60	GOAL	4.420E+00	.220E+03	5.00	5.00	0.050

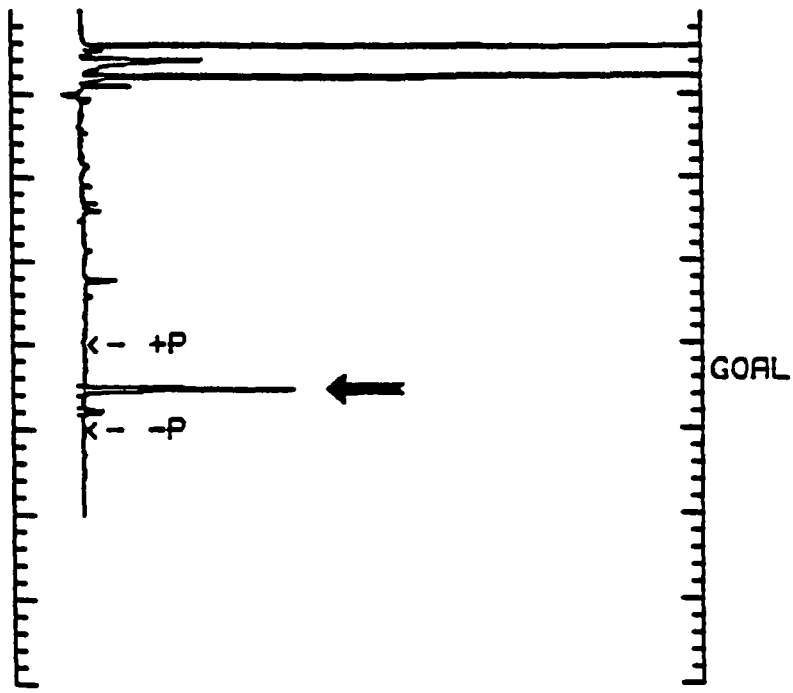
Figure 67. Soybean Fortification 0.10 PPM (Rtx-50 confirmatory)

Data file: M08S13 RAR number: 92-0107
Method file: GOALSOVm Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: NA Cal. Curve: 11/21/94
Date: 21 Nov 1994 19:21 Method: GOALSOVm Analyst : SZ
Interface: 707 Cycle#: 13 Channel#: A

Instrument: VARIAN 3300, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.1 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
22.60	GOAL	2.580E+01	.121E+04	5.00	5.00	0.500

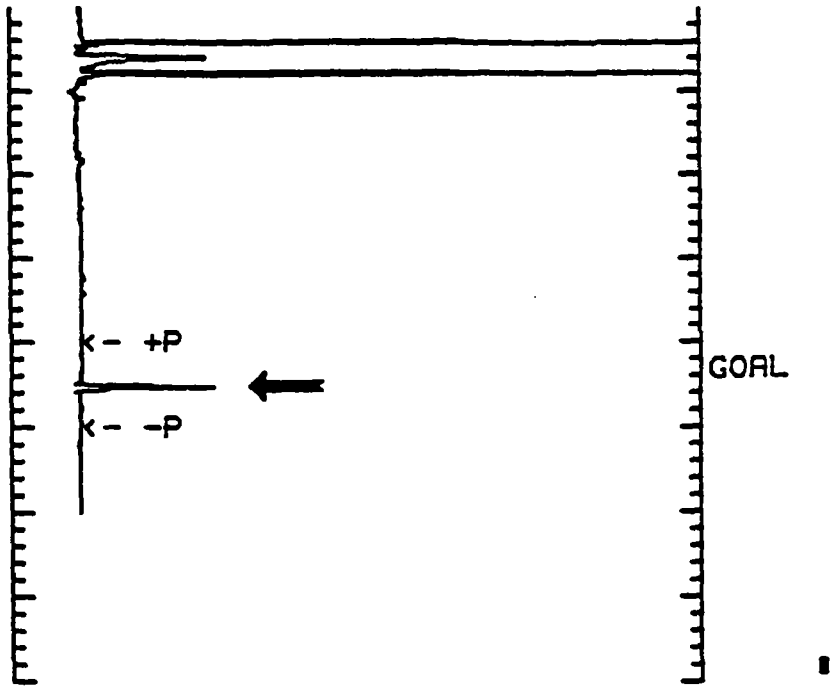
Figure 68. Soybean Fortification 0.50 PPM (Rtx-50 confirmatory)

Data file: M08S14 RAR number: 92-0107
Method file: GOALSOVm Sample No: 014
Type: FORTIFICATION Component: SOYBEAN

Sample Name: NA Cal. Curve: 11/21/94
Date: 21 Nov 1994 19:58 Method: GOALSOVm Analyst : SZ
Interface: 707 Cycle#: 14 Channel#: A

Instrument: VARIAN 3500, GC 14
Column: RTX-50, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 5C End Time-Temp (deg-min): 250 C-25
Prog Slope (# or Linear): NA Inj Port Temp: 265C
Flowrate/Gas: 3.5ML/MIN Split Ratio: NA
Det 1-Type & Temp: ECD/270C Det 2-Type & Temp: NA

Plot times: 0 to 40 minutes
Plot range: 40 millivolts (-1.1 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (mL)	Samp Wt.	ug Added
22.59	GOAL	1.620E+01	.785E+03	50.0	5.00	2.50

Figure 69. Standard 0.005 µg/ml (GC/ECD)

Data file: L0784 Type: STANDARD

Sample Name: NA Cal. Curve: 07/07/95

Date: 7 Jul 1995 17:05 Method: GOALVs Analyst : SS SE

Interface: 711 Cycle0: 4 Channel0: A

Instrument: VARIAN 3800,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

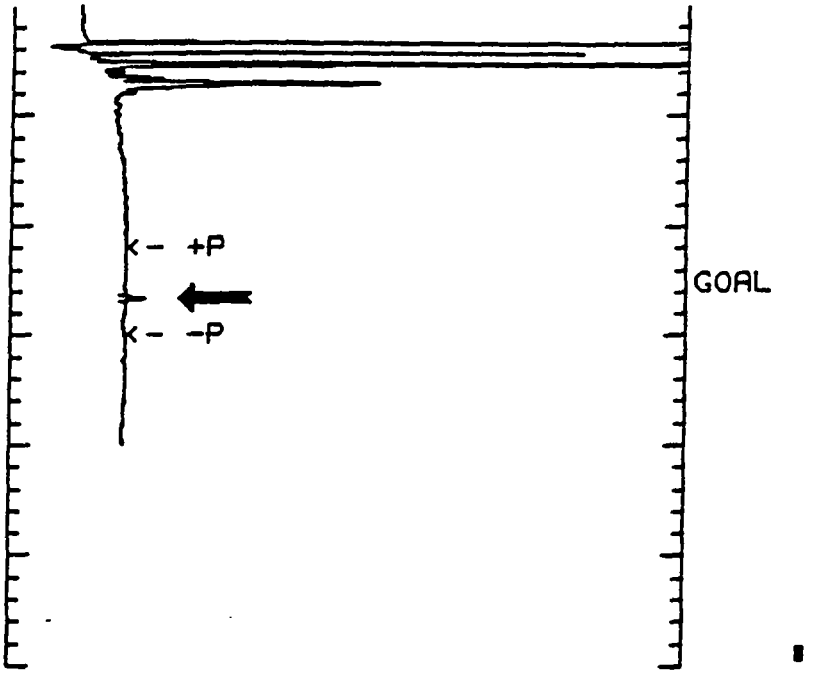
Prog Slope (0 or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE · Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.24 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.34	GOAL	0.005	1.620E+00	1.320E+02

Figure 70. Standard 0.01 µg/ml (GC/ECD)

Data file: L0783 Type: STANDARD

Sample Name: NA Cal. Curve: 07/07/95

Date: 7 Jul 1995 16:48 Method: GOALVa Analyst : AS

Interface: 711 Cycle#: 3 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

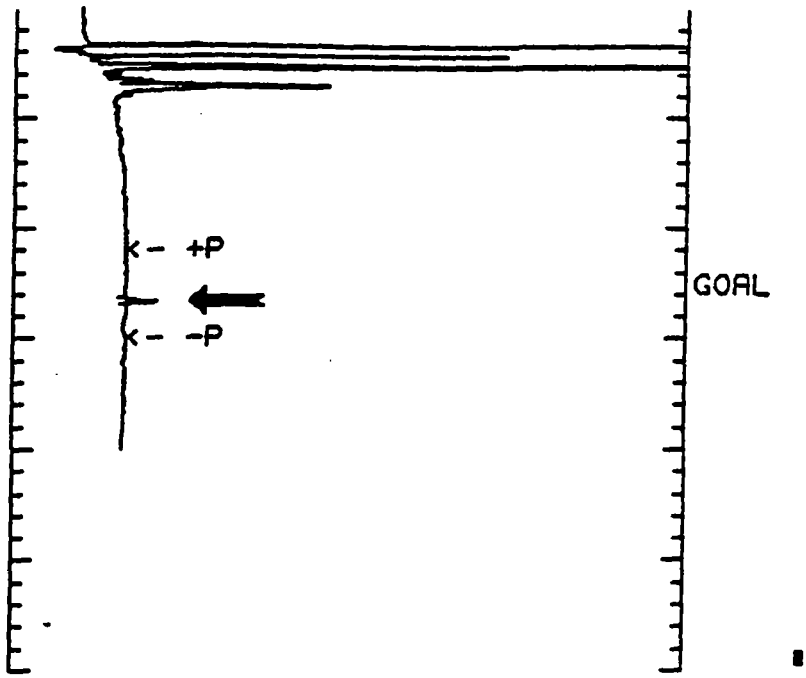
Prog Slope (S or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.6ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes

Plot range: 50 millivolts (-.34 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.34	GOAL	0.010	2.830E+00	2.300E+02

Figure 71. Standard 0.02 µg/ml (GC/ECD)

Data file: L07811 Type: STANDARD

Sample Name: NA Cal. Curve: 07/07/95

Date: 7 Jul 1995 20:02 Method: GOALVa Analyst : SS

Interface: 711 Cycle0: 11 Channel0: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

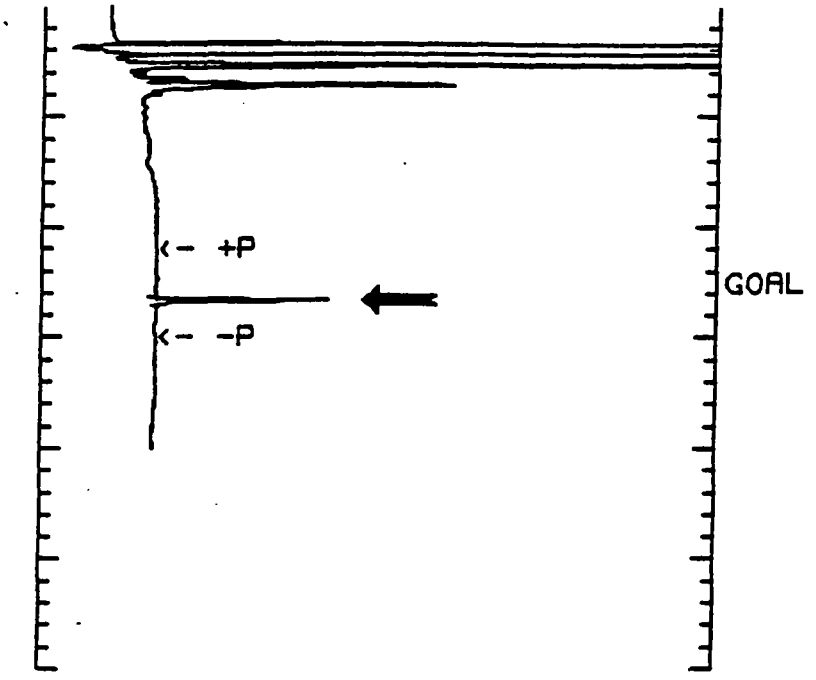
Prog Slope (0 or Linear): NA Inj Port Temp: 255

Flowrate/Gas: 3.5ml/minHE · Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes

Plot range: 50 millivolts (-.25 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.32	GOAL	0.050	1.550E+01	1.260E+03

Figure 72. Standard 0.10 µg/ml (GC/ECD)

Data file: L0782 Type: STANDARD

Sample Name: NA Cal. Curve: 07/07/95

Date: 7 Jul 1995 16:15 Method: GOAL7a Analyst : SS SZ

Interface: 711 Cycle#: 2 Channel#: A

Instrument: VARIAN 3800,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

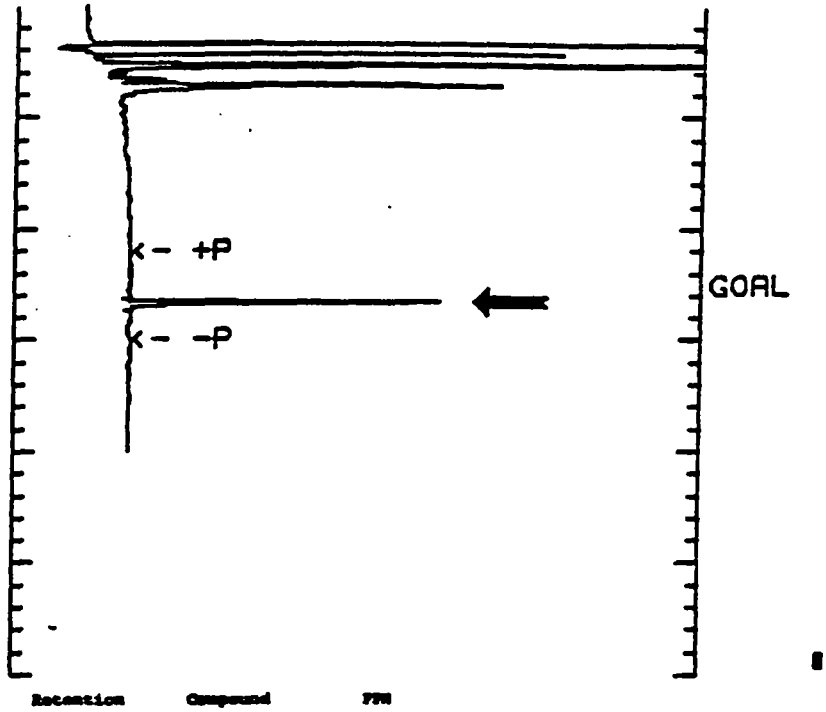
Prog Slope (S or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/min He Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.26 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.34	GOAL	0.100	2.810E+01	2.250E+03

Figure 73. Standard 0.15 µg/ml (GC/ECD)

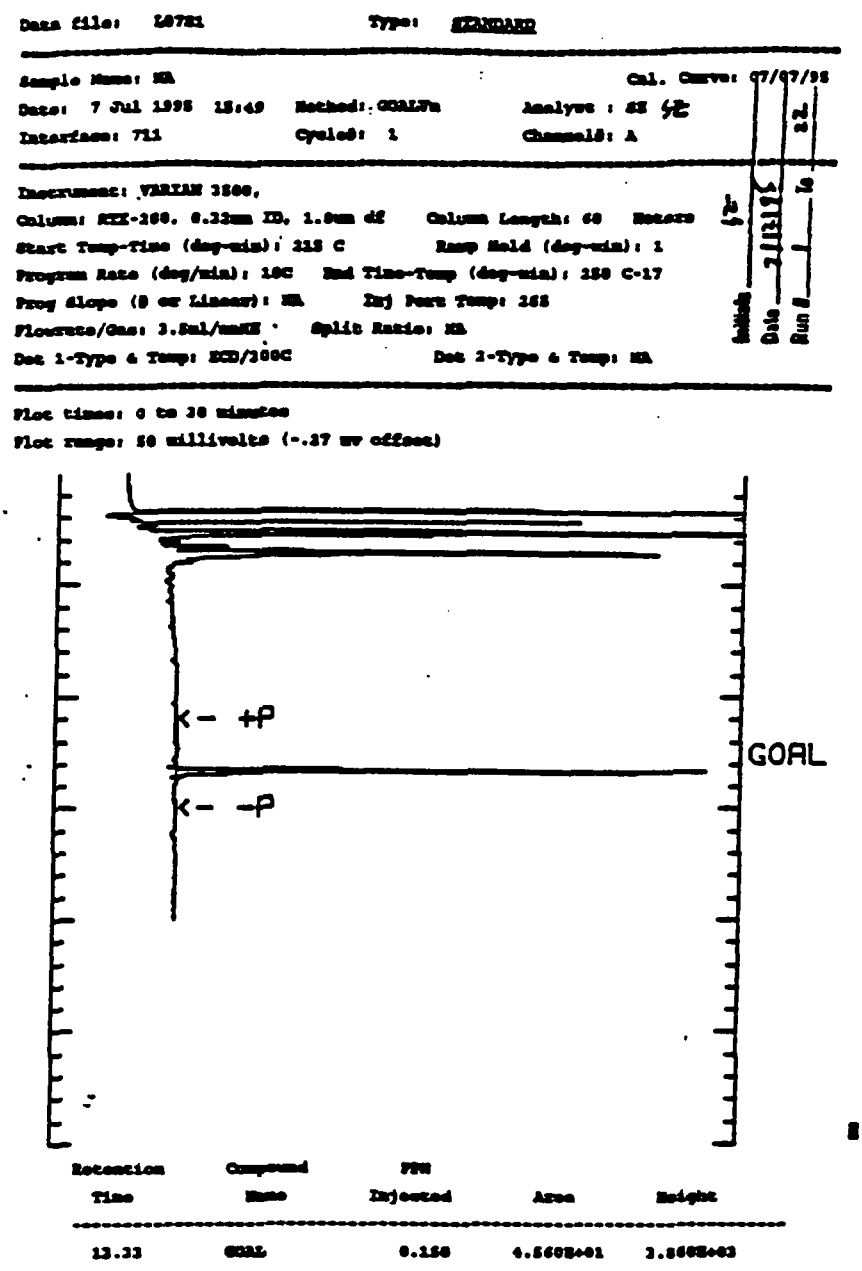
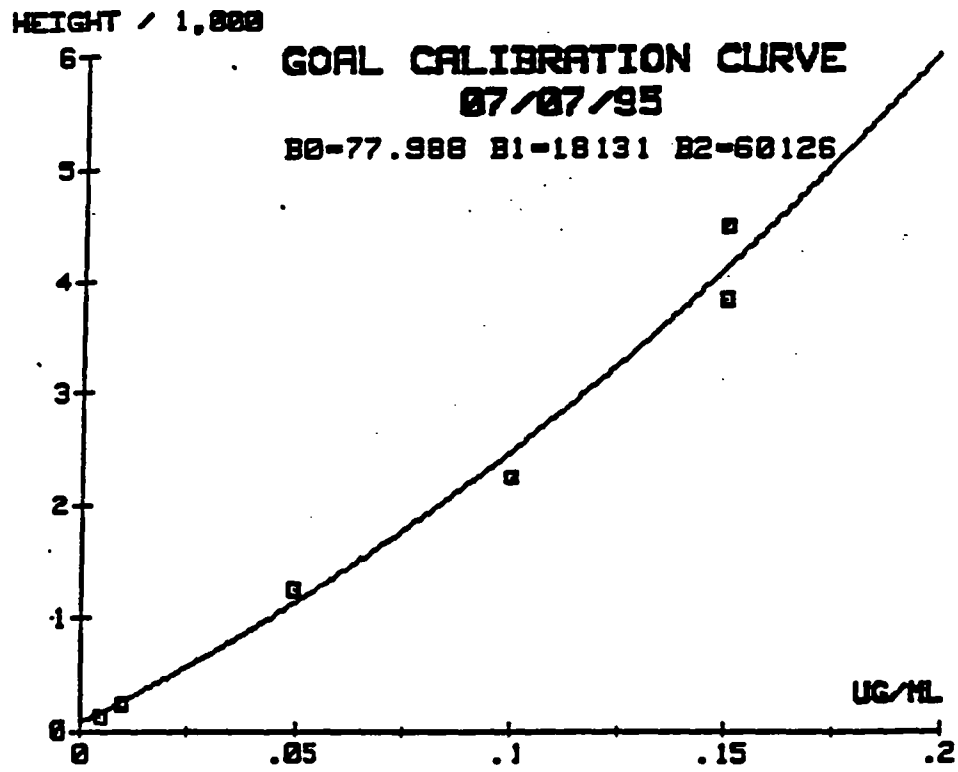


Figure 74. Oxyfluorfen Calibration Curve (GC/ECD)

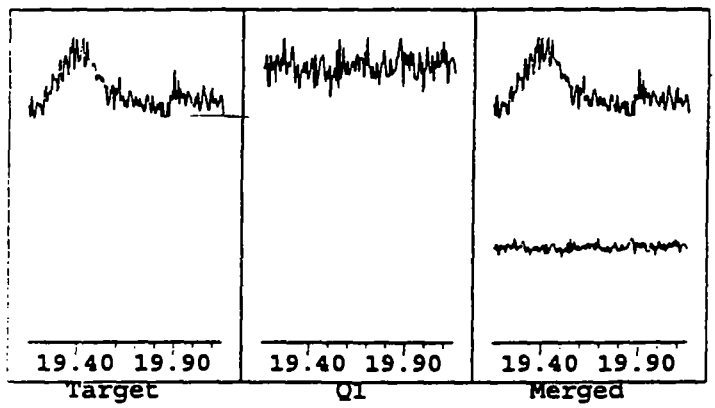


Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data.

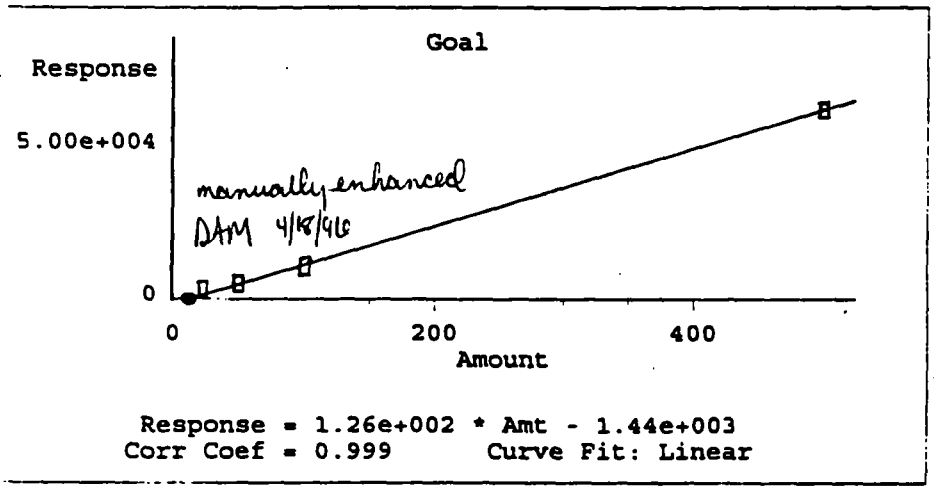
Figure 75. Grape Control (GC/MS)

File : C:\HPCHEM\1\DATA\N08S9.D
Operator : sz
Acquired : 9 Nov 95 10:41 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 9
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.37
Concentration: 12.33
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.37	19.16	114	sys def
Q1	361.00	0.0	0.0- 22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

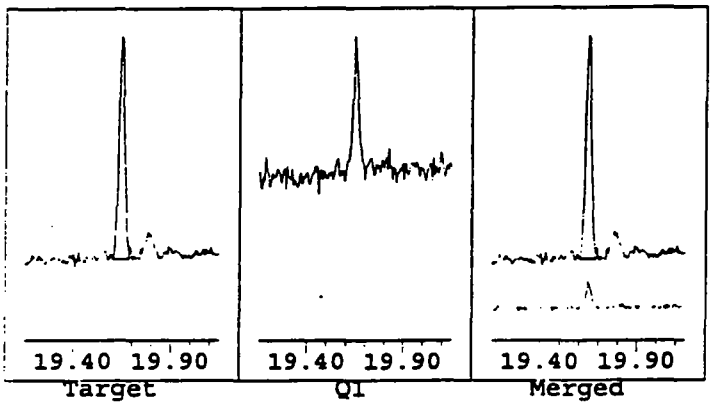


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

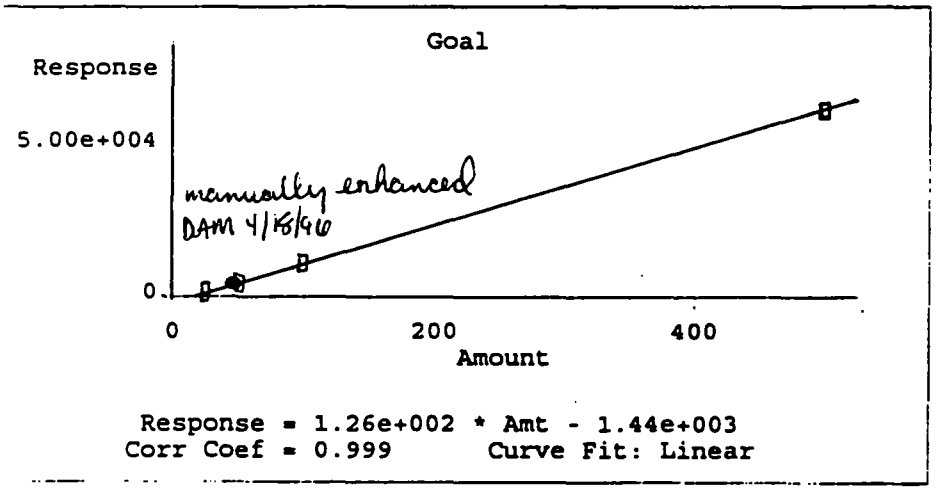
Figure 76. Grape Fortification 0.025 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N08S10.D
Operator : sz
Acquired : 9 Nov 95 11:19 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 10
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 46.28
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	4397	sys def
Q1	361.00	13.5	0.0-	22.0	19.64	to	592	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

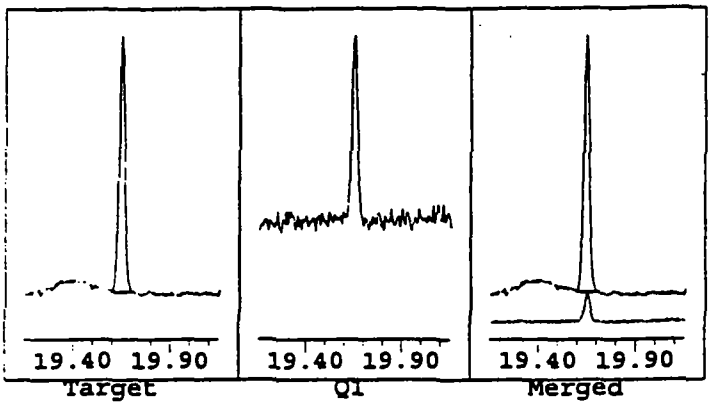


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

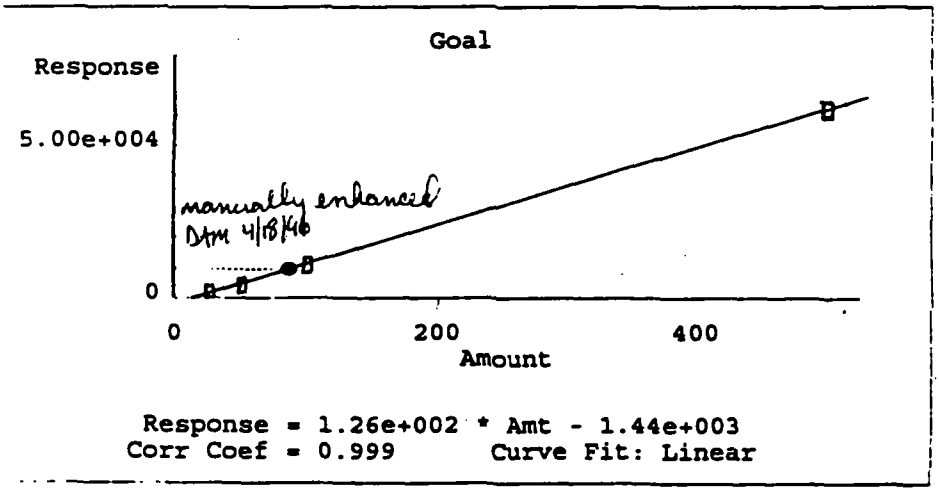
Figure 77. Grape Fortification 0.05 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N08S11.D
Operator : sz
Acquired : 9 Nov 95 11:57 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 11
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 85.98
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	9405	sys def
Q1	361.00	11.5	0.0-	22.0	19.65	to	1084	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

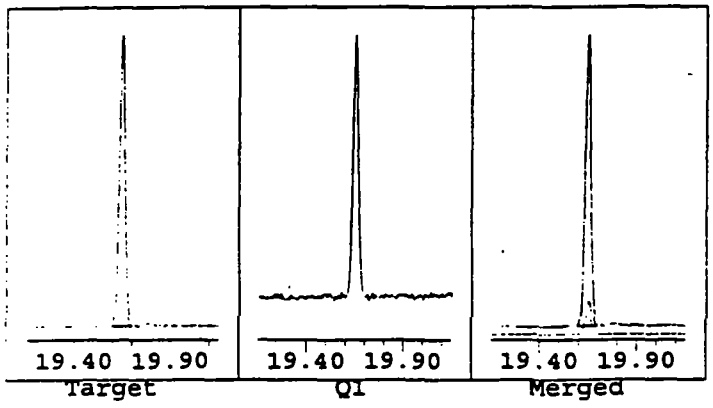


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

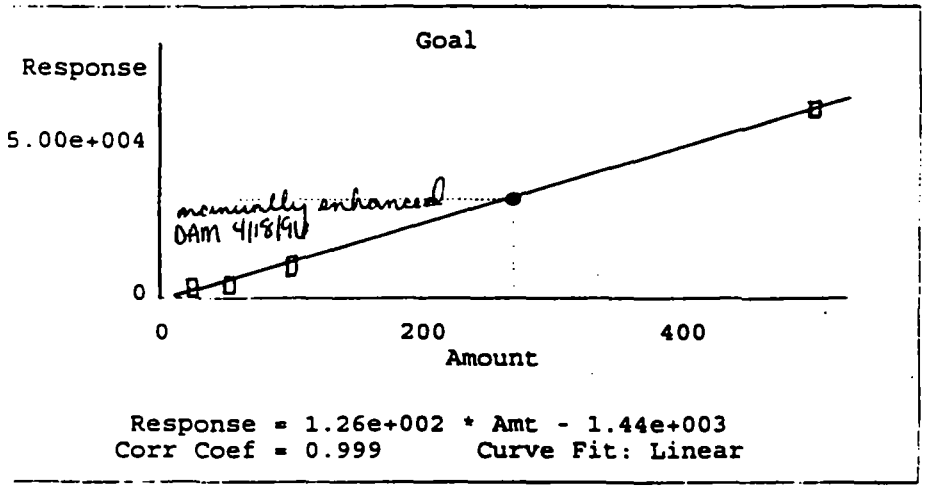
Figure 78. Grape Fortification 0.125PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N08S12.D
Operator : sz
Acquired : 10 Nov 95 12:35 am using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 12
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 269.02
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.16	32497	sys def
Q1	361.00	11.4	0.0-	22.0	19.65	to	3689	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

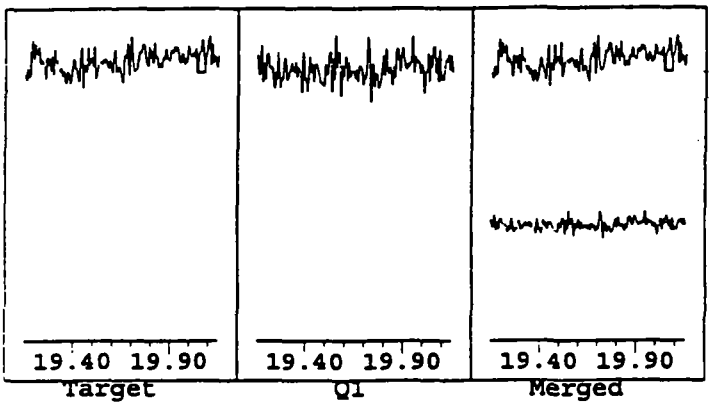


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

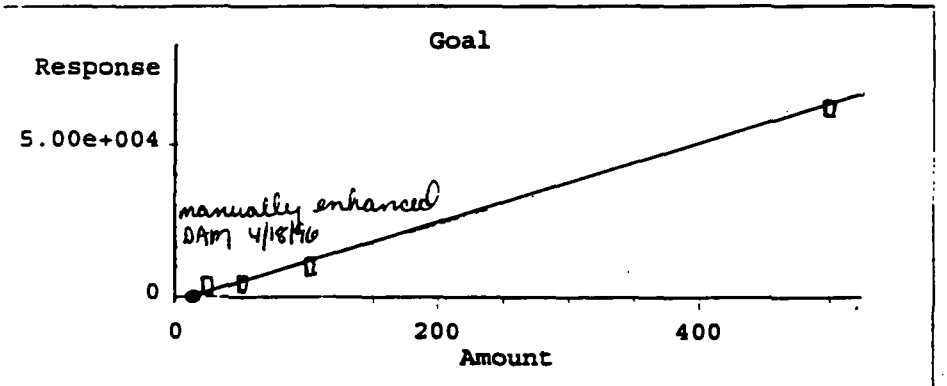
Figure 79. Peach Control (GC/MS)

File : C:\HPCHEM\1\DATA\N08S5.D
Operator : sz
Acquired : 9 Nov 95 8:08 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 5
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 20.06
Concentration: 12.30
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		20.06	19.16	110	sys def
Q1	361.00	0.0	0.0- 22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

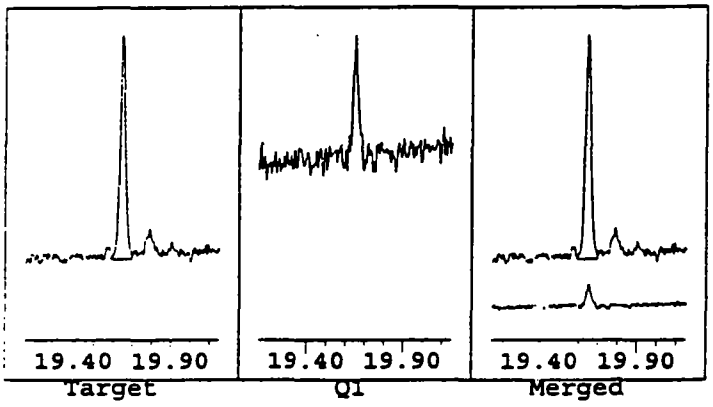


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

Figure 80. Peach Fortification 0.025 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N08S6.D
Operator : sz
Acquired : 9 Nov 95 8:46 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 6
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 42.26
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	3890	sys def
Q1	361.00	10.5	0.0-	22.0	19.65	to	410	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

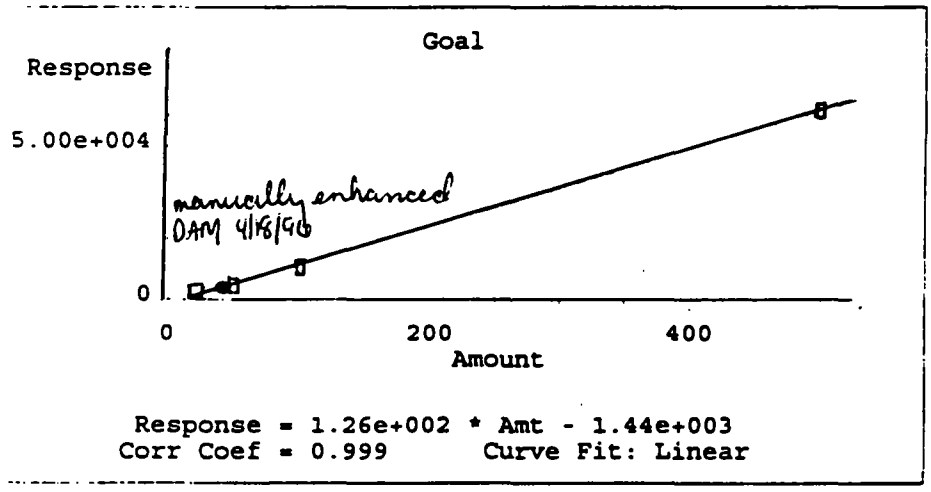
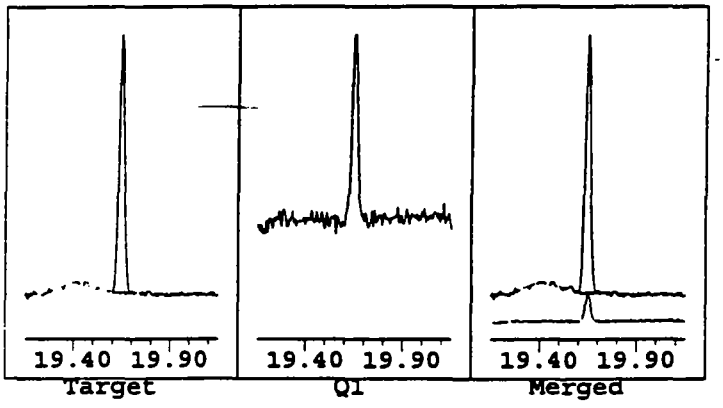


Figure 81. Peach Fortification 0.05 PPM (GC/MS)

```
File        : C:\HPCHEM\1\DATA\N08S7.D
Operator    : sz
Acquired    :  9 Nov 95   9:24 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info   : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 7
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

     Compound:        Goal
     Ret Time:        19.64
     Concentration:   82.39
     Pk # and Type:   1      RTEINT used
```



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	8952	sys def
Q1	361.00	12.4	0.0-	22.0	19.65	to	1110	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

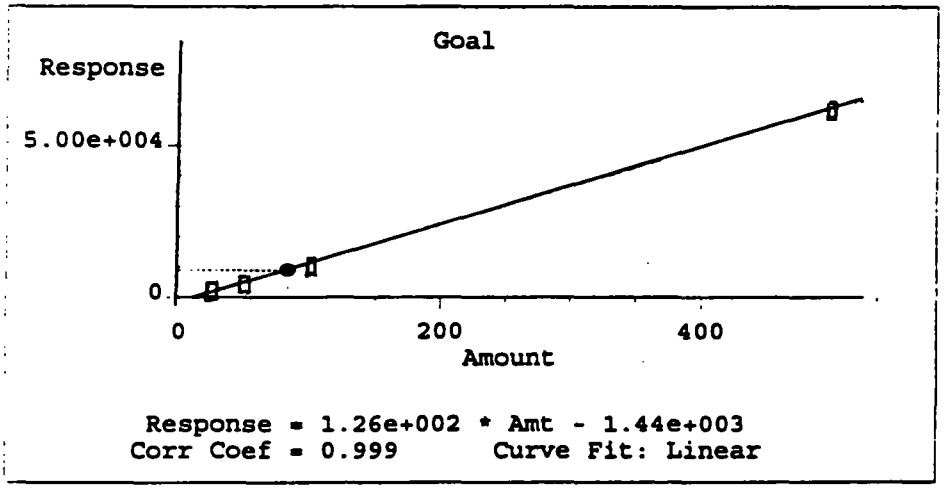
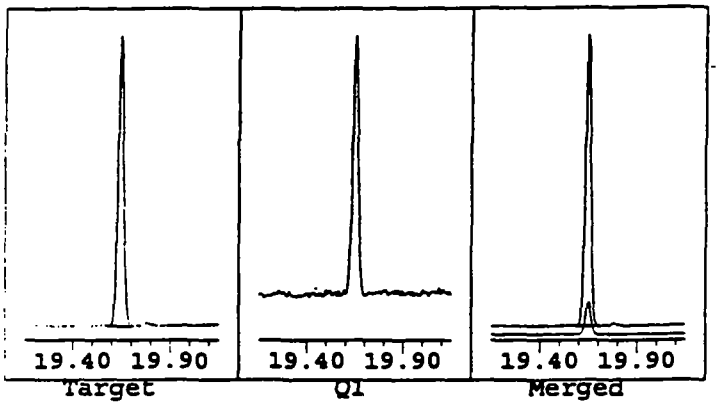


Figure 82. Peach Fortification 0.125 PPM (GC/MS)

```
File         : C:\HPCHEM\1\DATA\N08S8.D
Operator     : sz
Acquired     :  9 Nov 95  10:03 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info    : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 8
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

     Compound:        Goal
     Ret Time:        19.64
     Concentration:   255.18
     Pk # and Type:   1      RTEINT used
```



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	30752	sys def
Q1	361.00	11.3	0.0-	22.0	19.65	to	3485	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

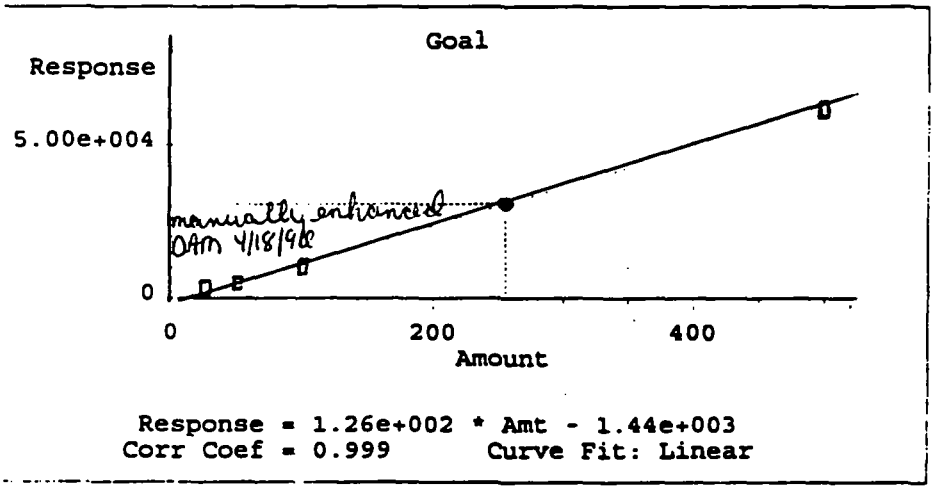
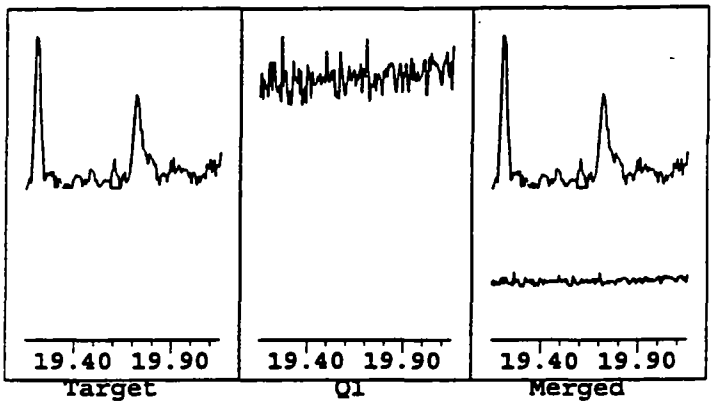


Figure 83. Peanut Nutmeat Control (GC/MS)

File : C:\HPCHEM\1\DATA\N09S3.D
Operator : sz
Acquired : 10 Nov 95 4:39 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 nutmeat
Misc Info : blk SW=5g, FV=2.5ml
Vial Number: 3
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.60
Concentration: 27.22
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.60	19.16	197	sys def
Q1	361.00	0.0	0.0-	22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

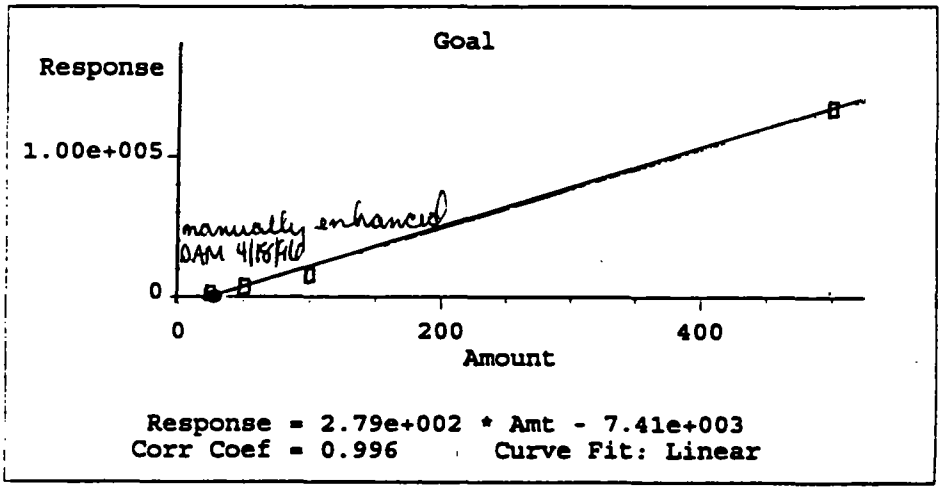
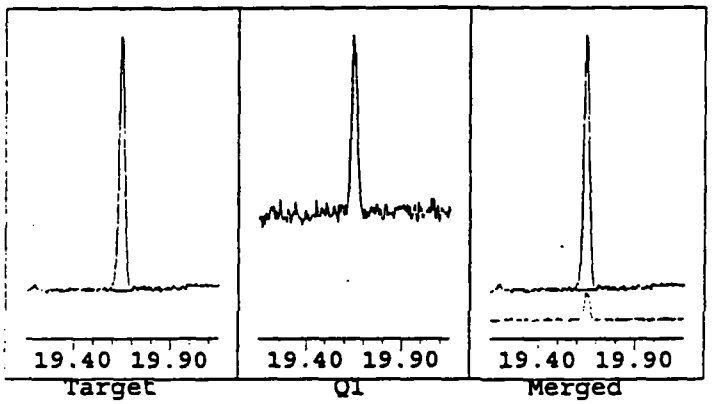


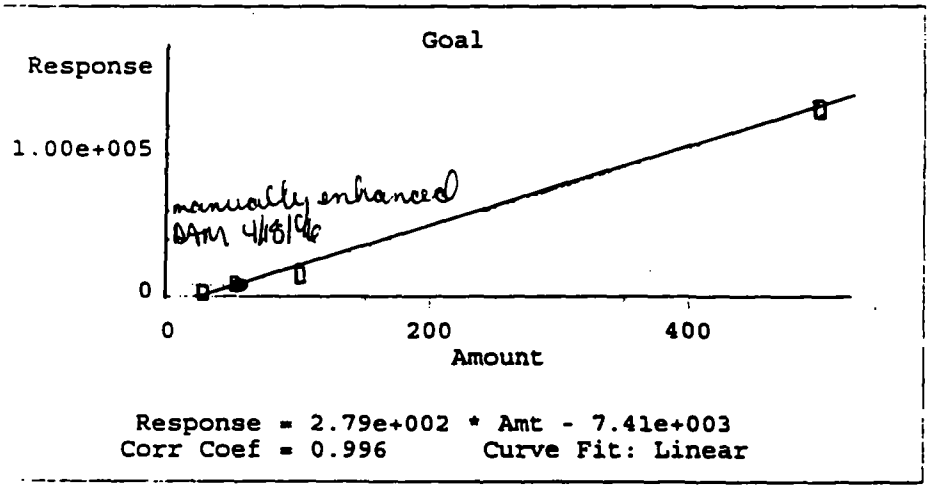
Figure 84. Peanut Nutmeat Fortification 0.025 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S7.D
Operator : sz
Acquired : 10 Nov 95 7:12 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 nutmeat
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 7
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 54.38
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	7784	sys def
Q1	361.00	11.5	0.0-	22.0	19.64	to	899	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

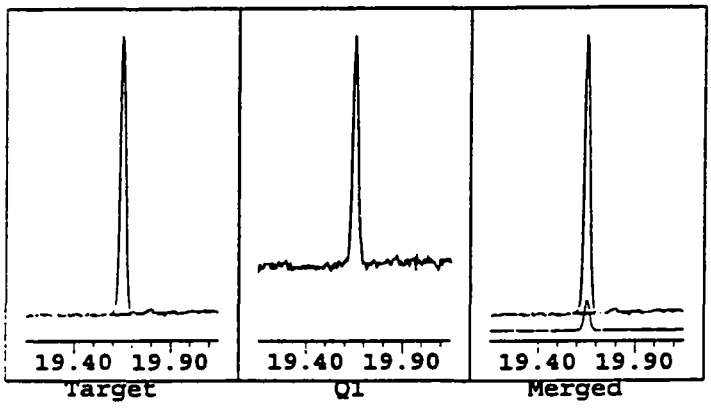


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

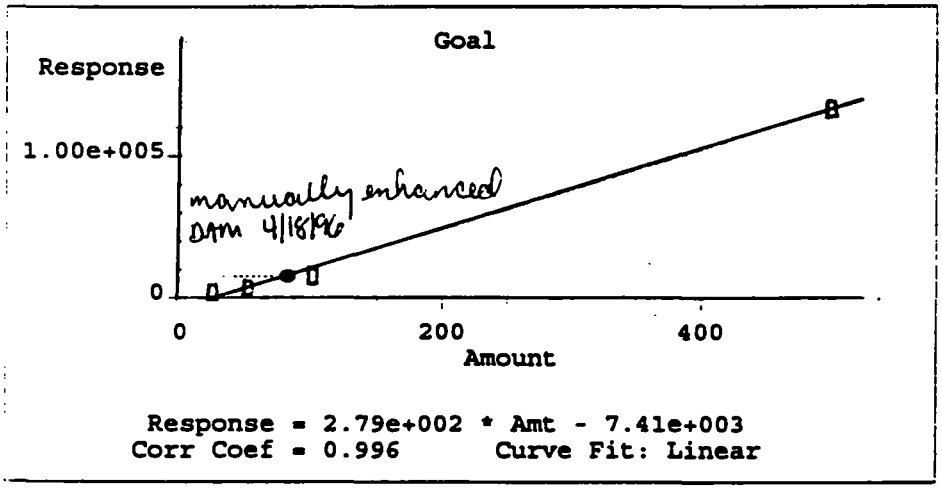
Figure 85. Peanut Nutmeat Fortification 0.05 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S11.D
Operator : sz
Acquired : 10 Nov 95 9:44 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 nutmeat
Misc Info : spkb SW=5 FV=2.5 0.05ppm
Vial Number: 11
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 80.70
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	15137	sys def
Q1	361.00	11.2	0.0-	22.0	19.65	to	1700	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

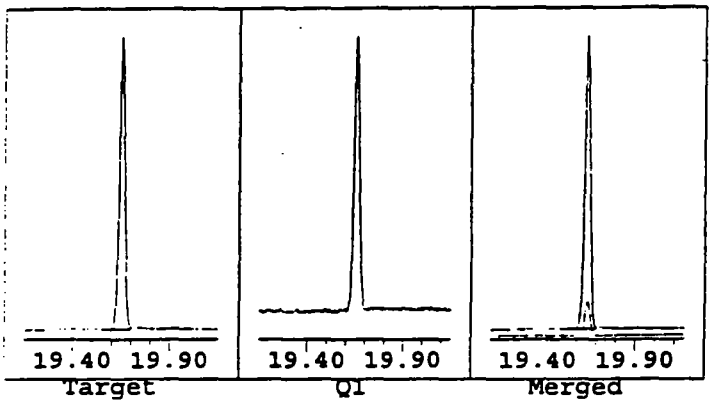


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

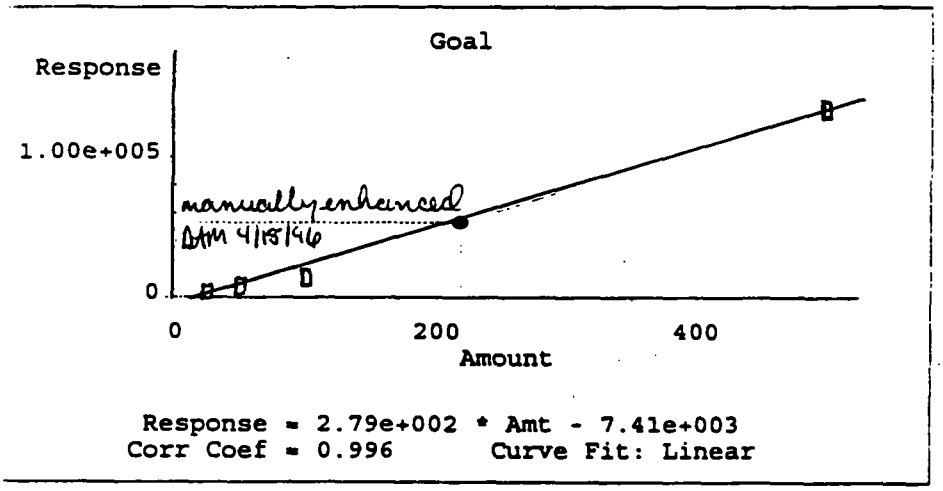
Figure 86. Peanut Nutmeat Fortification 0.125 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S13.D
Operator : sz
Acquired : 10 Nov 95 11:01 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 nutmeat
Misc Info : spkb SW=5 FV=2.5 0.125ppm
Vial Number: 13
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 217.28
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.16	53290	sys def
Q1	361.00	11.5	0.0-	22.0	19.65	to	6113	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

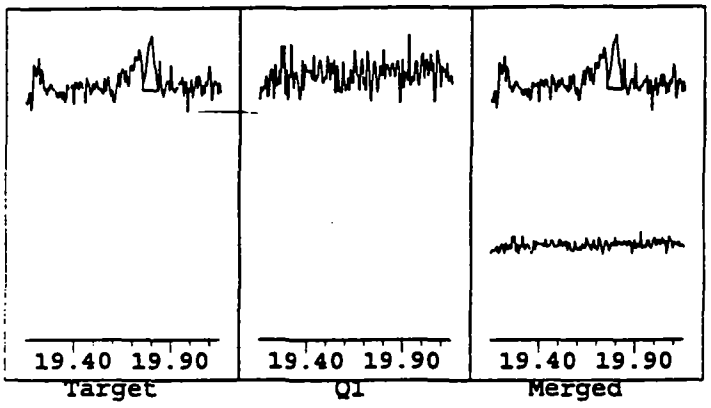


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

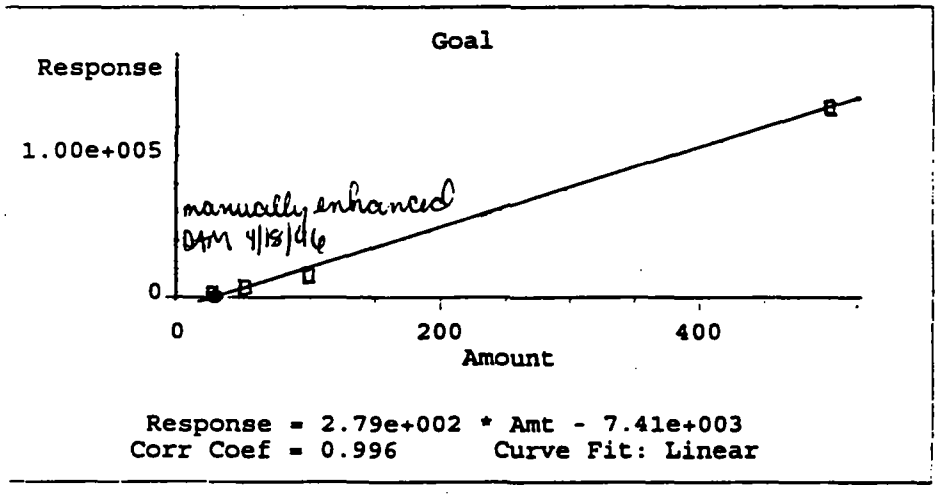
Figure 87. Peanut Shell Control (GC/MS)

```
File        : C:\HPCHEM\1\DATA\N09S4.D
Operator    : sz
Acquired    : 10 Nov 95    5:17 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 shell
Misc Info   : blk SW=5g, FV=2.5ml
Vial Number: 4
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

     Compound:        Goal
     Ret Time:        19.79
     Concentration:   28.01
     Pk # and Type:   1      RTEINT used
     # Qualifiers Not Satisfied
```



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.79	19.16	418	sys def
Q1	361.00	0.0	0.0-	22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

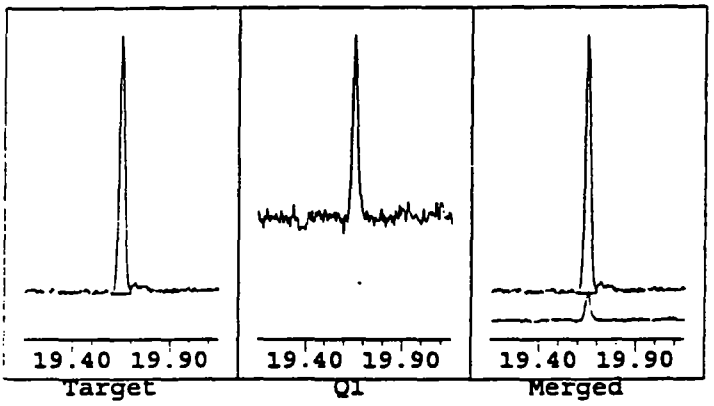


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

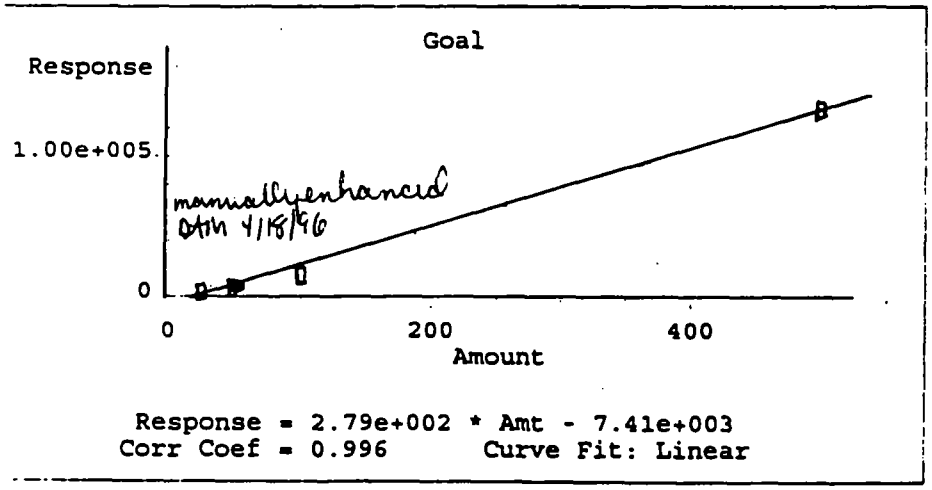
Figure 88. Peanut Shell Fortification 0.025 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S8.D
Operator : sz
Acquired : 10 Nov 95 7:50 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 shell
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 8
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 52.23
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	7183	sys def
Q1	361.00	11.3	0.0-	22.0	19.65	to	814	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

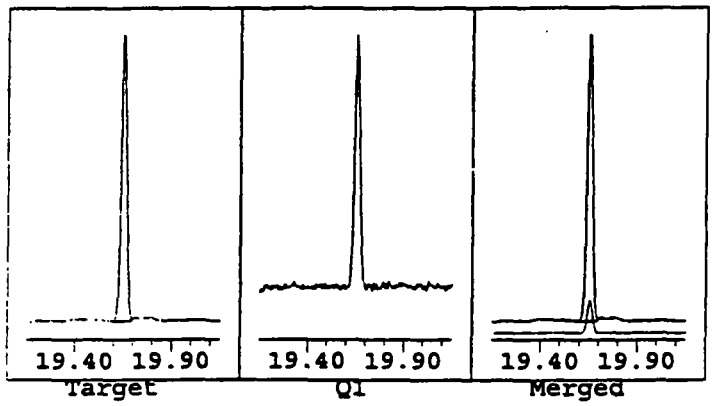


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

Figure 89. Peanut Shell Fortification 0.05 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S12.D
Operator : sz
Acquired : 10 Nov 95 10:23 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 shell
Misc Info : spkb SW=5 FV=2.5 0.05ppm
Vial Number: 12
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 117.28
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	25354	sys def
Q1	361.00	11.3	0.0-	22.0	19.65	to	2876	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

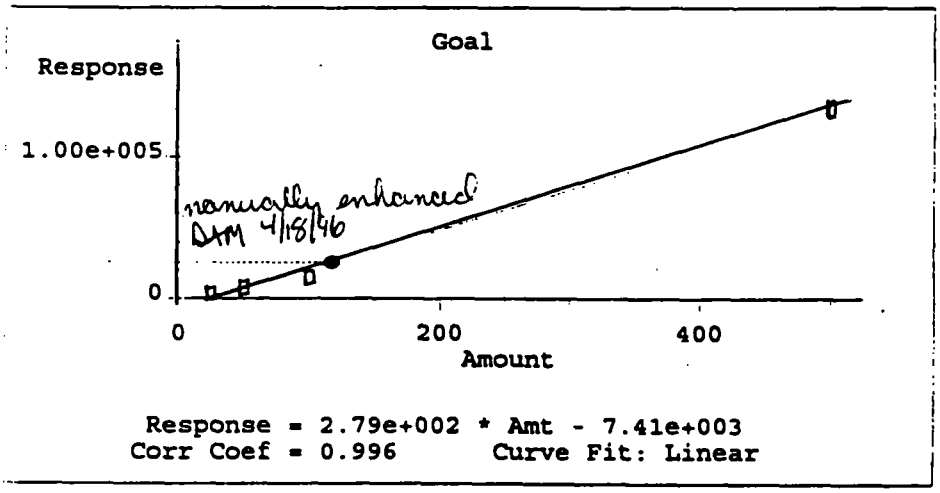
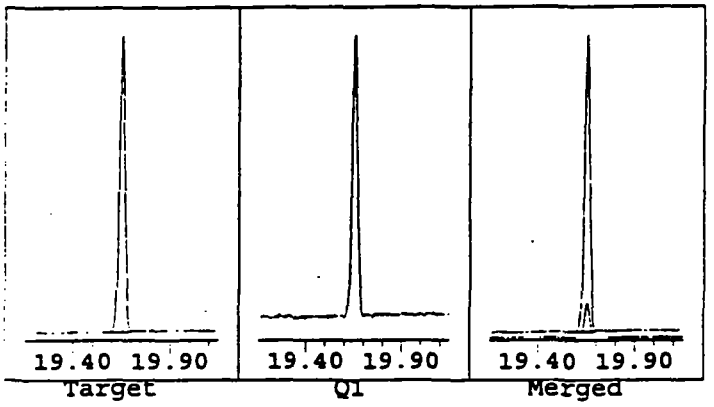


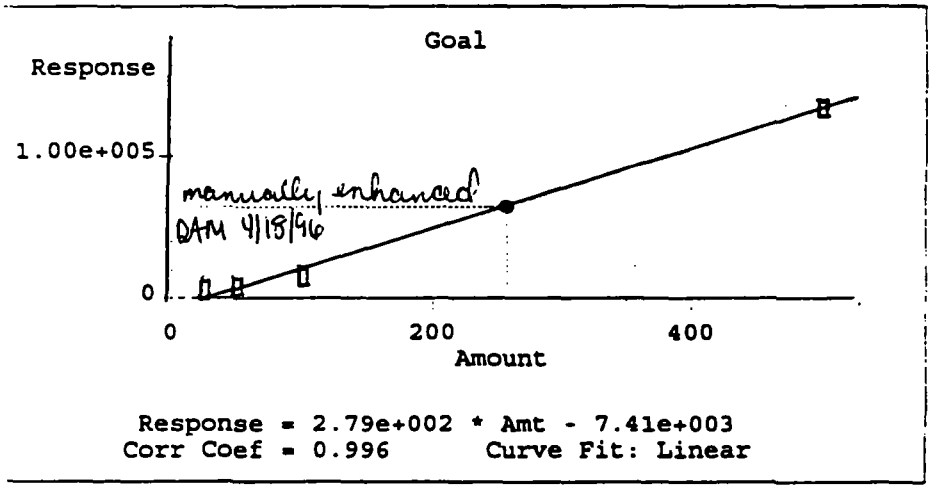
Figure 90. Peanut Shell Fortification 0.125 PPM (GC/MS)

File : C:\HPCHEM\1\DATA\N09S14.D
Operator : sz
Acquired : 10 Nov 95 11:39 pm using AcqMethod GOAL1
Sample Name: 93-0157-004 shell (RE) RB 11/15/95
Misc Info : spk@ SW=5 FV=2.5 0.125ppm
Vial Number: 14 (RE) RB 11/15/95
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 256.92
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.16	64363	sys def
Q1	361.00	11.5	0.0-	22.0	19.66	to	7428	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

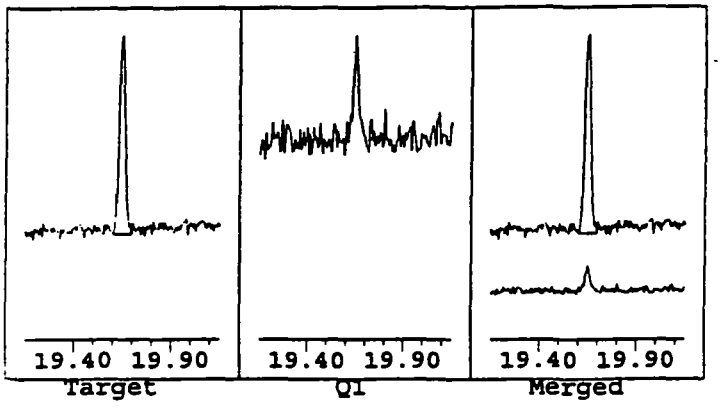


Response = 2.79e+002 * Amt - 7.41e+003
Corr Coef = 0.996 Curve Fit: Linear

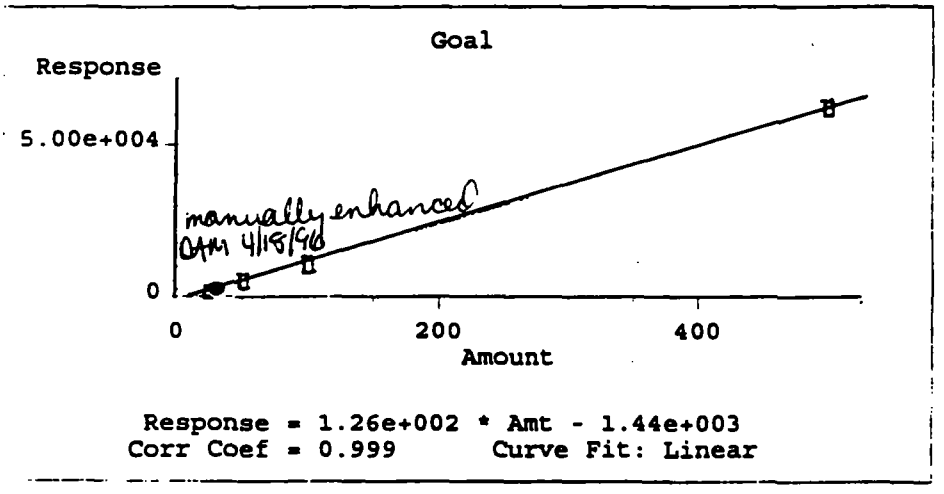
Figure 91. Standard 0.025 µg/ml (GC/MS)

File : C:\HPCHEM\1\DATA\N08S4.D
Operator : sz
Acquired : 9 Nov 95 7:30 pm using AcqMethod GOAL1
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 4
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 30.45
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.16	2400	sys def
Q1	361.00	12.6	0.0-	22.0	19.65	to	303	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

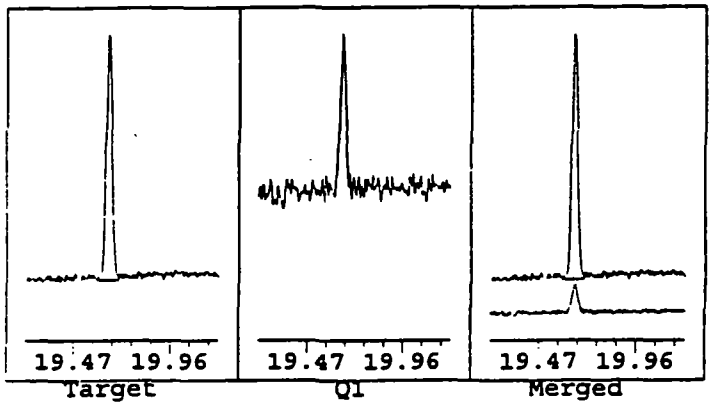


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

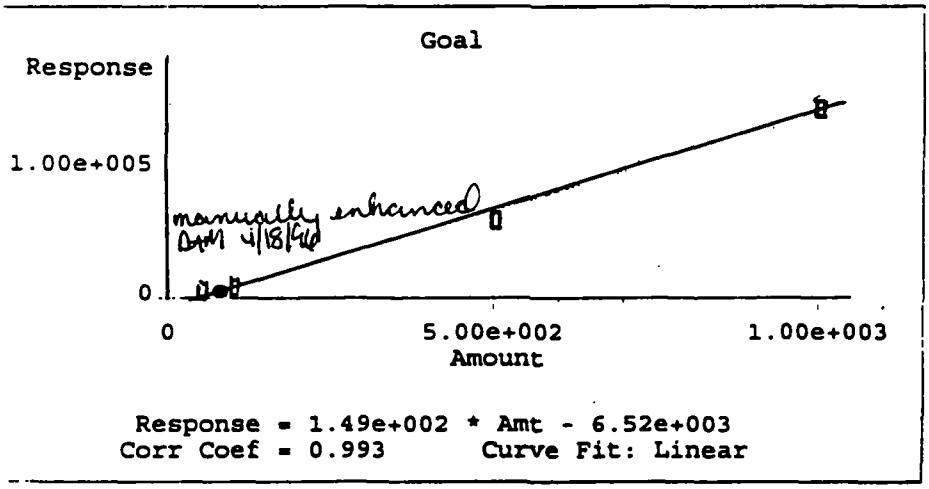
Figure 92. Standard 0.05 µg/ml (GC/MS)

File : C:\HPCHEM\1\DATA\N08S3.D
Operator : sz
Acquired : 9 Nov 95 6:51 pm using AcqMethod GOAL1
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 3
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 79.04
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.22	5254	sys def
Q1	361.00	12.6	0.0-	22.0	19.65	to	662	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

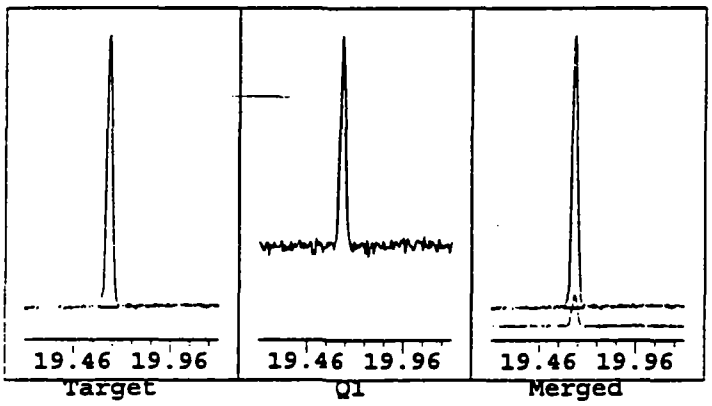


Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

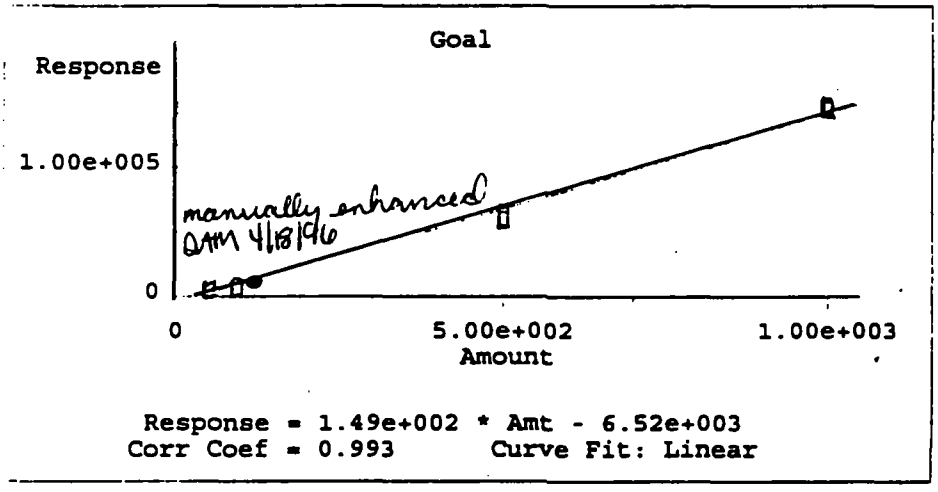
Figure 93. Standard 0.10 µg/ml (GC/MS)

File : C:\HPCHEM\1\DATA\N08S2.D
Operator : sz
Acquired : 9 Nov 95 6:13 pm using AcqMethod GOAL1
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 2
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 119.45
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.65	19.22	11274	sys def
Q1	361.00	11.5	0.0- 22.0	19.65	to	1294	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

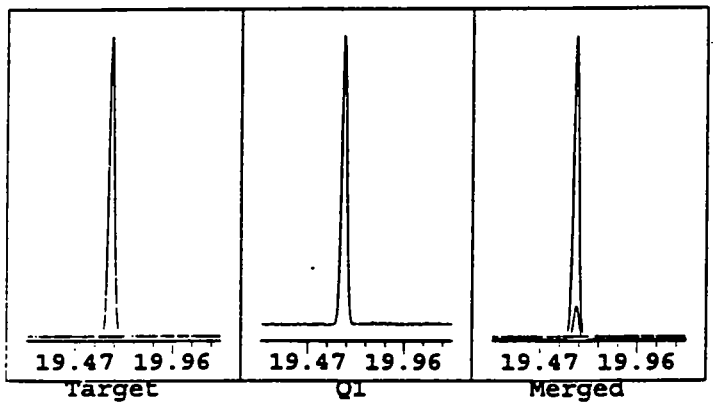


Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

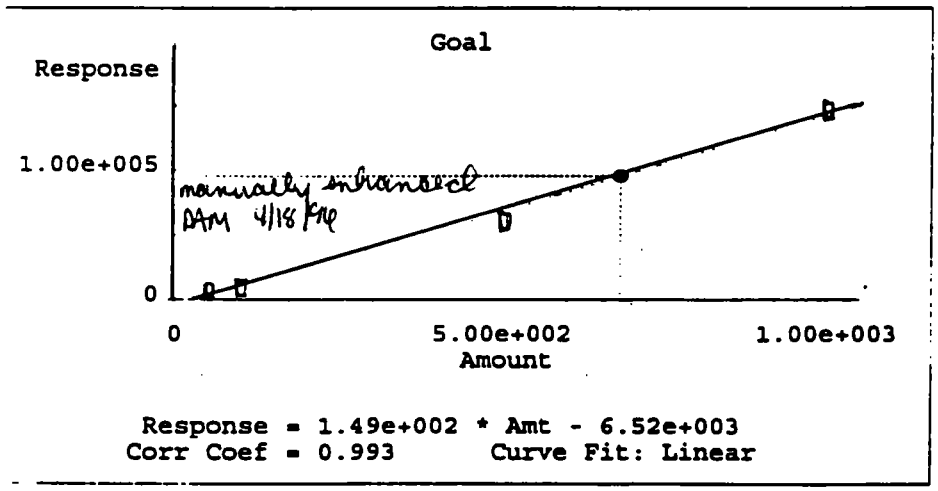
Figure 94. Standard 0.50 µg/ml (GC/MS)

File : C:\HPCHEM\1\DATA\N08S1.D
Operator : sz
Acquired : 9 Nov 95 5:35 pm using AcqMethod GOAL1
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 1
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 682.50
Pk # and Type: 1 RTEINT used

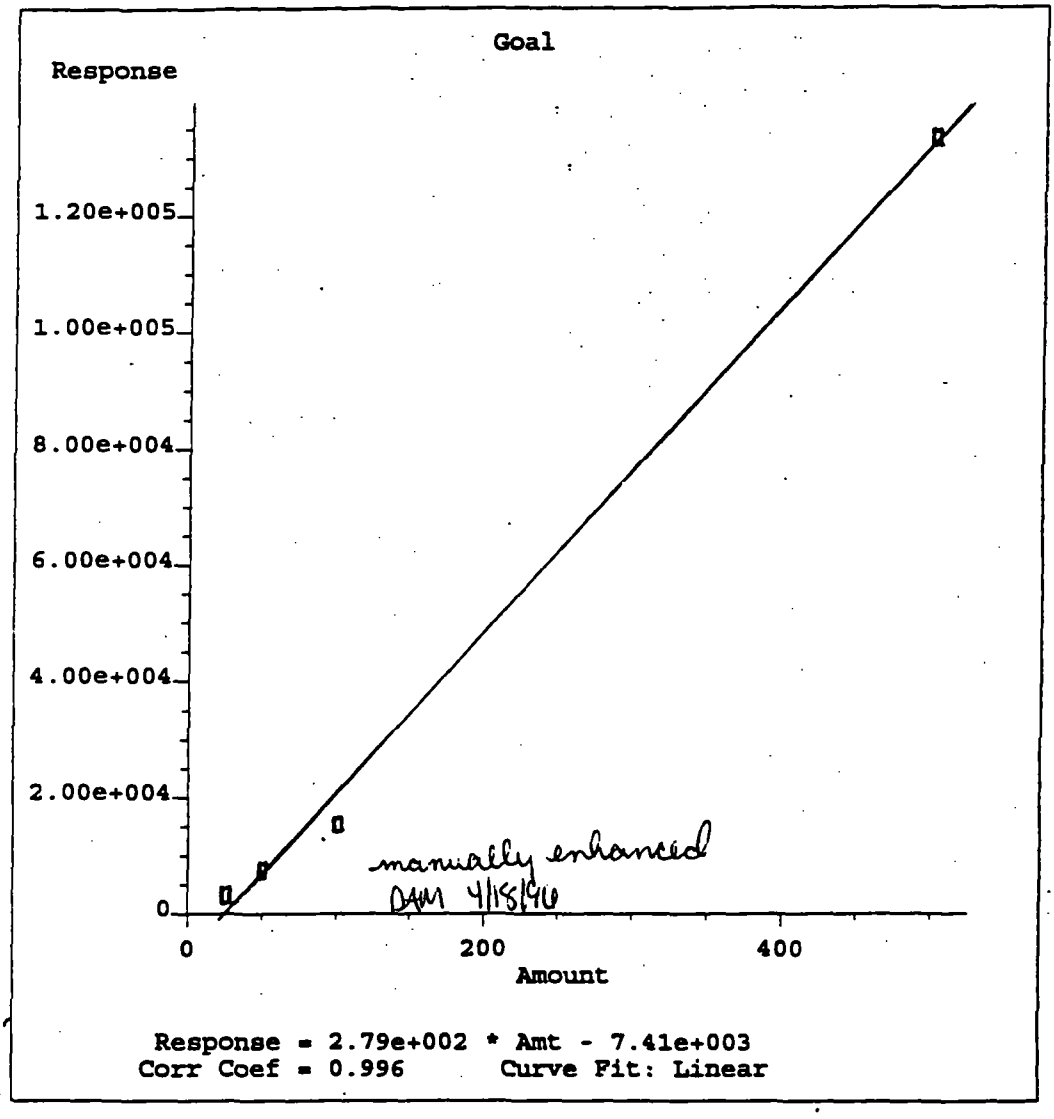


	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.22	95162	sys def
Q1	361.00	11.1	0.0-	22.0	19.65	to	10593	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def



Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

Figure 95. Oxyfluorfen Calibration Curve (GC/MS)



Method Name: C:\HPCHEM\1\METHODS\GOAL1.M
Calibration Table Last Updated: Tue Nov 14 09:59:48 1995

Appendix 1

GC/MS Method Validation Protocol
Certificate of Analysis (RH-32915)
GLP Statements
Quality Assurance Statements

I. **Protocol Number:** **34P-95-92**

Project Number: **3107.14**

II. **Study Title**

Method Validation- GC/MS Confirmatory Detection in Oxyfluorfen (Goal®) Crop Residue Analytical Method, TR 34-94-150

III. **Study Objective**

The objective is to validate the Oxyfluorfen (GOAL®) Crop Residue Analytical Method, TR 34-94-150, using GC/MS as the confirmatory detection method. The crops that will be used are peanut nutmeat, peanut shell, peach, and grape*. The data will be used in the reissue of this residue method as the Tolerance Enforcement Method, TR 34-95-111.

*These samples will be taken from the residue analysis covered in subprotocol 34P-94-65A.

IV. **Sponsor**

Rohm and Haas Company
727 Norristown Road
Spring House, PA 19477-0904

V. **Testing Laboratory**

Centre Analytical Laboratories
3048 Research Drive
State College, PA 16801

VI. **Personnel**

5.1	Study Director:*	Dennis A. Martin Rohm and Haas Company 215-619-5576
5.3	Principal Investigator:	Shaozhi Zheng Centre Analytical Laboratories

5.2	Contract Laboratory Coordinator:**	Roberta C. Regetta Rohm and Haas Company 215-619-5588
5.4	RMEF Manager:	Dr. Stanley S. Stavinski Rohm and Haas Company

*Primary contact for technical issues
**Primary contact for non-scientific issues

VII. Dates

Proposed Analytical Start Date: October 1995

Proposed Experimental Completion: November 1995

Proposed Final Report Date: November 1995

VIII. Identification and Justification For Selection of Test System

The test system for this study is defined as sets of untreated crop samples collected from field trials. Samples will be identified by the Rohm and Haas sample numbers defined later in this protocol. The samples are selected to support magnitude of residue studies. The crops specifically to be used in this study are peanut nutmeat, grape, peanut shell, and peach.

IX. Experimental Design and Methods to Control Bias

Untreated crop samples will be fortified with oxyfluorfen and run through the method to validate the GC/MS method as the confirmatory detection technique. Sample analysis will follow documented standard operating procedures. Methods to control bias include analysis of untreated control samples, fortification recovery data, and generation of standard curves.

X. Method

All analyses and preparation of stock fortification solutions will be performed using the residue analytical method TR 34-94-150, "Oxyfluorfen (GOAL®) Crop Residue Analytical Method." However, there is the addition of GC/MS detection, developed by Centre

Analytical Laboratories, as the mode of quantitation in place of GC/ECD. (Data acceptability - SOP RMEF.RES.05.02)

XI. Test and Reference Substances

1. Test and Reference Material

Standard	Lot	Purity/%	Appearance	Expiration Date
RH-32915 * oxyfluorfen	RPO8674FP	99.4	Pale yellow powder	August 2, 2000

Analytical grade compounds will be used as both a test substance, for fortifications, and a reference substance, to generate calibration curves. The reference material has been GLP analyzed and possesses a certificate of analysis. The characterization of the reference substance is detailed in Rohm and Haas report, APR-95-334.

* RH-32915 and RH-2915 identify the same standard. The former appears on the certificate of analysis, the latter is used in the residue database.

2. Chemicals- The grade and supplier of chemicals are defined in the method.

3. Hazard Information- Current MSD sheets for the standards will be available by the testing laboratory.

XII. Treatment of Test System

The samples used are untreated control samples from field studies. The RMEF RAR code system will be used to identify all samples. The samples have been processed according to SOP SAMP.02.06; and, they are maintained in frozen storage until analysis.

The following samples will be analyzed. Each set will consist of one control and three fortifications. The fortifications will be 1 at the LOQ, 1 at 2 X LOQ, and 1 at 5 X LOQ. The grape samples are taken from subprotocol 34P-94-65A.

RAR Code	Sample No.	Variety	Component
93-0157	004	peanut	nutmeat
93-0157	004	peanut	shell
94-0117	001	peach	fruit
92-0100	005	Cabernet grape	fruit

XIII. Records

Originals of the items listed below, and any other raw data generated will be provided to the Study Director.

1. Chromatograms

   All chromatograms will contain the following:

   A. Sample identification, date, final sample volume, sample weight, arrows indicating the area of interest, the analyst's initials, and injection number corresponding to the run sheet.

   B. Fortifications will additionally include the µg added and the sample number of the sample that was fortified.

   C. Standard chromatograms will additionally include the µg/ml concentration.

   D. The first standard chromatogram in a daily run should also include temperatures, flow rates, column parameters, gases, instrument parameters, and instrument type if any of these differ from the method.

2. Storage History and Chain of Custody Records

3. Method Run Sheets

4. Diskettes of Results

5. Analyst's Bench Sheet

XIV. Reporting

1. Data Reporting

   The testing laboratory will provide the original version of run sheets, standard curves, and any other raw data generated. Rohm and Haas will provide the original processing records. Work perfomed will be certified by the signature of all personnel involved in the analysis. Signed GLP Compliance and Quality Assurance Statements should also be provided.

2. Final Report

The analytical data generated under this protocol will be used in the reissue of the Oxyfluorfen Crop Residue Analytical Method, TR 34-94-150, as the Oxyfluorfen Crop Tolerance Enforcement Method, TR 34-95-111. The final method report will be written by the Study Director upon completion of this analysis. The report will include data generated under other protocols covering residue analysis of samples used in the reregistration of oxyfluorfen. The report will include a description of the materials and methods used, the residue analytical data generated, along with signed and dated Study Compliance and Quality Assurance Statements.

XV. GLP Statement

This analysis will be conducted in compliance with FIFRA's Good Laboratory Practice Standard (40 CFR 160). All raw data generated in this study will be archived by the Rohm and Haas Company. The Study Director will be responsible for the overall conduct of the study and the final report. The Principal Investigator is the Study Director's delegate responsible for this protocol, oversight of the analysis, the analytical experimentation, and will review the protocol, procedures, Good Laboratory Practice and safety guidelines with all support personnel prior to their participation in this study. Any deviations will be recorded. Changes in plans will be approved by the Study Director and attached to this protocol.

XVI. Quality Assurance

The Centre Analytical Laboratories, Inc's QA Unit will conduct in-progress inspections, audit the raw data to ensure the integrity of the study, and will report findings, noting any problem found and corrective actions taken, to site management, the study director, Rohm and Haas QAU, and Rohm and Haas Management.

XVII. Signatures

Study Director:

Dennis A. Martin 10/19/95

Dennis A. Martin Date

RMEF Manager:

Oct 19/95

Dr. Stanley S. Stavinski Date

Principal Investigator:

Shaozhi Zheng 10/24/95

Shaozhi Zheng Date

Reviewed by QAU

Rhonda S. Krick 10/19/95

Rhonda S. Krick Date

TR 34 95 111

000147

PROTOCOL MODIFICATION FORM

SUBPROTOCOL/ PROTOCOL No. 34P-95-92 **STUDY DIRECTOR** Dennis A. Martin

STUDY TITLE Method Validation - GC/MS Confirmatory Detection in Oxyfluorfen (Goal) Crop Residue Analytical Method (TE 34-94-150)

MODIFICATION:

① Analyze crop samples generated by GC/ECD as well as GC/MS, using method found in TE 34-94-150.

JUSTIFICATION:

② Results are needed on both detection methods to use for comparison.

EFFECT ON STUDY:

None.

Originator: Dennis A. Martin 11-14-95
signature date

Study Director Approval: Dennis A. Martin 11-14-95
signature date

TR 34 95 111

000148

PROTOCOL MODIFICATION FORM

SUBPROTOCOL/ PROTOCOL No. 34P-95-92 **STUDY DIRECTOR** D.A. Martin

STUDY TITLE Method Validation- GC/MS Confirmatory Detection in Oxyfluorfen (Goal®) Crop Residue Analytical Method, TR 34-94-150

MODIFICATION:

The LOQ for the confirmatory method using GC/MS is 0.025 ppm, instead of 0.01 ppm as the primary method dictates.

JUSTIFICATION:

The sensitivity of the confirmatory detection method does not need to be the same as the primary method. The LOQ of 0.025 ppm results in the best chromatography for this method.

EFFECT ON STUDY:

There will be no effect on the study.

Originator: Jennis A. Martin 12/18/95
signature date

Study Director Approval: Jennis A. Martin 12/18/95
signature date

TR 34 95-111

Centre Analytical Laboratories, Inc.
3048 Research Drive State College, PA 16801
Phone: (814) 231-8032 Facsimile: (814) 231-1253

000149

PROTOCOL/METHOD MODIFICATION FORM

Subprotocol/Protocol No: 34P-95-92
Study Director: Dennis Martin

CAL Project Number: 002-165 Sponsor Study Number: 3107.14* ~~34P-95-92~~

Study Title: Method Validation - GC/MS Confirmatory Detection in Oxyfluorfen (Goal) Crop Residue Analytical method, TR34 ~~95-150~~.

MODIFICATION:

1. In step 6.6, 40 ml of 50:50 (V/V) ethyl acetate/hexane was used instead of 25 ml of 5:95 (V/V) ethyl acetate/hexane.
2. The final volume was changed from 5ml to 2.5ml.

JUSTIFICATION:

1. ~~2 modifications were issued~~. SZ 11/14/95
1. ensure the good recovery
2. ensure the fortification ~~lev~~ sample within the standard range.

EFFECT ON STUDY:

1. none
2. none.

Originator: Shaozhi Zheng 11/10/95
Date

Study Director: Dennis A. Martin 1/10/96
Date

TR 34 95 111

Centre Analytical Laboratories, Inc.
3048 Research Drive State College, PA 16801
Phone: (814) 231-8032 Facsimile: (814) 231-1253

000150

Page 1 of 1

PROTOCOL/METHOD MODIFICATION FORM

Subprotocol/Protocol No: 34P-95-92
Study Director: Dennis A. Martin

CAL Project Number: 002-165 Sponsor Study Number: 3107.14

Study Title: Method Validation - GC/MS Confirmatory Detection in Oxyfluorfen (Goal) Crop Residue Analytical Method, TR34-94-150

MODIFICATION:

1. 1000 ug/ml stock solution was made instead of 200ug/ml.

JUSTIFICATION:

1. It is laboratory practice to prepare stock solution concentration of 1000ug/ml in order to use the solution for different projects.

EFFECT ON STUDY:

1. None.

Originator: Shaozhi Zheng 1/2/96
Date

Study Director: Dennis A. Martin 1/10/96
Date

July 1993/0

TR 34 95 111

Centre Analytical Laboratories, Inc.
3048 Research Drive State College, PA 16801
Phone: (814) 231-8032 Facsimile: (814) 231-1253

0001451

PROTOCOL/SOP/METHOD DEVIATION

Deviation Number: 1
Date of Occurrence: 10/31/95

CAL Project Number: 002-165 Sponsor Study Number: 3107.14

DESCRIPTION OF DEVIATION

Is this deviation a ✓ PROTOCOL, or ____ SOP, or ____ METHOD deviation?

standard (RH-32915) purity is 99.7% and expiration date is 7/12/2000.

ACTIONS TAKEN

i.e., amendment issued, SOP revision, etc...

protocol deviation was issued.

Recorded By/Date: Shaozhi Zheng 1/3/96

IMPACT ON THE STUDY

None.

Dennis A. Martin 1/10/96
Study Director Signature Date

CAL QAU Review GW 1/5/96

May 1993/1

TR 34 95 111

INDEPENDENCE MALL WEST PHILADELPHIA, PA. 19105, U.S.A. TELEPHONE (215) 592-3000
CABLE ADDRESS: ROHMHAAS TELEX 845-247 TWX 710-670-5335 TELECOPIER (215) 592-3377
REPLY TO:
RESEARCH LABORATORIES
727 NORRISTOWN ROAD
P.O. BOX 904
SPRING HOUSE, PA 19477-0904
(215) 641-7000
(215) CH 2-0400

000152

ROHM AND HAAS COMPANY

CERTIFICATE OF ANALYSIS
ANALYTICAL STANDARD

RH NUMBER:	**32915**
LOT NUMBER:	**RPO8674FP**
MATERIAL NAME:	**GOAL**
PRODUCTLINE:	**GOAL**
PURITY:	**99.4 %**
GLP ANALYSIS:	**YES**
STORAGE CONDITIONS:	**ROOM TEMP. OR BELOW**
APPEARANCE:	**PALE YELLOW POWDER**
EXPIRATION DATE:	**02-aug-2000**
DATE OF LAST ANALYSIS:	**02-aug-1995**

QUERIED BY Denise Millegan **DATE** 28-sep-1995

(Signature)

Supporting documentation for this information is retained at the Rohm and Haas Company in the AgChem Analytical Standard Archive.

GLP COMPLIANCE STATEMENT

CAL Study Number 002-165 "Method Validation - GC/MS Confirmatory Detection in Oxyfluorfen (Goal®) Crop Residue Analytical Method, TR 34-94-150" was performed in compliance with EPA Good Laboratory Practice Standards (40 CFR, Part 160; FR. 8/17/89) by Centre Analytical Laboratories, Inc.

Shaozhi Zheng
Shaozhi Zheng
Principal Investigator

4/14/96
Date

QUALITY ASSURANCE STATEMENT

CAL Study Number 002-165 was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, the Study Protocol and all applicable Good Laboratory Practice Standards. All findings were reported to the study director and management.

	Phase	Date Inspected	Date Reported to Principal Investigator and CAL management	Date Reported to Study Director and Sponsor management
1.	Protocol Review	11/01/95	11/30/95	12/18/95
2.	Extraction	11/02/95	11/30/95	12/18/95
3.	Extraction	11/09/95	11/30/95	12/18/95
4.	Raw Data Review	12/06/95	01/10/96	02/02/96

Gaya Wickremesinhe
Gaya Wickremesinhe
Quality Assurance Officer

4/4/96
Date

The GLP and QA statements that follow are for additional fortifications submitted after completion of the reports, as listed below:

TR #	MRID #	Protocol #	Crop
34-95-116	43794003	34P-95-30A	fig
34-95-119	43794006	34P-95-33A	olive
34-95-117	43794004	34P-95-31A	pomegranate

GLP COMPLIANCE STATEMENT

CAL Study Number 002-146 "GOAL® 1.6E Field Residue Trials on Figs" was performed in compliance with EPA Good Laboratory Practice Standards (40 CFR, Part 160; FR. 8/17/89) by Centre Analytical Laboratories, Inc.

Shaozhi Zheng — 3/19/96

Shaozhi Zheng
Principal Investigator

Date

QUALITY ASSURANCE STATEMENT

CAL Study Number 002-146 was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, Study Protocol and all applicable Good Laboratory Practice Standards. All findings were reported to the study director and management.

	Phase	Date Inspected	Date Reported to Principal Investigator and CAL management	Date Reported to Study Director and Sponsor management
1.	Protocol Review	07/10/95	07/20/95	08/18/95
2.	Extraction	07/11/95	08/16/95	08/18/95
3.	Raw Data Review	08/07-08/95	08/16/95	08/18/95
4.	Raw Data Review	08/18/95	09/25/95	12/18/95

Gail L. Keller — 3/19/96

Gail L. Keller
Quality Assurance Officer

Date

GLP COMPLIANCE STATEMENT

CAL Study Number 002-151 "GOAL® 1.6E Field Residue Trials on Olives" was performed in compliance with EPA Good Laboratory Practice Standards (40 CFR, Part 160; FR. 8/17/89) by Centre Analytical Laboratories, Inc.

Shaozhi Zheng — 3/19/96

Shaozhi Zheng
Principal Investigator

Date

QUALITY ASSURANCE STATEMENT

CAL Study Number 002-151 was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, Study Protocol and all applicable Good Laboratory Practice Standards. All findings were reported to the study director and management.

Phase	Date Inspected	Date Reported to Principal Investigator and CAL management	Date Reported to Study Director and Sponsor management
1. Protocol Review	07/07/95	07/20/95	08/18/95
2. Extraction	07/07/95	08/16/95	08/18/95
3. Raw Data Review	08/08/95	08/16/95	08/18/95
3. Raw Data Review	08/18/95	09/25/95	12/18/95

Gail L. Keller — 3/19/96

Gail L. Keller
Quality Assurance Officer

Date

GLP COMPLIANCE STATEMENT

CAL Study Number 002-147 "GOAL® 1.6E Field Residue Trials on Pomegranates" was performed in compliance with EPA Good Laboratory Practice Standards (40 CFR, Part 160; FR. 8/17/89) by Centre Analytical Laboratories, Inc.

Shaozhi Zheng — 3/19/96

Shaozhi Zheng
Principal Investigator

Date

QUALITY ASSURANCE STATEMENT

CAL Study Number 002-147 was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, Study Protocol and all applicable Good Laboratory Practice Standards. All findings were reported to the study director and management.

	Phase	Date Inspected	Date Reported to Principal Investigator and CAL management	Date Reported to Study Director and Sponsor management
1.	Protocol Review	07/10/95	07/20/95	08/18/95
2.	Extraction	07/07/95	08/16/95	08/18/95
3.	Raw Data Review	08/08/95	08/16/95	08/18/95
3.	Raw Data Review	08/18/95	09/25/95	12/18/95

Gail L. Keller

Gail L. Keller
Quality Assurance Officer

3/19/96

Date

The GLP and QA statements that follow are for peach fortifications which will be reported, as listed below:

TR #	MRID #	Protocol #	Crop
34-95-114	not yet assigned as of study completion	34P-95-35A 34P-95-51A	peach

GLP Compliance Statement

We, the undersigned, hereby certify that the data contained in this raw data package for Rohm & Haas Sub Protocol Number 34P-95-35A, "GOAL® 1.6E Field Residue Trials on Peaches," was generated in compliance with Good Laboratory Practice Standards (40 CFR Part 160) applicable to analytical testing facilities.

Kathryn Kobtan for Melinda Lalko n/c — 04AUG 95

Melinda Lalko, Residue Lab Supervisor — [Date]
Principal Investigator

Diana Price, Quality Assurance Supervisor — 04 Aug 95 — [Date]

Kathleen Lacey, Laboratory Director — 04AUG 95 — [Date]

Quality Assurance Statement

This data package for Rohm & Haas Sub Protocol Number, 34P-95-35A, "GOAL® 1.6E Field Residue Trials on Peaches," has been reviewed by the Quality Assurance Unit of McKenzie Laboratories, Inc. It has been found to accurately describe and/or identify the methods and protocol followed in the conduct of the study. In addition, the Quality Assurance Unit conducted the following inspections of this study and has submitted written reports of these inspections to the Study Director, their management, and to the management of McKenzie Laboratories, Inc.

Date Inspected	Phase	Date reported to: Study Director and Management	Date reported to: McKenzie Management
06 Jul 95	• Fortification • Extraction • Partitioning • Concentration • Protocol/Amendments • Test System Receipt Documentation • Analytical Reference Substance Receipt Documentation • Chain of Custody • Study Notebook • Training Files • Standard Preparation Documentation	17 Jul 95*	12 Jul 95
07 Jul 95	• Column Clean-up • Centrifuge • Equipment/Instrument Use Logs • Data Packages	17 Jul 95*	12 Jul 95
04 Aug 95	• Data Package Review • Review of Study Package	04 Aug 95*	04 Aug 95

* Date inspection report was sent to the Study Director.

Diana L Price, Quality Assurance Supervisor

04 Aug 95
[Date]

TR 34 95 111

000165

GOAL® 1.6E Field Residue Trials on Peaches
Protocol Number: 34P-95-51A

GLP Compliance Statement

We, the undersigned, hereby certify that the data contained in this report was generated in compliance with Good Laboratory Practice Standards (40 CFR Part 160) applicable to analytical testing facilities. All original raw data (or certified copies thereof) have been provided to Rohm and Haas Company and copies (or facility-related originals) have been retained at McKenzie Laboratories, Inc.

Diana Price, Quality Assurance Supervisor — [Date] 15 Dec 95

Melinda Lalko, Principal Investigator
Residue Laboratory Supervisor — [Date] 15 Dec 95

Kathryn Koktavy
Residue Laboratory Manager — [Date] 15 Dec 95

Kathleen Lacey, Laboratory Director — [Date] 15 DEC 95

Quality Assurance Statement

This data package for Sub Protocol Number: 34P-95-51A (Goal ® 1.6E Field Residue Trials on Peaches), has been reviewed by the Quality Assurance Unit of McKenzie Laboratories, Inc. It has been found to accurately describe and/or identify the methods and protocol followed in the conduct of the study. In addition, the Quality Assurance Unit conducted the following inspections of this study and has submitted written reports of these inspections to the Study Director, their management, and to the management of McKenzie Laboratories, Inc.

		Date reported to:	
Date Inspected	Phase	Study Director and Management	McKenzie Management
21 Nov 95	• Fortification • Extraction • Partitioning • Protocol/Amendments • Study Notebook	*11 Dec 95	07 Dec 95
22 Nov 95	• Training Files	*11 Dec 95	07 Dec 95
28 Nov 95	• Column Clean-up • Chain-of-Custody • Gas Chromatography • Test System Receipt Documentation	*11 Dec 95	07 Dec 95
29 Nov 95	• Maintenance Logs • Equipment/Instrument Use Logs	*11 Dec 95	07 Dec 95

* Date inspection report was sent to the Study Director/ Study Director Management.

Diana L Price, Quality Assurance Supervisor

14 Dec 95
[Date]

Appendix II

Representative Analytical Run (GC/ECD)
Representative Analytical Run (GC/MS)

TR 34 95 111

000168

GC RUN SHEET

Date 7/28/95
Analyst Julie Bunton
Instrument Varian 3500 GC14
Analytical Method TR34-94-150
Notebook Ref. 002-146
Crop Fig

Compound GDAL
Data Reduction Method GDAL.Vm
Raw Data File L26JA
Date Extracted 7/25-26/95
Date Injected 7/28/95. 7/29/95

Inj. No	STD (µg/mL)	RAR No.	Spl. No	Sample Component	Sample Weight (g)	Final Volume (mL)	Fortification Level (µg)	Notes
1	0.005							
2	0.01							
3	0.1							
4	0.1							
5		94-0142	001	Fig	5.0	5.0		
6		↓	↓	↓	↓	↓	0.05	Spk A
7							↓	Spk A$_2$
8								Spk A$_3$
9		↓	↓	↓	↓	↓		Spk A$_4$
10	0.01							
11	0.05							
12		94-0142	001	Fig	5.0	5.0	0.1	Spk B$_1$
13		↓	↓	↓	↓	↓	↓	Spk B$_2$
14							0.25	
15						↓	0.5	
16		↓	↓	↓	↓	100.0	2.5	
17	0.005							
18	0.05							
19	0.15							
20	0.15							
21								
22								
23								
24								
25								
26								

MA→B 7/28/95

NOTES: Samples stored in freezer 1DA prior to analysis. JB 7/28/95

Signature Julie Bunton

June 1995/1

TR 34 95 111

000169

Data file: L26JA1 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 16:22 Method: GOALVa Analyst : JB

Interface: 707 Cycle#: 1 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

Prog Slope (# or Linear): NA Inj Port Temp: 265°C Cycle 1-20

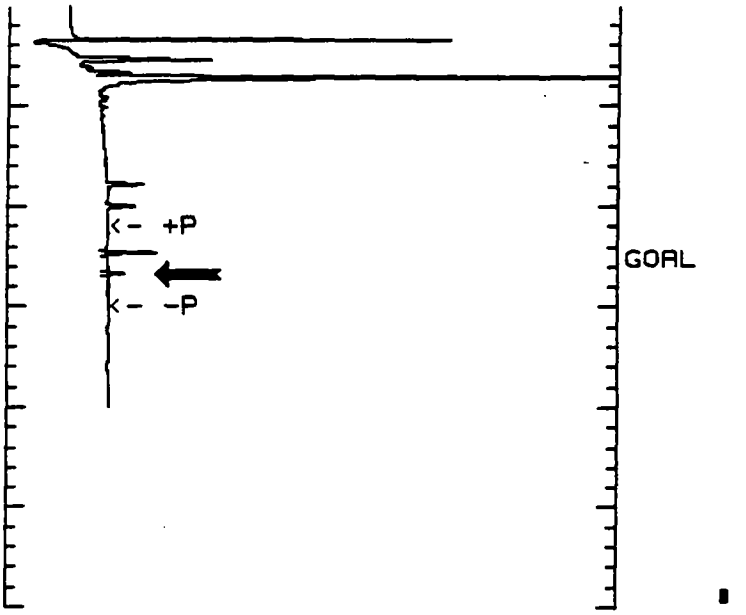
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.13 mv offset)

Initials JB Date 8/7/95 Run# 1 to 20



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.005	1.460E+00	1.270E+02

TR 34 95 111

000170

Data file: L26JA1 Type: STANDARD

Sample Name: MA Cal. Curve: 07/28/95

Date: 28 Jul 1995 16:47 Method: GOALVa Analyst : JB

Interface: 707 Cycle#: 2 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 315 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

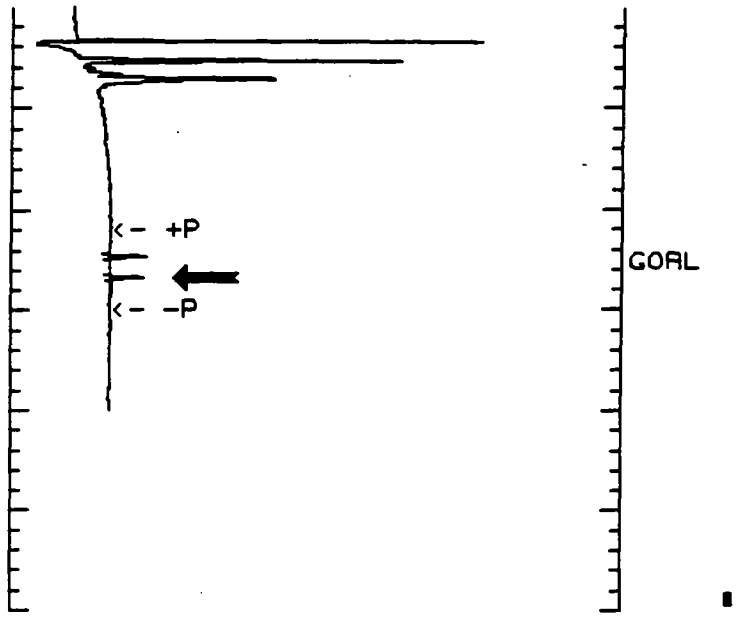
Prog Slope (# or Linear): MA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: MA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.12 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.37	GOAL	0.010	1.220E+00	2.660E+02

TR 34 95 111

Data file: L26JA3 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 17:12 Method: GOALVa Analyst : JB

Interface: 707 Cycle#: 3 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

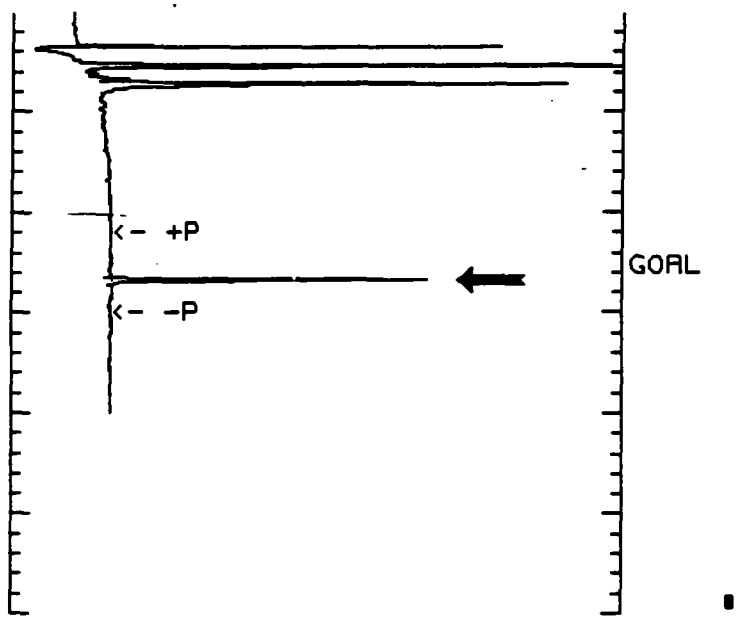
Prog Slope (0 or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.13 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.38	GOAL	0.100	3.090E+01	2.590E+03

TR 34 95 111

000172

Data file: L26JA4 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 17:37 Method: GOALVa Analyst : JB

Interface: 707 Cycle#: 4 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

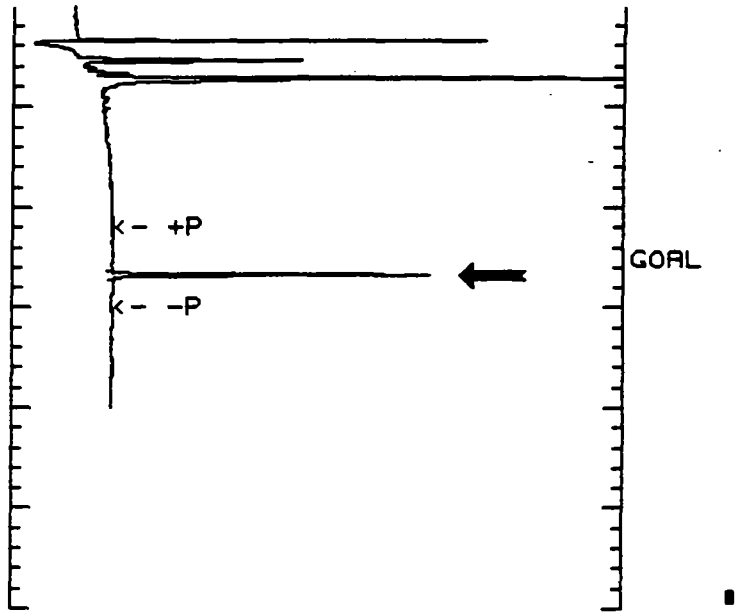
Prog Slope (# or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.15 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.37	GOAL	0.100	3.060E+01	2.590E+03

TR 34 95 111

000173

Data file: L26JA10 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 20:08 Method: GOALVm Analyst : JB

Interface: 707 Cycle#: 10 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

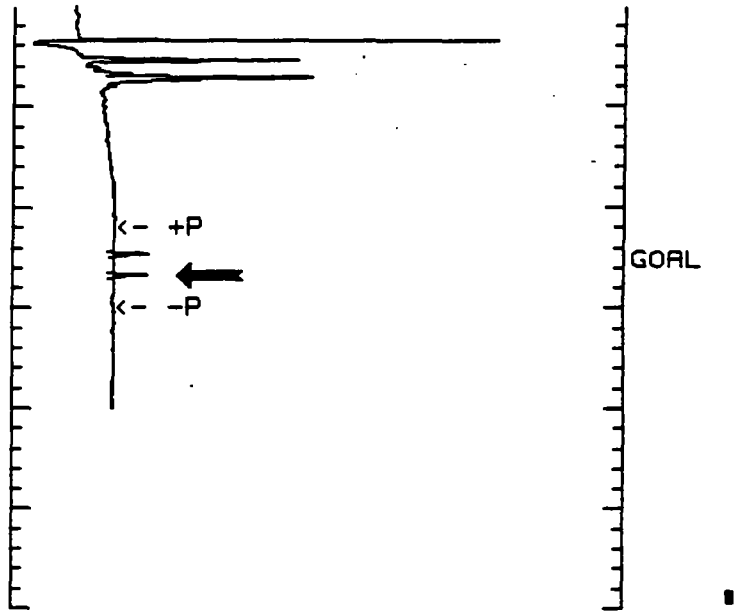
Prog Slope (# or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.6ml/mnHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.094 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.37	GOAL	0.010	3.370E+00	2.760E+02

TR 34 95 111

000174

Data file: L26JA11 Type: STANDARD

Sample Name: NA Cal. Curve: 07/26/95

Date: 28 Jul 1995 20:33 Method: GOALVm Analyst : JB

Interface: 707 Cycle#: 11 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

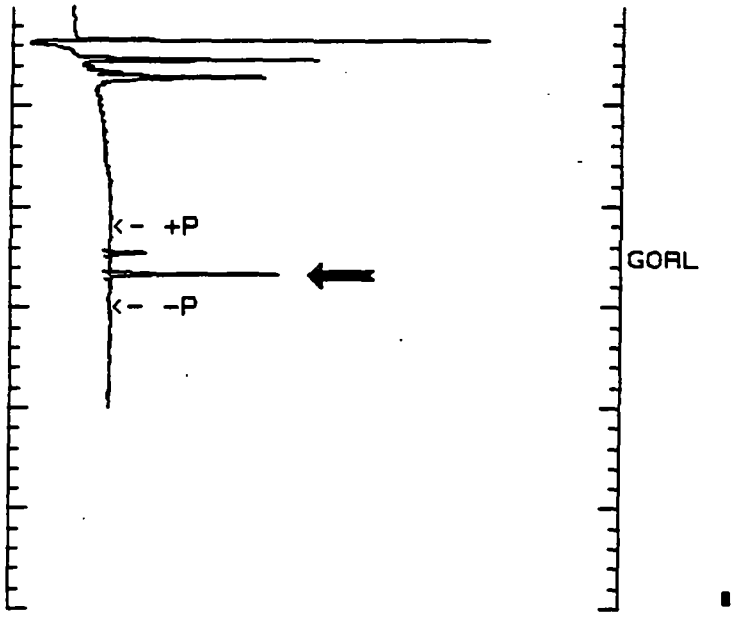
Prog Slope (0 or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.14 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.050	1.680E+01	1.380E+03

TR 34 95 111

000175

Data file: L36JA17 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 23:05 Method: GOALVm Analyst : JB

Interface: 707 Cycle#: 17 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

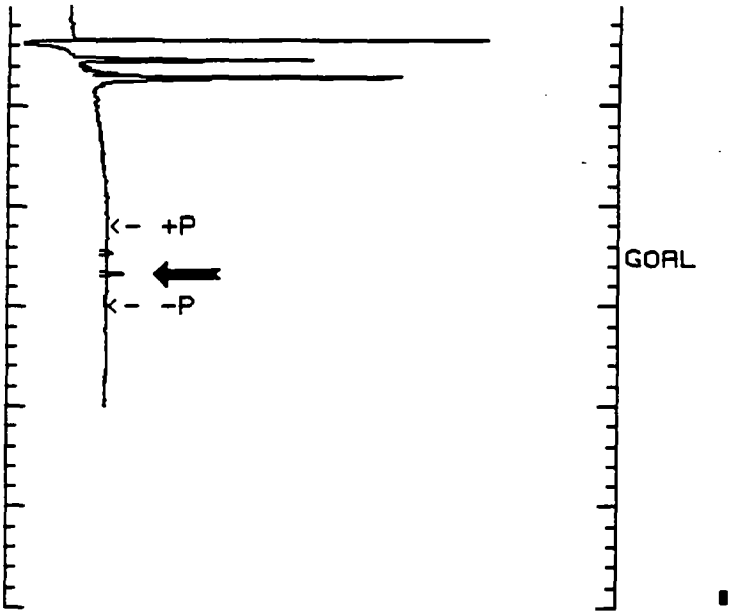
Prog Slope (# or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/mHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.15 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.005	1.540E+00	1.320E+02

TR 34 95 111

000176

Data file: L26JA18 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 23:30 Method: GOALVm Analyst : JB

Interface: 707 Cycle#: 18 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

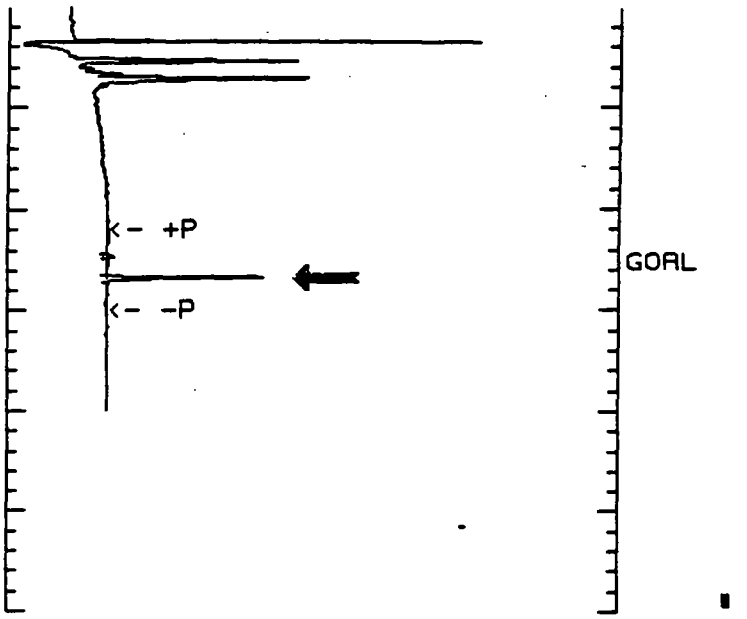
Prog Slope (0 or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.15 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.050	1.560E+01	1.290E+03

TR 34 95 111

000177

Data file: L26JA19 Type: STANDARD

Sample Name: NA Cal. Curve: 07/28/95

Date: 28 Jul 1995 23:55 Method: GOALVa Analyst : JB

Interface: 707 Cycle#: 19 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

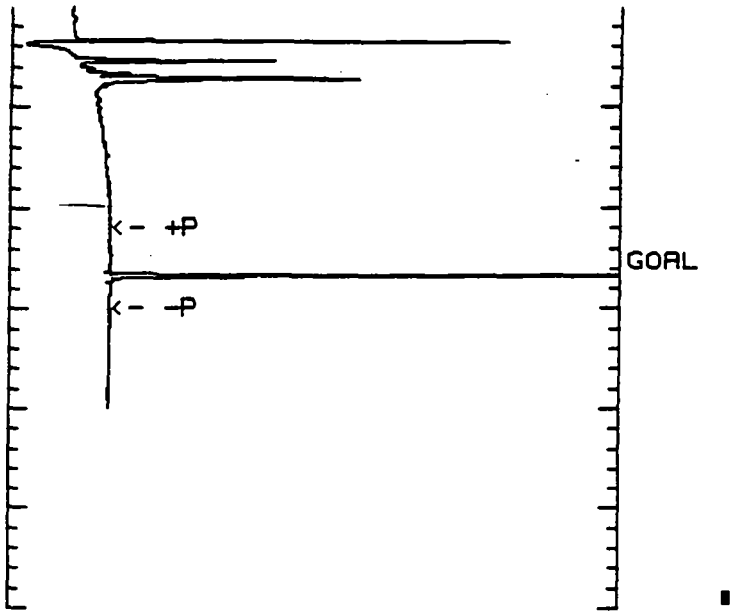
Prog Slope (0 or Linear): NA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: NA

Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.15 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.150	5.270E+01	4.580E+03

Data file: L36JA30 Type: STANDARD

Sample Name: MA Cal. Curve: 07/28/95

Date: 29 Jul 1995 00:21 Method: GOALVm Analyst : JB

Interface: 707 Cycle#: 20 Channel#: A

Instrument: VARIAN 3500,

Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters

Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1

Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17

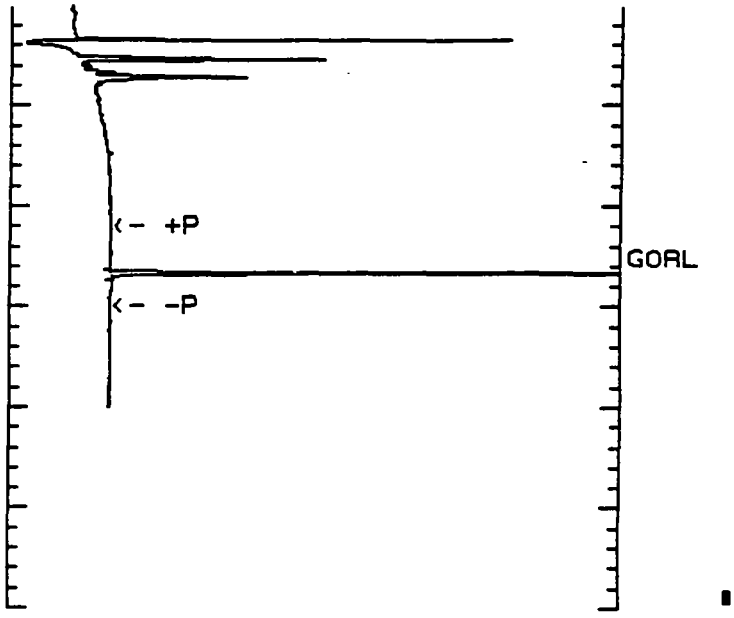
Prog Slope (0 or Linear): MA Inj Port Temp: 265

Flowrate/Gas: 3.5ml/minHE Split Ratio: MA

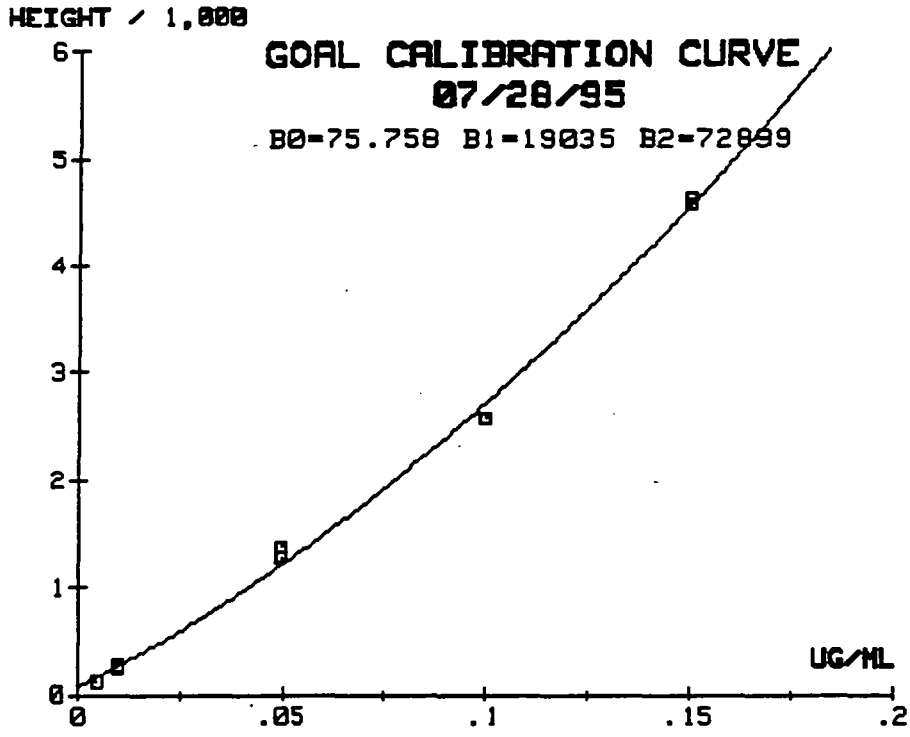
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot times: 0 to 30 minutes

Plot range: 50 millivolts (-.16 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
13.36	GOAL	0.150	5.250E+01	4.640E+03



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

TR 34 95 111

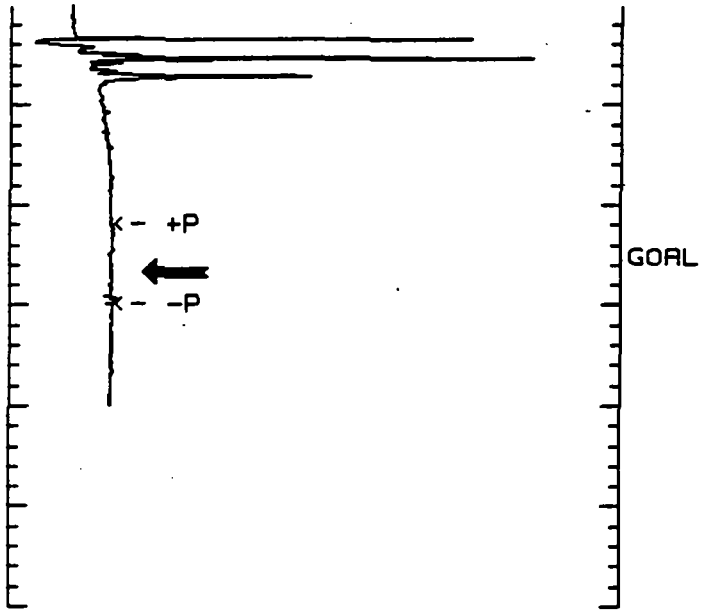
000180

Data file: L36JA5 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: SAMPLE Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 18:02 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 5 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.13 mv offset)



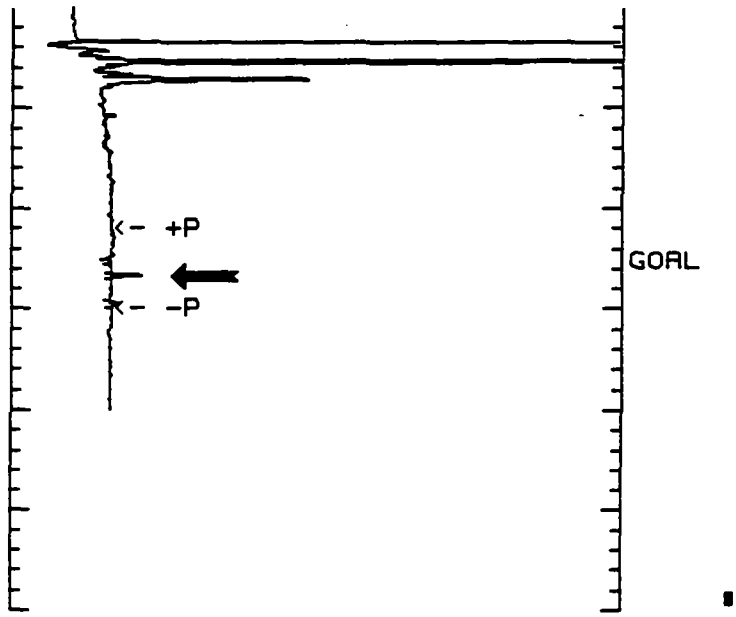
Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.
13.37	GOAL	0.000E+00	.000E+00	5.00	5.00

Data file: L26JA6 RAR number: 94-0142
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: MA Cal. Curve: 07/28/95
Date: 28 Jul 1995 18:27 Method: GOALVm Analyst : JB
Interface: 707 Cycle#: 6 Channel#: A

Instrument: VARIAN 3800,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): MA Inj Port Temp: 265
Flowrate/Gas: 3.8ml/minHE Split Ratio: MA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot time: 0 to 20 minutes
Plot range: 50 millivolts (-.17 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	2.970E+00	.249E+03	5.00	5.00	0.050

TR 34 95 111

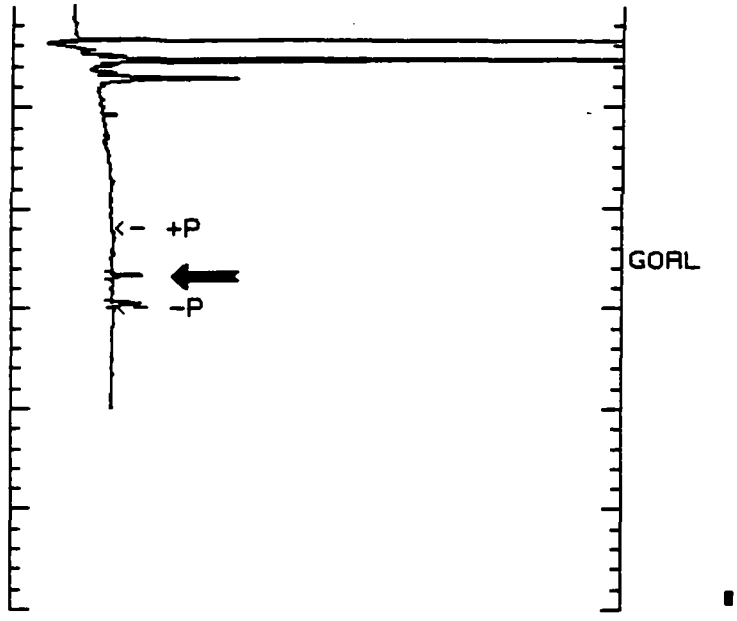
000182

Data file: L36JA7 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 18:52 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 7 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/min HE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.12 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.36	GOAL	2.990E+00	.246E+03	5.00	5.00	0.050

TR 34 95 111

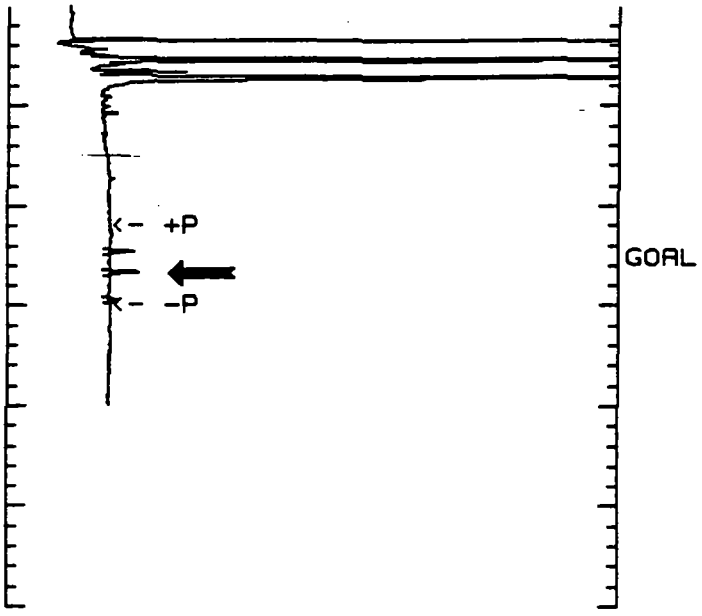
000183

Data file: L26JA8 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 19:18 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 8 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/mnHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.14 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	2.920E+00	.241E+03	5.00	5.00	0.050

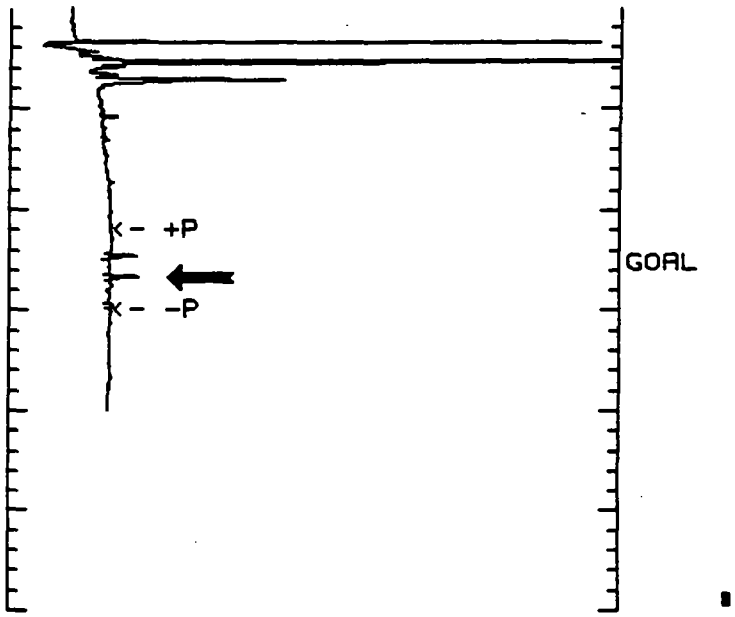
TR 34 95 111

Data file: L36JA9 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 19:43 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 9 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.15 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	2.770E+00	.234E+03	5.00	5.00	0.050

TR 34 95 111

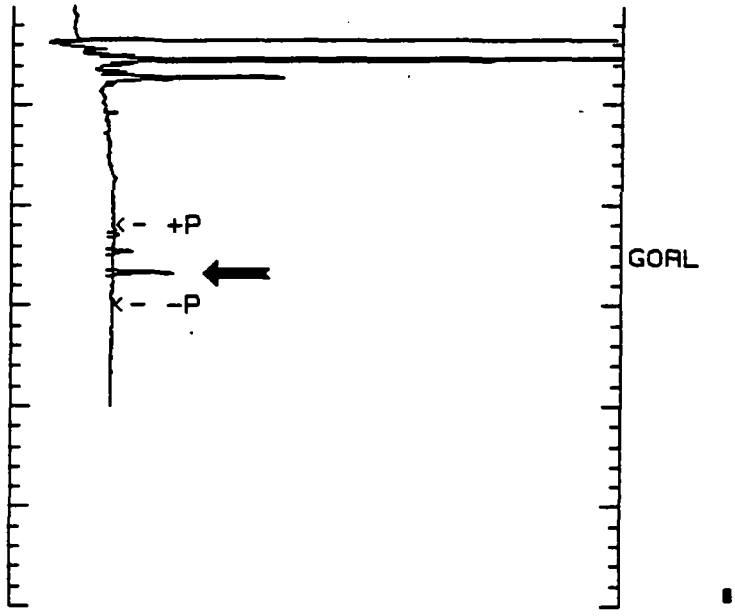
000185

Data file: L26JA12 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 20:59 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 12 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.095 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	6.140E+00	.499E+03	5.00	5.00	0.100

TR 34 95 111

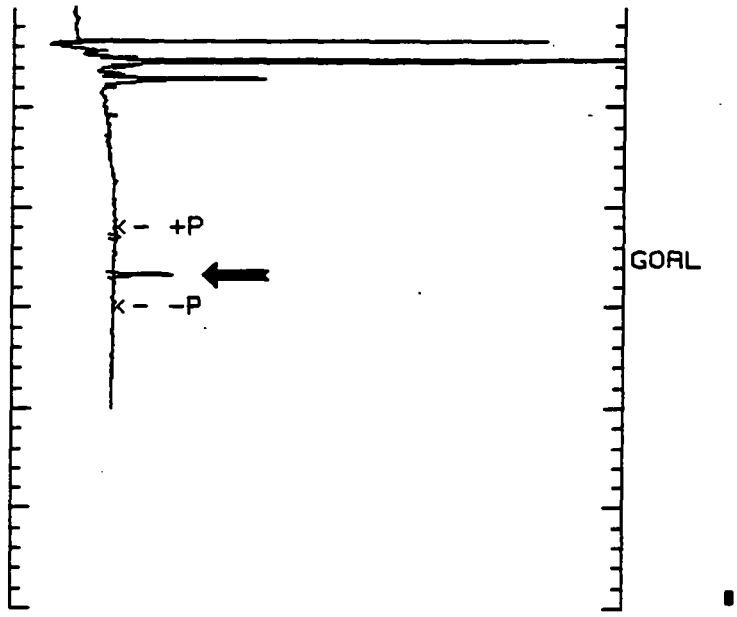
000186

Data file: L36JA13 RAR number: 94-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 21:24 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 13 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (# or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot times: 0 to 30 minutes
Plot range: 50 millivolts (-.061 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	5.780E+00	.483E+03	5.00	5.00	0.100

TR 34 95 111

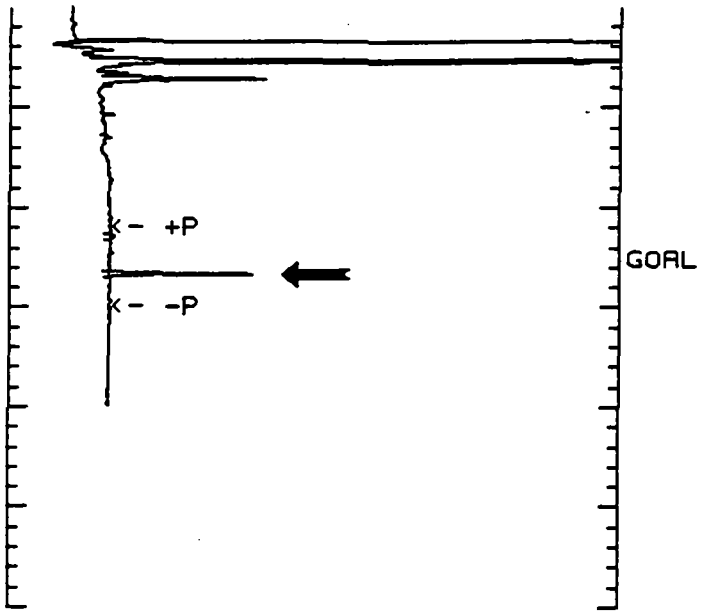
000187

Data file: L26JA14 RAR number: 94-0142
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: NA Cal. Curve: 07/28/95
Date: 28 Jul 1995 21:49 Method: GOALVm Analyst : JB
Interface: 707 Cycle#: 14 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): NA Inj Port Temp: 265
Flowrate/Gas: 3.5ml/minHE Split Ratio: NA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: NA

Plot time: 0 to 20 minutes
Plot range: 50 millivolts (-.11 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	1.440E+01	.118E+04	5.00	5.00	0.250

TR 34 95 111

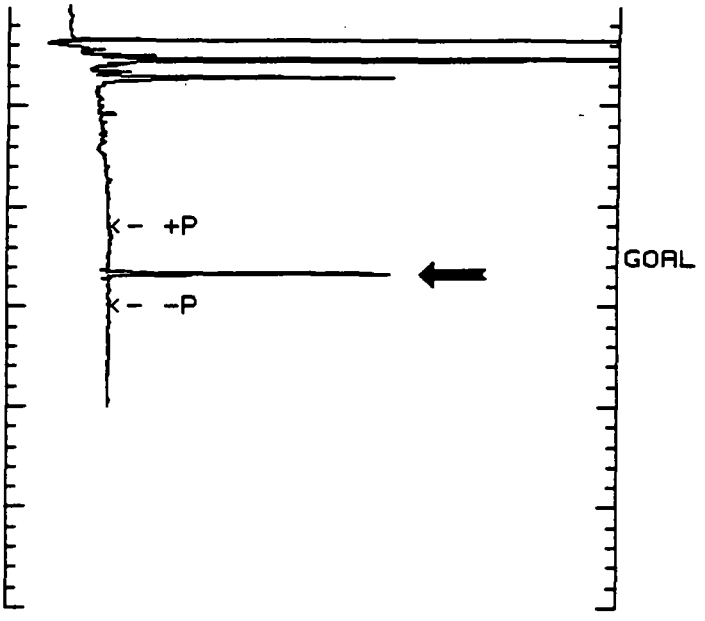
000188

Data file: L26JA15 RAR number: 94-0142
Method file: GOALVm Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: MA Cal. Curve: 07/28/95
Date: 26 Jul 1995 22:15 Method: GOALVm Analyst : JB
Interface: 707 Cycle#: 15 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): MA Inj Port Temp: 265
Flowrate/Gas: 3.8ml/mnHE Split Ratio: MA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.893 mv offset)



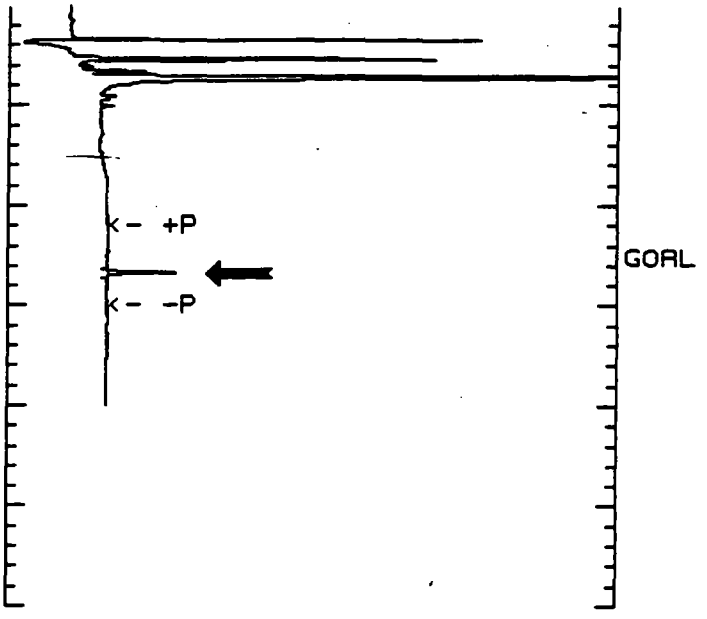
Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.37	GOAL	2.760E+01	.232E+04	5.00	5.00	0.500

Data file: L36JA16 RAR number: 96-0142
Method file: GOALVa Sample No: 001
Type: FORTIFICATION Component: FIG

Sample Name: MA Cal. Curve: 07/28/95
Date: 28 Jul 1995 22:40 Method: GOALVa Analyst : JB
Interface: 707 Cycle#: 16 Channel#: A

Instrument: VARIAN 3500,
Column: RTX-200, 0.32mm ID, 1.0um df Column Length: 60 Meters
Start Temp-Time (deg-min): 215 C Ramp Hold (deg-min): 1
Program Rate (deg/min): 10C End Time-Temp (deg-min): 250 C-17
Prog Slope (0 or Linear): MA Inj Port Temp: 265
Flowrate/Gas: 3.6ml/minHE Split Ratio: MA
Det 1-Type & Temp: ECD/300C Det 2-Type & Temp: MA

Plot time: 0 to 30 minutes
Plot range: 50 millivolts (-.15 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	Volume (ml)	Samp Wt.	ug Added
13.36	GOAL	6.850E+00	.561E+03	100	5.00	2.50

TR 34 95 111

000190

Calibration Standards
07/28/95

Concentration of GOAL	Peak Height
0.150	4580.0
0.150	4640.0
0.100	2590.0
0.100	2590.0
0.050	1380.0
0.050	1290.0
0.010	266.0
0.010	276.0
0.005	127.0
0.005	132.0

Least Squares Calibration Equation
Standard Curve Number 07/28/95

Height = 75.758 + 19035*(Ug/Ml) + 72899*(Ug/Ml)^2

94-0142 Fortifications

Sample Num.	Comp.	Peak Height	Ug/Ml GOAL Found	Final Volume (Ml)	Control Corr. (Ug)	PPM GOAL Found	PPM GOAL Added	Pct. Recov.
001	FIG	249.0	0.0088	5.00	0.00	0.0088	0.010	88.
001	FIG	246.0	0.00866	5.00	0.00	0.00866	0.010	86.6
001	FIG	241.0	0.00841	5.00	0.00	0.00841	0.010	84.1
001	FIG	234.0	0.00806	5.00	0.00	0.00806	0.010	80.6
001	FIG	499.0	0.0206	5.00	0.00	0.0206	0.020	103.
001	FIG	483.0	0.0199	5.00	0.00	0.0199	0.020	99.5
001	FIG	1180.0	0.0469	5.00	0.00	0.0469	0.050	97.8
001	FIG	2320.0	0.0881	5.00	0.00	0.0881	0.100	88.1
001	FIG	561.0	0.0234	100	0.00	0.468	0.500	93.6

94-0142 Samples

Sample Num.	Comp.	Peak Height	Ug/Ml GOAL Found	Final Volume (Ml)	Ug GOAL Found	Sample Weight (G)	Recov Fact.	PPM GOAL Found
001	FIG	0.0	0.00	5.00	0.00	5.00	1.00	0.00

TR 34 95 111

000191

GC/MS RUN SHEET

Date 11/9/95
Analyst Shaozhi zheng
Instrument GC/MS #4
Analytical Method TR34-94-150
Notebook Ref. 002-165
Crop fruit (Grape, peach)

Compound Goal
Data Reduction Method NA
Raw Data File N085
Date Extracted 11-8-95
Date Injected 11/9-10/95
SZ 11/18/95

The preceding tune(s) apply to the following analysis

Inj. No	STD (µg/mL)	RAR No.	Spl. No	Sample Component	Sample Weight (g)	Final Volume (mL)	Fortification Level (µg)	Notes
1 **	0.5							
2	0.1							
3	0.05							
4	0.025							
5		94-0117	001	fruit	5	2.5		BLK
6		↓	↓	↓	↓	↓	0.125	SPKA
7							0.25	SPKB
8							0.625	SPKC
9		92-0100	005					BLK
10		↓	↓				0.125	SPKA
11							0.25	SPKB
12							0.625	SPKC
13	WASH							
14	0.025							
15	0.05							
16	0.1							
17	0.5							
18								
19								
20								
21					SZ			
22				11/9/95				
23								
24								
25								
26								

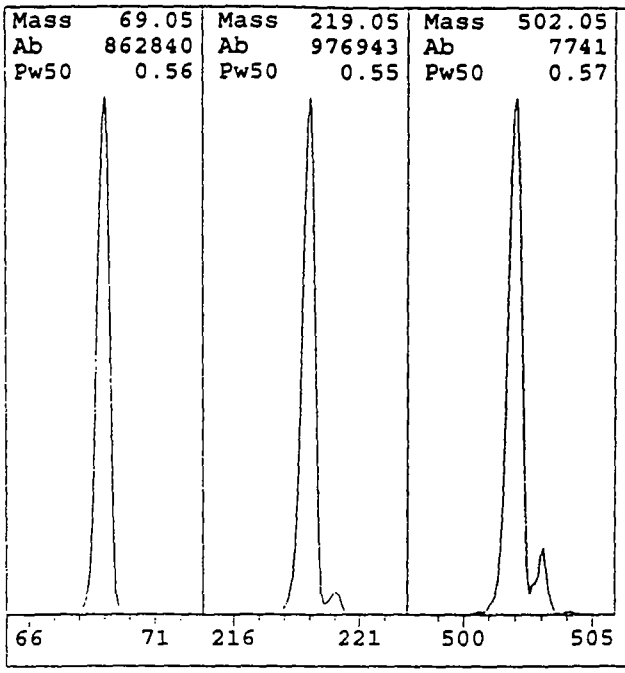
NOTES: Samples were stored in freezer #10A before injected.
** it is not included in the standard curve. SZ 12/18/95
94-0117 (peach)
92-0100 (grape) Dennis J. Martin 3/19/96 12/18/95

Signature Shaozhi zheng

C00192

TR 34 95 111

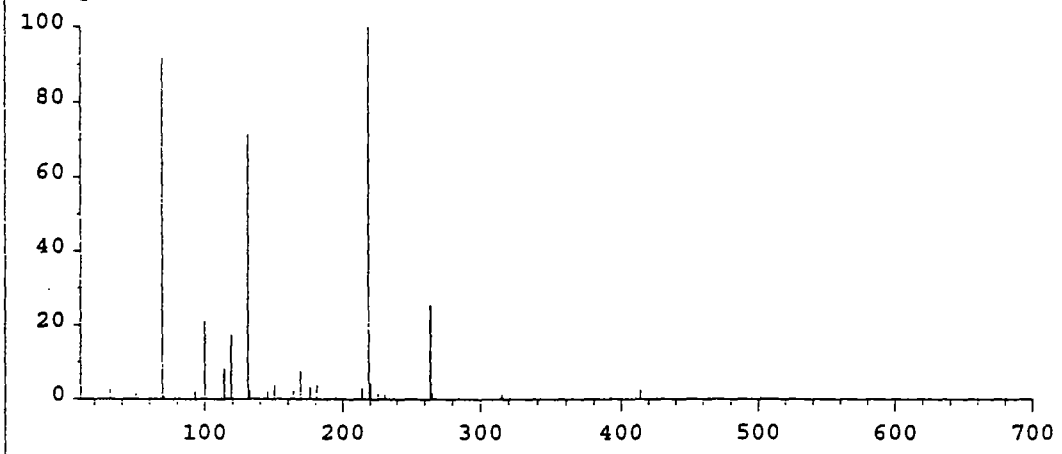
Thu Nov 09 10:41:22 1995 C:\HPCHEM\1\5972\TARGET.U



EMVolts	1965	AmuGain	544
Xray	34.2	AmuOffs	91
Emission	50.0	219Wid	0.001
MS Temp	175	TTI	OFF
Vacuum	110	DC Pol	NEG
Samples	8	Repeller	29.90
Averages	3	IonFocus	74.0
StepSize	0.10	EntLens	0.00
MassGain	142	EntOffs	VAR
MassOffs	13	Filament	2

PFTBA OPEN

Scan: 10.00 - 700.00 Samples: 8 Thresh: 100 Step: 0.10
300 peaks Base: 218.95 Abundance: 840448



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	773760	100.00	70.00	8631	1.12
218.95	840448	108.62	219.95	37408	4.45
501.95	6749	0.87	502.95	737	10.92

TARGET MASS:	69	131	219	502
DYNAMIC ENT OFFSET:	7.0	10.3	14.6	10.3
TARGET ABUND(%):	100.0	74.0	115.0	0.9
ACTUAL TUNE ABUND(%):	100.0	77.6	108.6	0.9

Analytical Data Report for GC-MS Confirmation of Goal in Grape and Peach

Protocol No.:	34P-95-92	CAL Study No.:	002-165
Matrix:	Grape and Peach	Analyte:	Goal
Extraction Date:	11/8/95	Analysis Date:	11/9-10/95

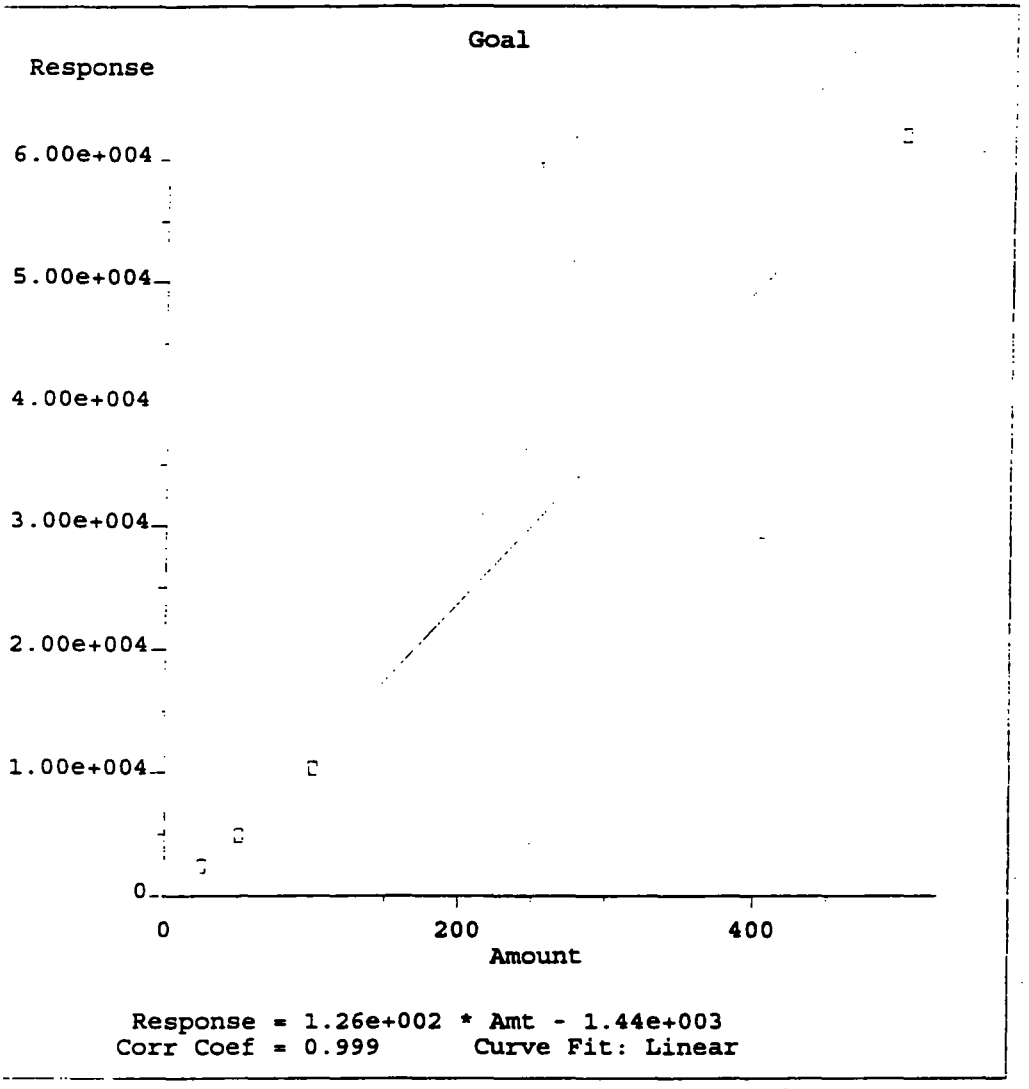
12/18/95

Peak height	µg/ml Std Injected	ng/ml Std Injected
2400	0.025	25
2465	0.025	25
4496	0.050	50
5254	0.050	50
9344	0.100	100
11274	0.100	100
61774	0.500	500

F(x) = 126 * Amt - 1440
Corr Coef = 0.999

R & H Sample ID	CAL No.	Final Vol. ml	Dilution Factor	Sample Wt. (g)	Peak Area	ng/ml Found	µg Found	ppm Found	ppm Added	% Recovery
94-0117-001*	952309 Blk	2.5	1	5.0	0	0.00	0.000	0.000	NA	NA
94-0117-001	952309 Spk A	2.5	1	5.0	3890	42.26	0.106	0.021	0.025	85
94-0117-001	952309 Spk B	2.5	1	5.0	8952	82.39	0.206	0.041	0.050	82
94-0117-001	952309 Spk C	2.5	1	5.0	30752	255.18	0.638	0.128	0.125	102
92-0100-005*	954668 Blk	2.5	1	5.0	0	0.00	0.000	0.000	NA	NA
92-0100-005	954668 Spk A	2.5	1	5.0	4397	46.28	0.116	0.023	0.025	93
92-0100-005	954668 Spk B	2.5	1	5.0	9405	85.98	0.215	0.043	0.050	86
92-0100-005	954668 Spk C	2.5	1	5.0	32497	269.02	0.673	0.135	0.125	108

* 94-0117 (peach)
92-0100 (grape) Dennis A. Martin 3/14/96

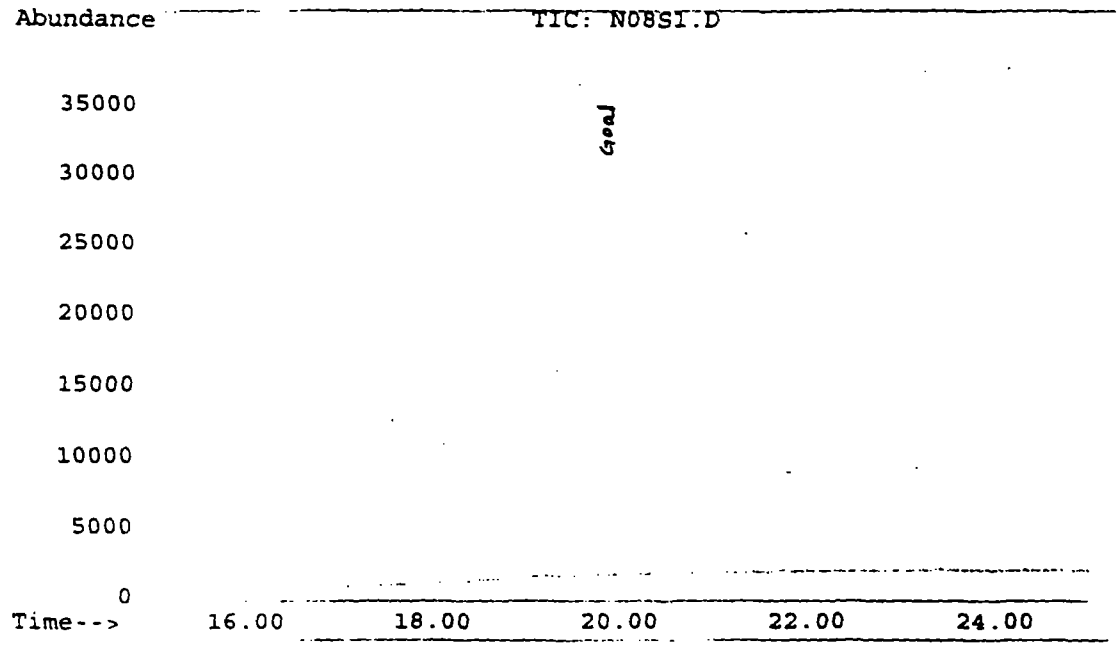


Method Name: C:\HPCHEM\1\METHODS\GOAL1.M
Calibration Table Last Updated: Fri Nov 10 08:35:09 1995

TR 34 95 111

000195

File : C:\HPCHEM\1\DATA\N08S1.D
Operator : sz
Acquired : 9 Nov 95 5:35 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 1



GC-MSD INSTRUMENTATION

a. Instrumentation: Hewlett-Packard model 5890 Series II Gas Chromatograph/model 5972 mass selective detector
b. Column: Rtx-50, (0.25 mm i.d. x 30 m. 0.5 µm film thickness
c. Oven Temperature: Hold at 100°C for 2 min., then 100 to 270°C at 10°C/min., then hold 15 min.
d. Injector Temperature: 280°C
e. Detector Temperature: 300°C
f. Carrier Gas: Helium
g. Carrier Gas Flow Rate: 2.18 ml/min.
h. Head Pressure: 20 Psi
i. Injection Mode: Splitless
j. Injection Liner: Silanized Single Taper
k. Injector Purge Delay: 1.0 min.
l. Septum Purge: 2 ml/min.
m. Injection Volume: 2 µl
n. Ionization Potential: 70 eV
o. Electron Multiplier Voltage: 1900 to 2318 V
p. Dwell Time: 100 msec

Initials SZ
Date 11/17/95
Run # N08S1 to N08S17

Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S1.D
Operator : sz
Acquired : 9 Nov 95 5:35 pm using AcqMethod GOAL1
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 1
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

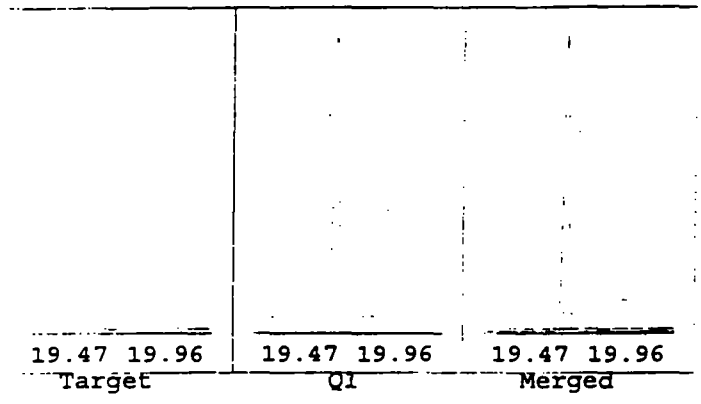
Time Reference Peaks:

Compound	Expected RT	Actual RT

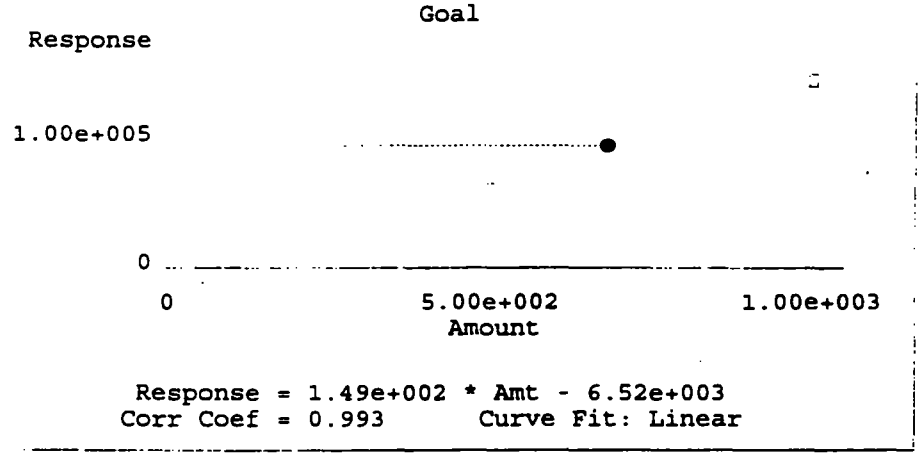
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S1.D
Operator : sz
Acquired : 9 Nov 95 5:35 pm using AcqMethod GOAL1
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 1
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 682.50
Pk # and Type: 1 RTEINT used



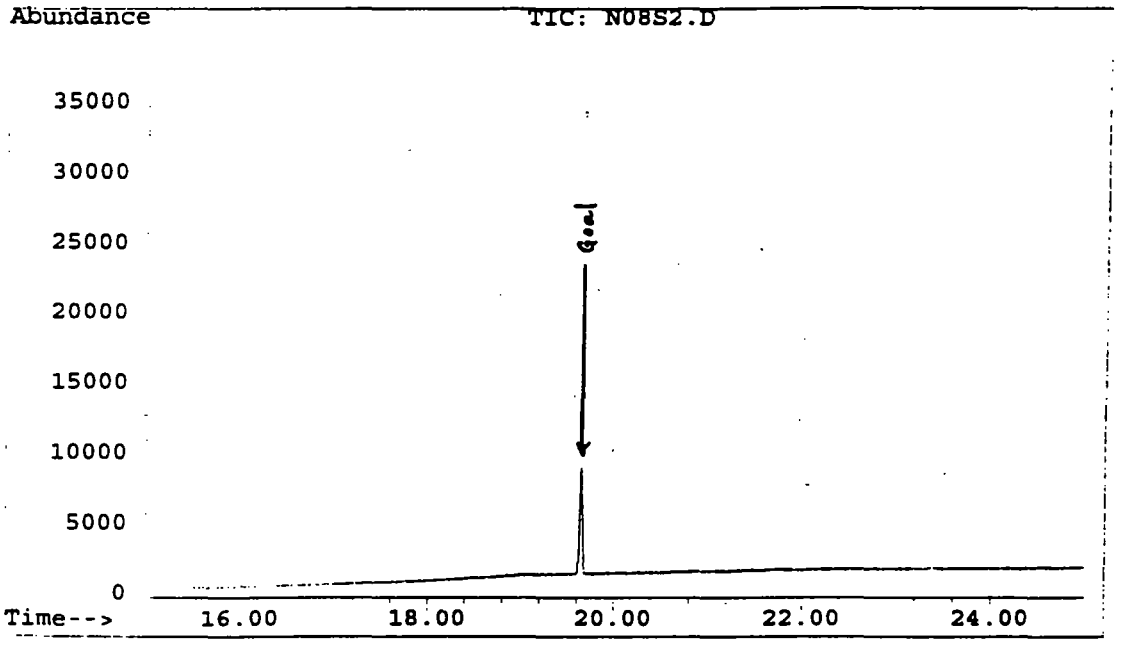
	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.22	95162	sys def
Q1	361.00	11.1	0.0-	22.0	19.65	to	10593	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def



TR 34 95 111

000198

File : C:\HPCHEM\1\DATA\N08S2.D
Operator : sz
Acquired : 9 Nov 95 6:13 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 2



Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S2.D
Operator : sz
Acquired : 9 Nov 95 6:13 pm using AcqMethod GOAL1
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 2
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

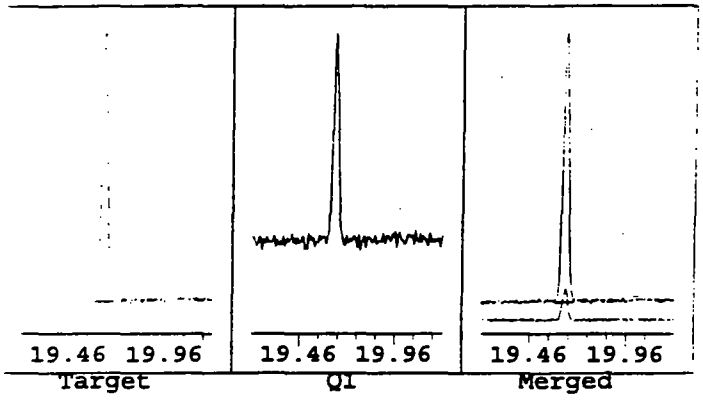
Number of Compounds in Database: 1

Time Reference Peaks:

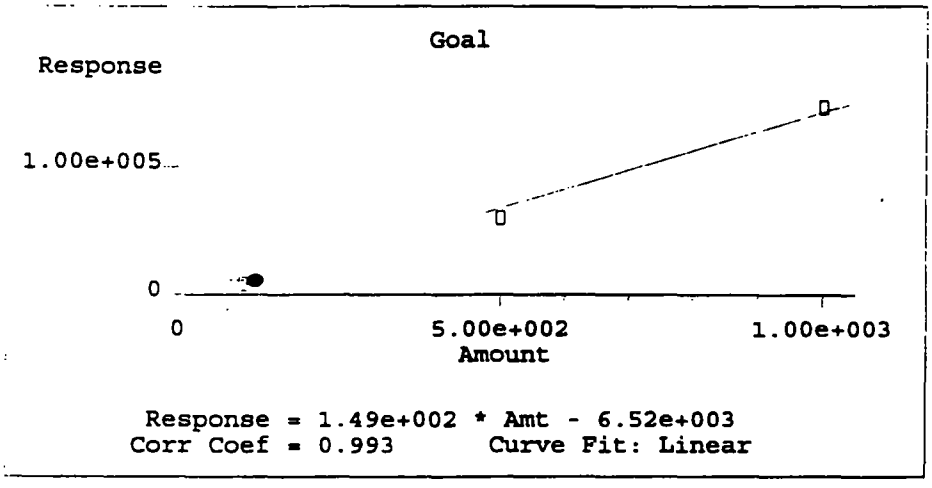
Compound	Expected RT	Actual RT

File : C:\HPCHEM\1\DATA\N08S2.D
Operator : sz
Acquired : 9 Nov 95 6:13 pm using AcqMethod GOAL1
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 2
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 119.45
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.22	11274	sys def
Q1	361.00	11.5	0.0-	22.0	19.65	to	1294	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

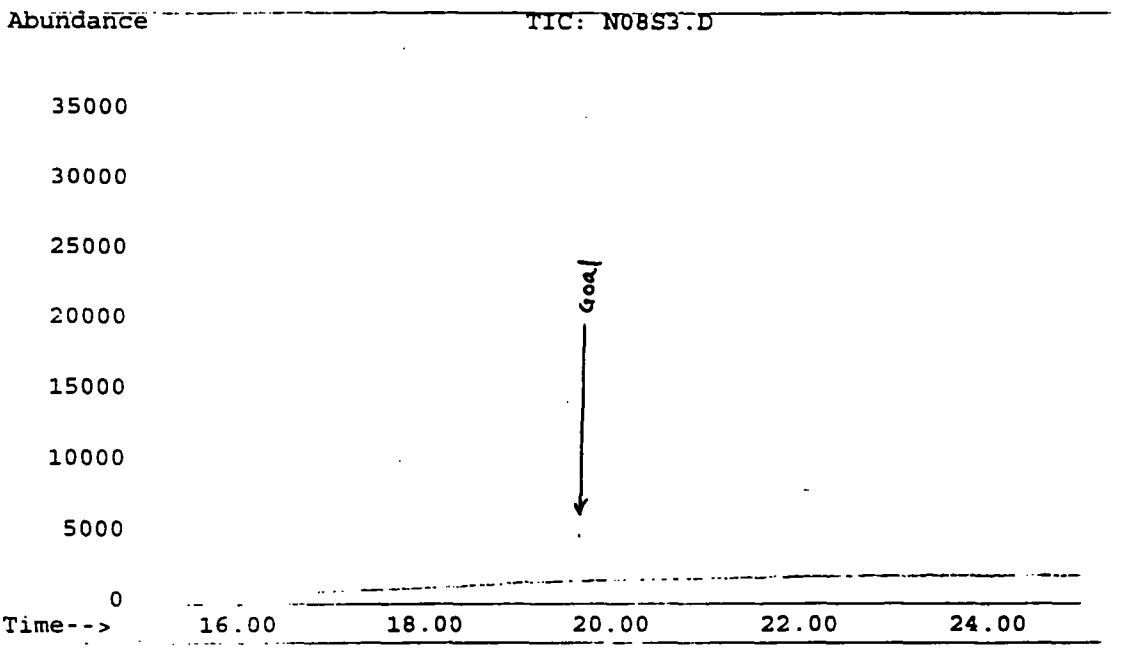


Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

TR 34 95 111

000201

File : C:\HPCHEM\1\DATA\N08S3.D
Operator : sz
Acquired : 9 Nov 95 6:51 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 3



Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S3.D
Operator : sz
Acquired : 9 Nov 95 6:51 pm using AcqMethod GOAL1
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 3
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

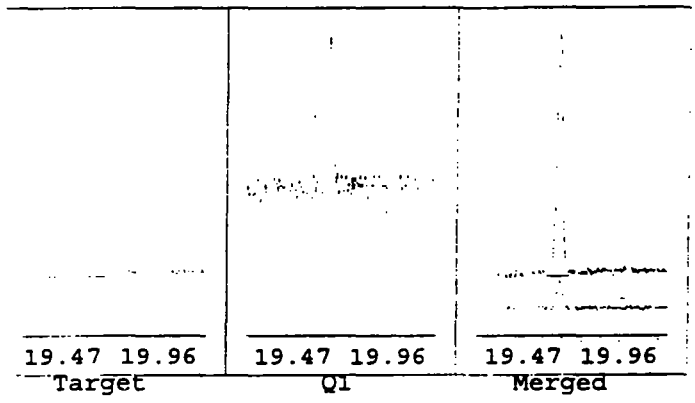
Time Reference Peaks:

Compound	Expected RT	Actual RT

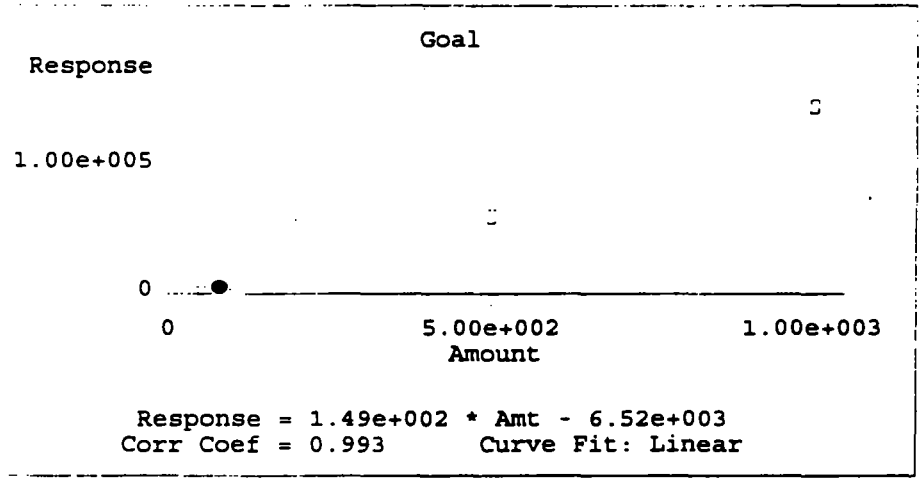
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S3.D
Operator : sz
Acquired : 9 Nov 95 6:51 pm using AcqMethod GOAL1
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 3
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 79.04
Pk # and Type: 1 RTEINT used



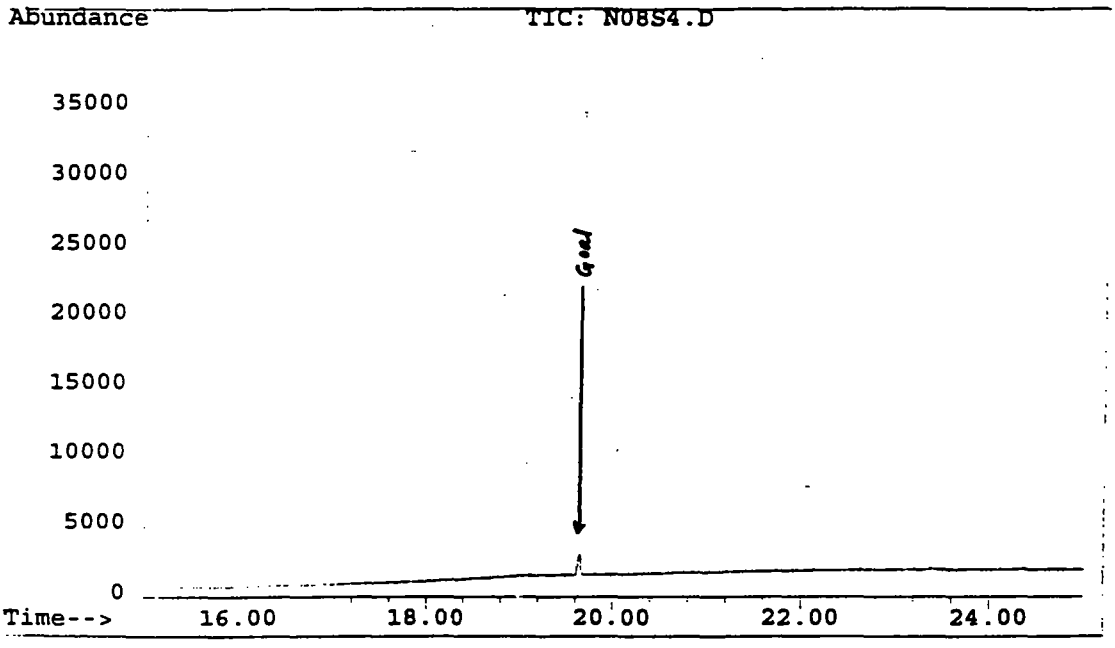
	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.64	19.22	5254	sys def
Q1	361.00	12.6	0.0- 22.0	19.65	to	662	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def



TR 34 95 111

000204

File : C:\HPCHEM\1\DATA\N08S4.D
Operator : sz
Acquired : 9 Nov 95 7:30 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 4



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S14.D
Operator : sz
Acquired : 10 Nov 95 1:52 am using AcqMethod GOAL1
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 14
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

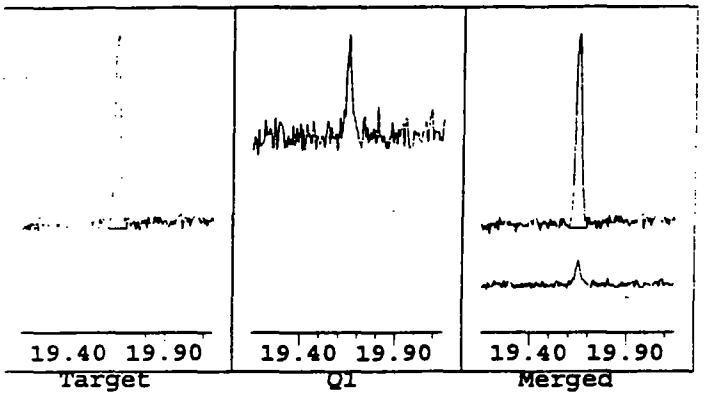
Time Reference Peaks:

Compound	Expected RT	Actual RT

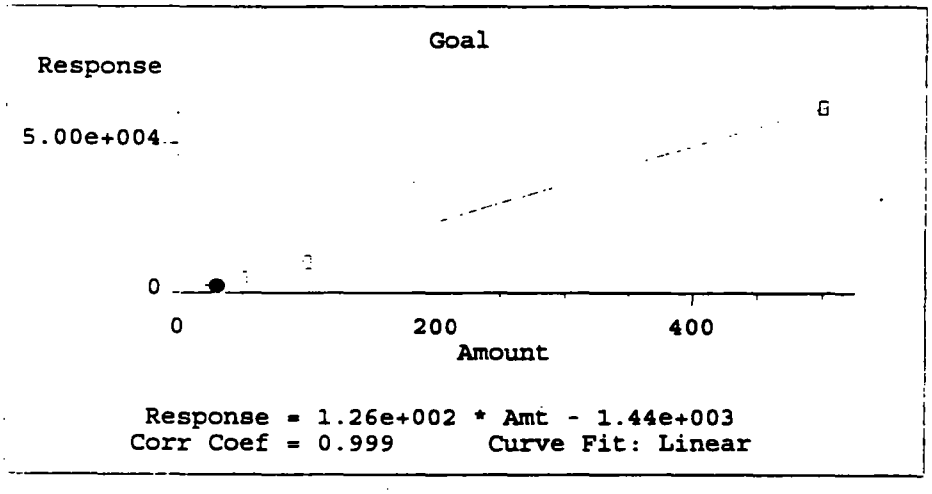
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S4.D
Operator : sz
Acquired : 9 Nov 95 7:30 pm using AcqMethod GOAL1
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 4
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 30.45
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.65	19.16	2400	sys def
Q1	361.00	12.6	0.0- 22.0	19.65	to	303	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

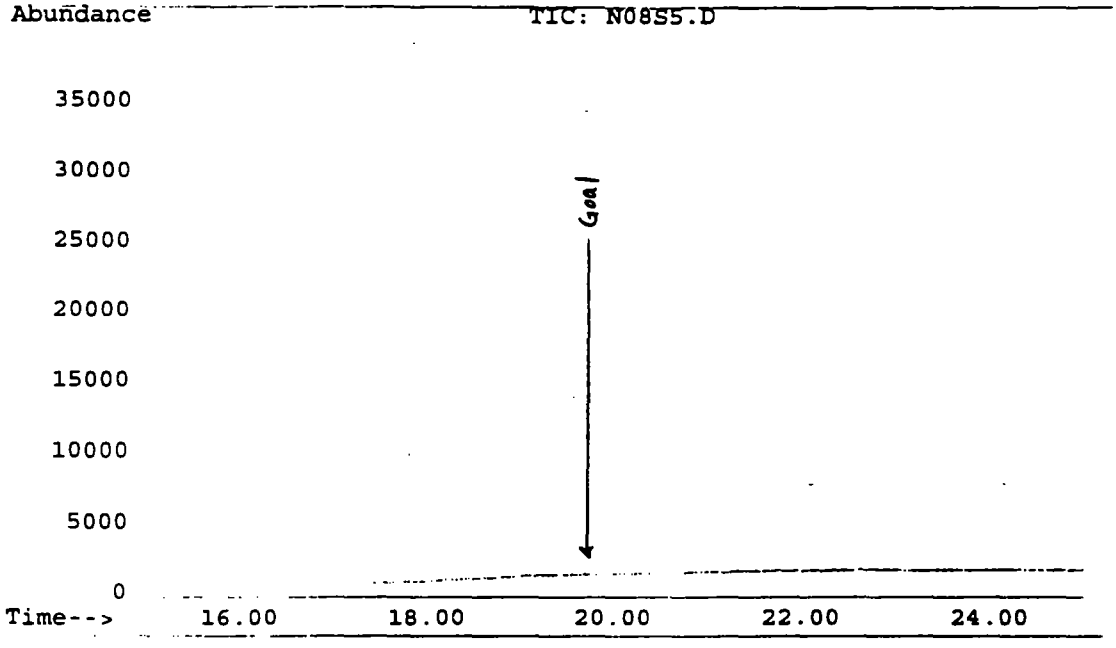


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000207

File : C:\HPCHEM\1\DATA\N08S5.D
Operator : sz
Acquired : 9 Nov 95 8:08 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 94-0117-001
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 5



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S5.D
Operator : sz
Acquired : 9 Nov 95 8:08 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 5
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

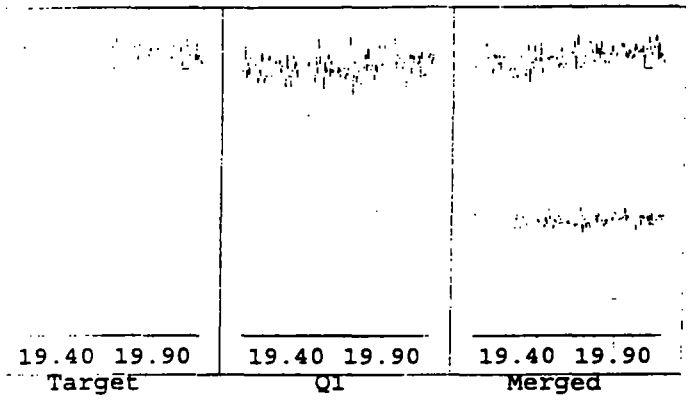
Time Reference Peaks:

Compound	Expected RT	Actual RT

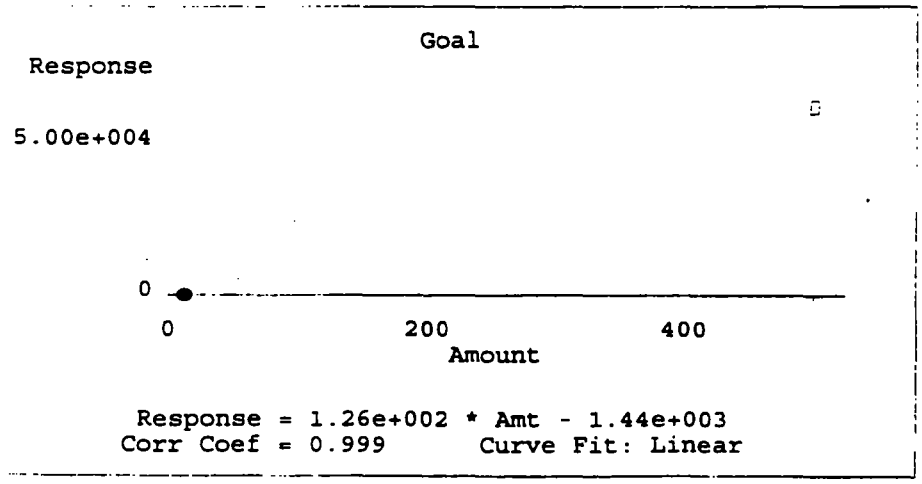
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S5.D
Operator : sz
Acquired : 9 Nov 95 8:08 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 5
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 20.06
Concentration: 12.30
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		20.06	19.16	110	sys def
Q1	361.00	0.0	0.0- 22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

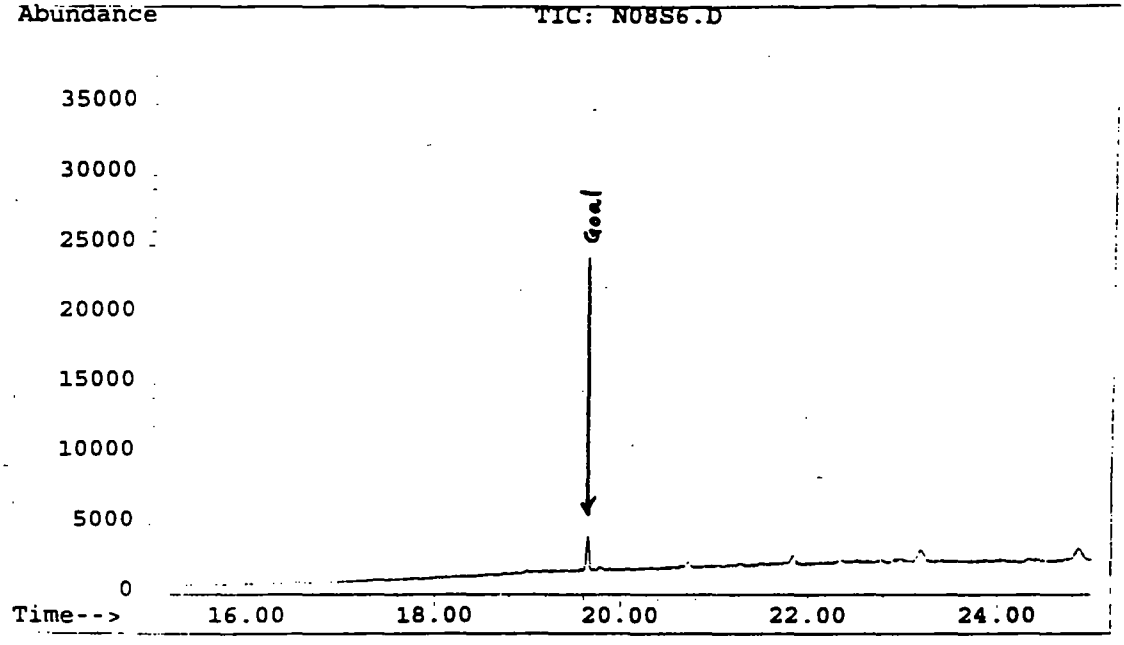


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000210

File : C:\HPCHEM\1\DATA\N08S6.D
Operator : sz
Acquired : 9 Nov 95 8:46 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 94-0117-001
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 6



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S6.D
Operator : sz
Acquired : 9 Nov 95 8:46 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 6
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

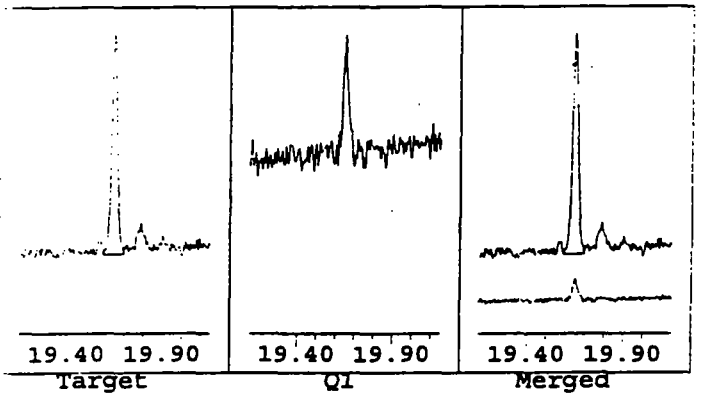
Compound	Expected RT	Actual RT

TR 34 95 111

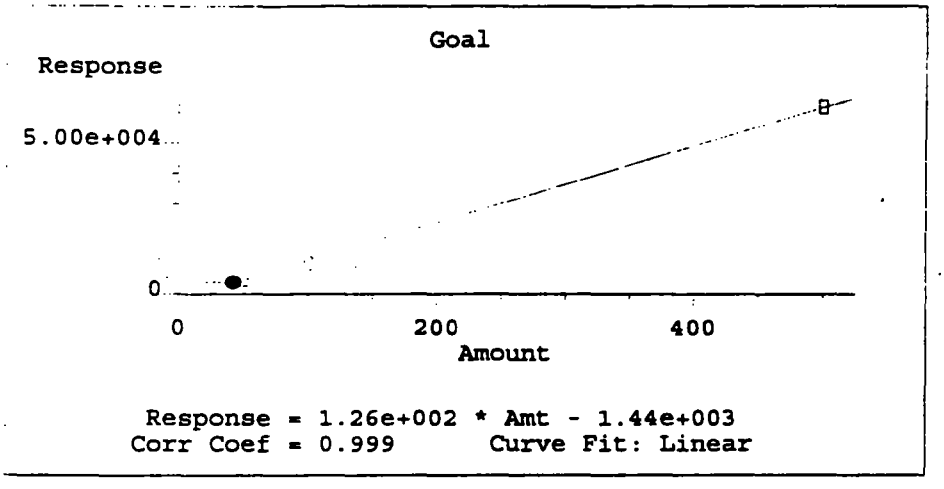
000212

File : C:\HPCHEM\1\DATA\N08S6.D
Operator : sz
Acquired : 9 Nov 95 8:46 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 6
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 42.26
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	3890	sys def
Q1	361.00	10.5	0.0-	22.0	19.65	to	410	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

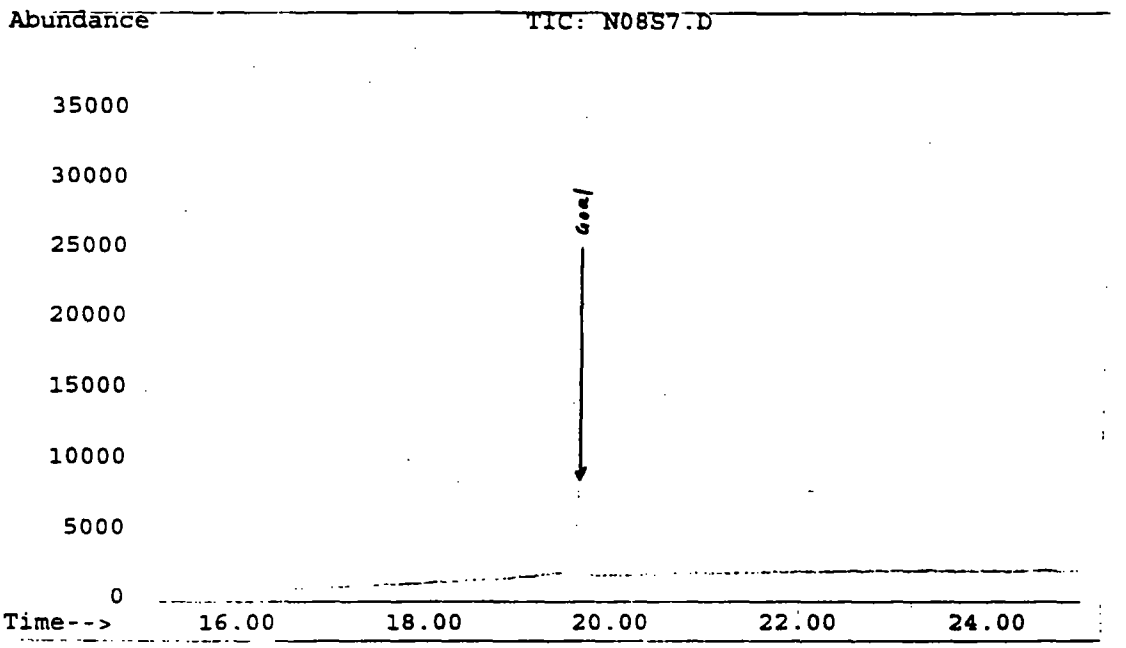


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000213

File : C:\HPCHEM\1\DATA\N08S7.D
Operator : sz
Acquired : 9 Nov 95 9:24 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 94-0117-001
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 7



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S7.D
Operator : sz
Acquired : 9 Nov 95 9:24 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 7
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

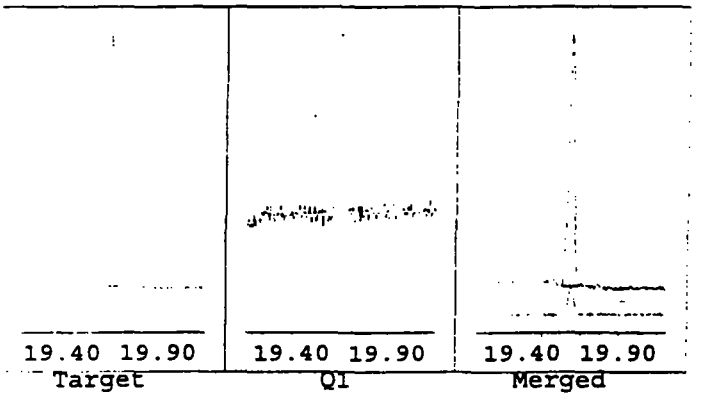
Time Reference Peaks:

Compound	Expected RT	Actual RT

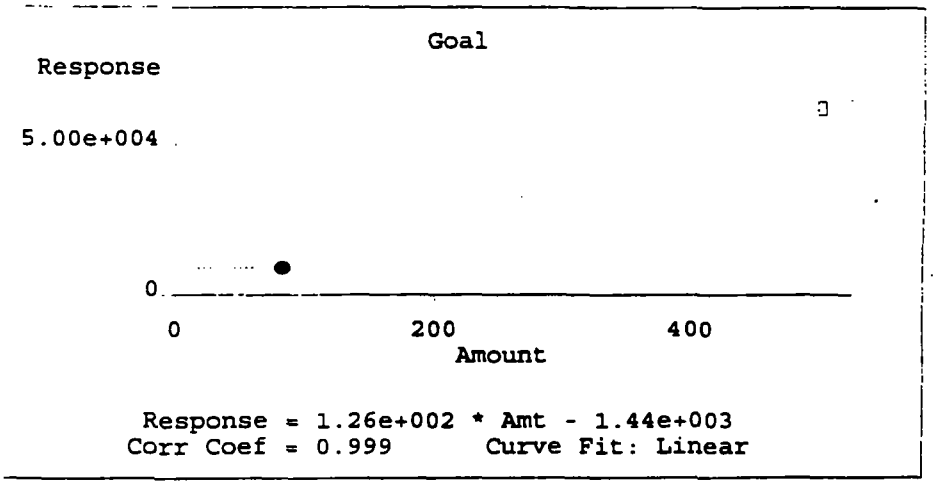
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S7.D
Operator : sz
Acquired : 9 Nov 95 9:24 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 7
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 82.39
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	8952	sys def
Q1	361.00	12.4	0.0-	22.0	19.65	to	1110	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

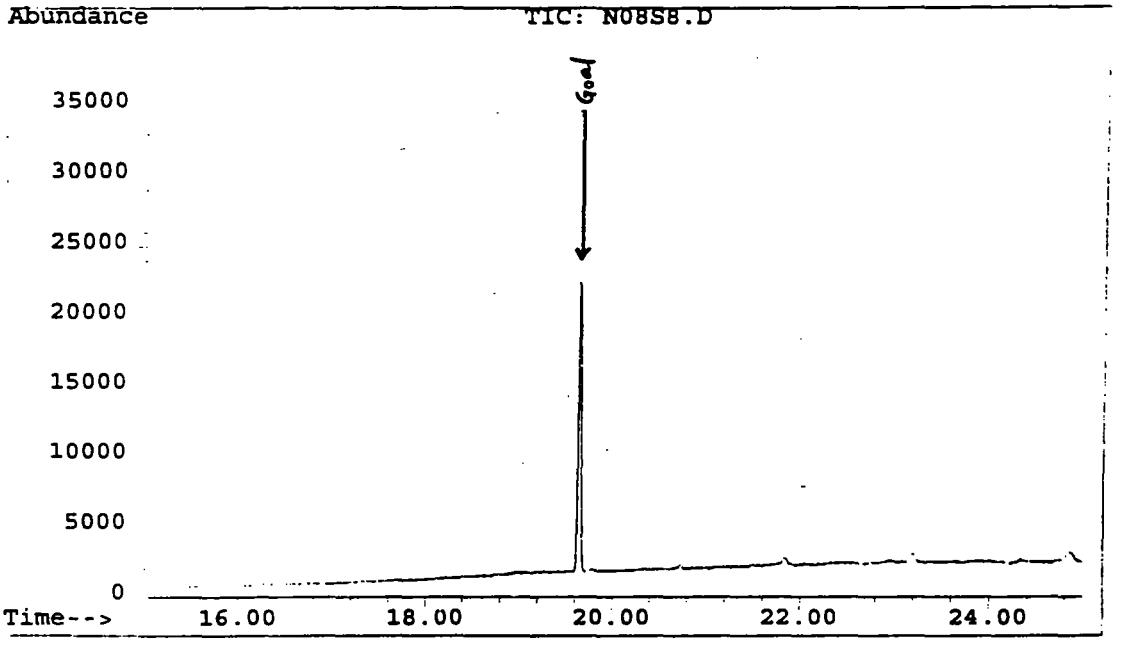


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000216

File : C:\HPCHEM\1\DATA\N08S8.D
Operator : sz
Acquired : 9 Nov 95 10:03 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 94-0117-001
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 8



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S8.D
Operator : sz
Acquired : 9 Nov 95 10:03 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 8
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

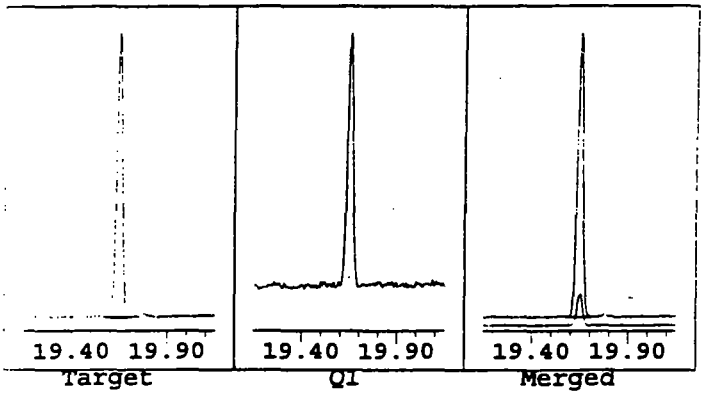
Compound	Expected RT	Actual RT

TR 34 95 11

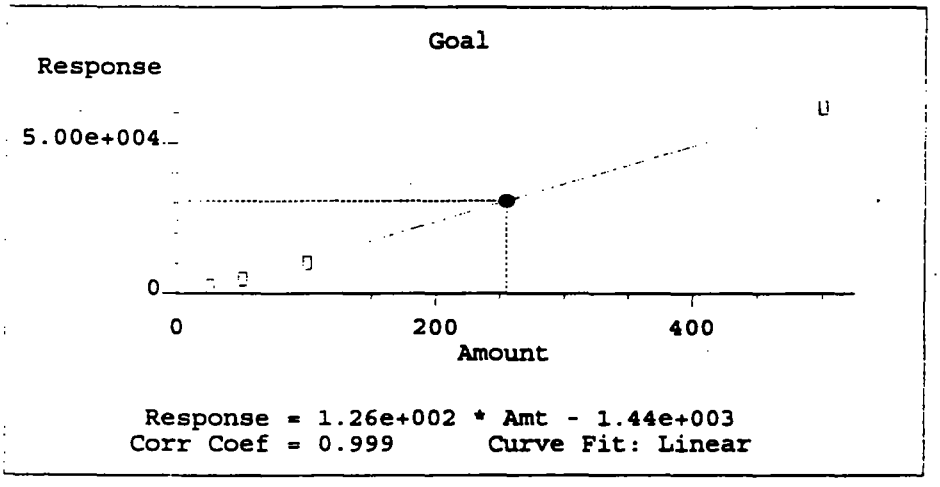
000218

File : C:\HPCHEM\1\DATA\N08S8.D
Operator : sz
Acquired : 9 Nov 95 10:03 pm using AcqMethod GOAL1
Sample Name: 94-0117-001
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 8
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 255.18
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.64	19.16	30752	sys def
Q1	361.00	11.3	0.0- 22.0	19.65	to	3485	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

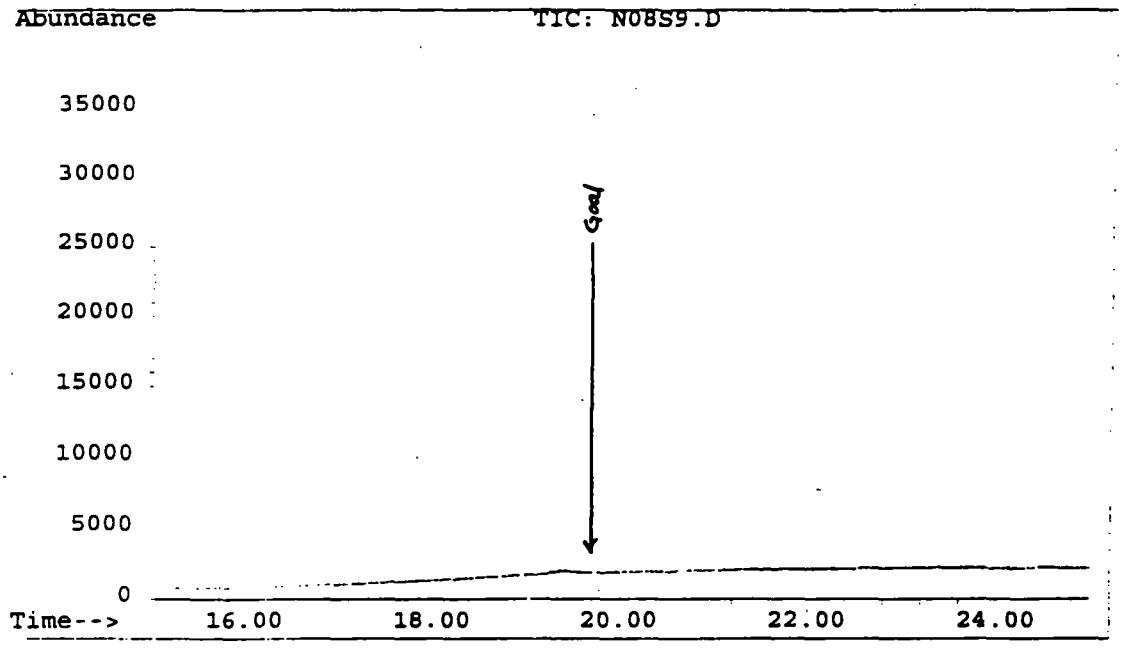


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000219

File : C:\HPCHEM\1\DATA\N08S9.D
Operator : sz
Acquired : 9 Nov 95 10:41 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 92-0100-005
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 9



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S9.D
Operator : sz
Acquired : 9 Nov 95 10:41 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 9
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

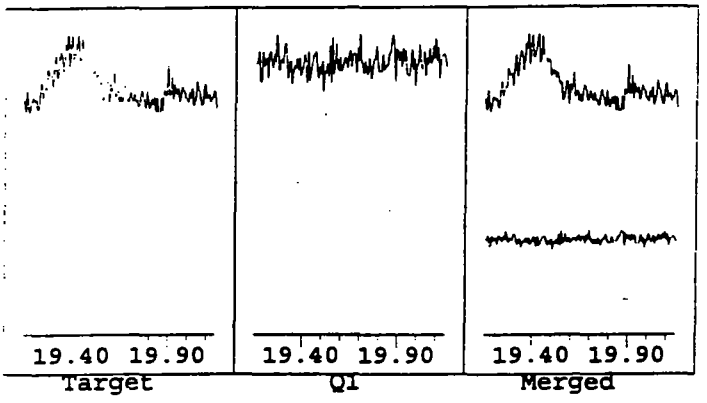
Compound	Expected RT	Actual RT

TR 34 95 111

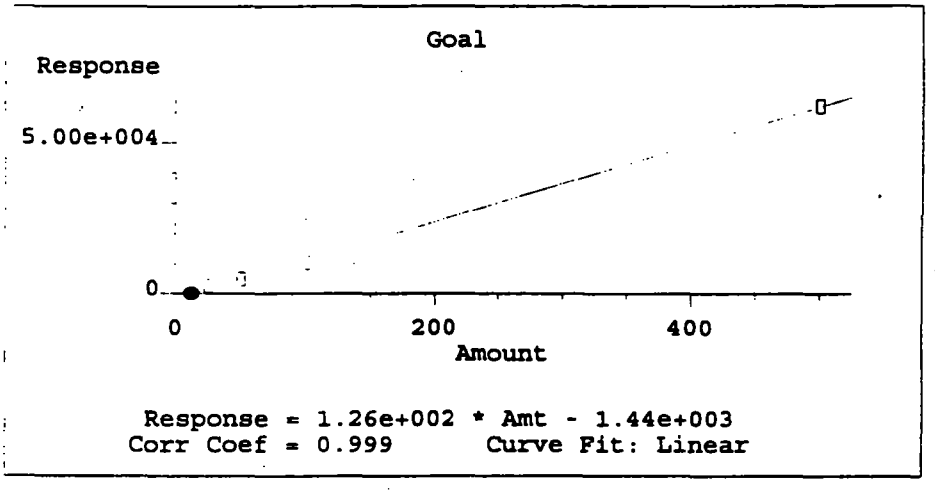
000221

File : C:\HPCHEM\1\DATA\N08S9.D
Operator : sz
Acquired : 9 Nov 95 10:41 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : blk SW=5g FV=2.5ml
Vial Number: 9
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.37
Concentration: 12.33
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.37	19.16	114	sys def
Q1	361.00	0.0	0.0- 22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

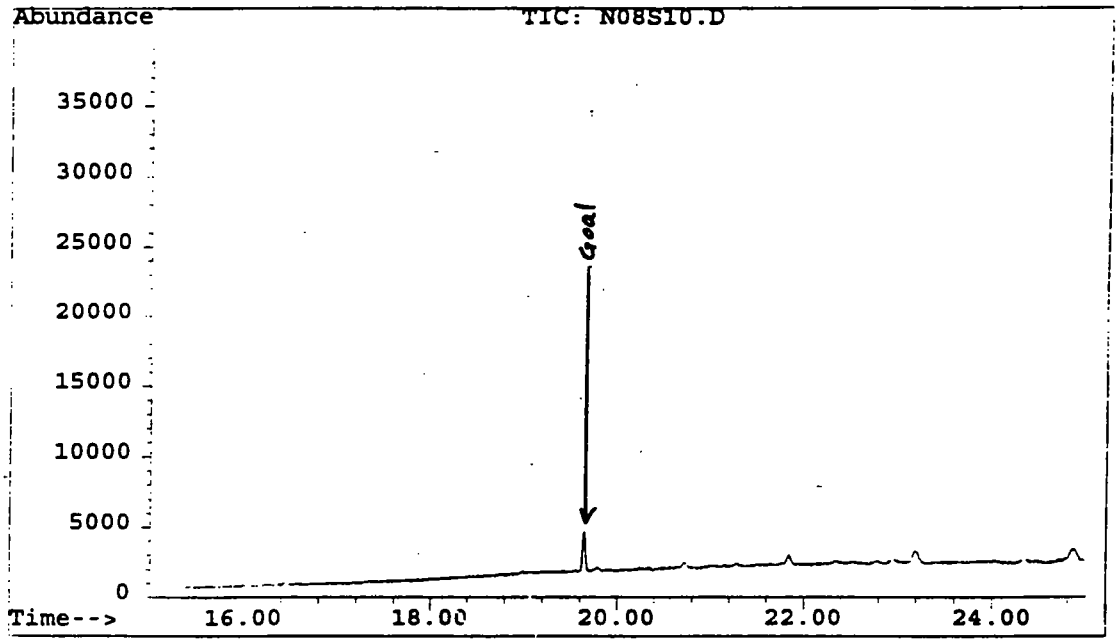


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000222

File : C:\HPCHEM\1\DATA\N08S10.D
Operator : sz
Acquired : 9 Nov 95 11:19 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 92-0100-005
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 10



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S10.D
Operator : sz
Acquired : 9 Nov 95 11:19 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 10
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

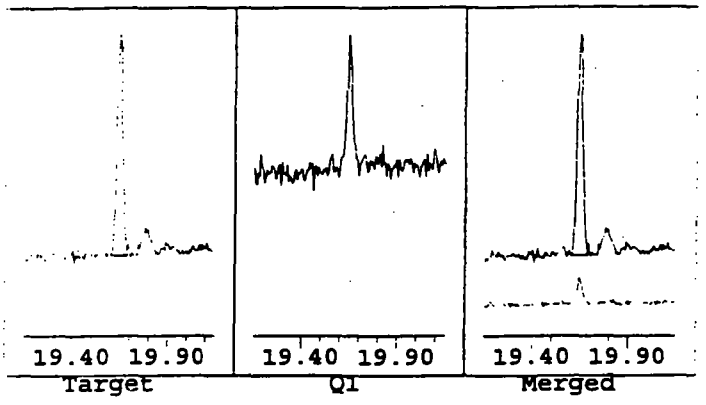
Number of Compounds in Database: 1

Time Reference Peaks:

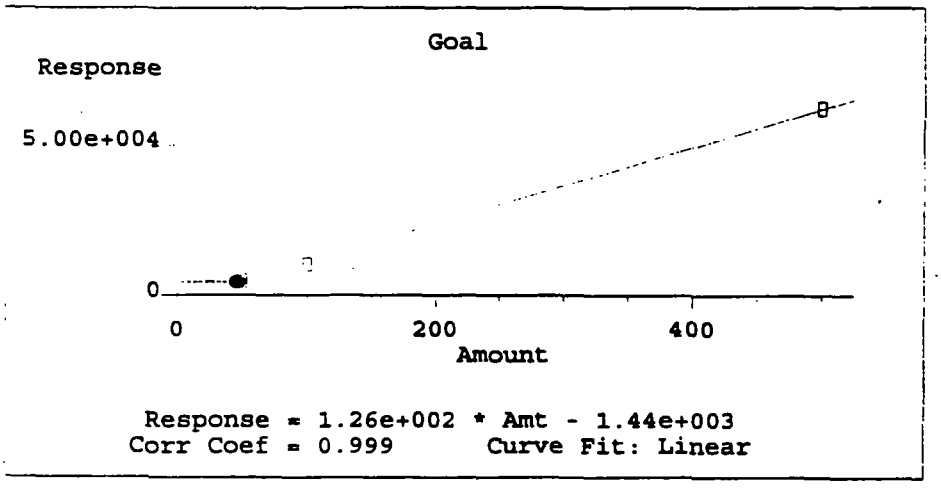
Compound	Expected RT	Actual RT

File : C:\HPCHEM\1\DATA\N08S10.D
Operator : sz
Acquired : 9 Nov 95 11:19 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spka SW=5g FV=2.5ml 0.025ppm
Vial Number: 10
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 46.28
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	4397	sys def
Q1	361.00	13.5	0.0-	22.0	19.64	to	592	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

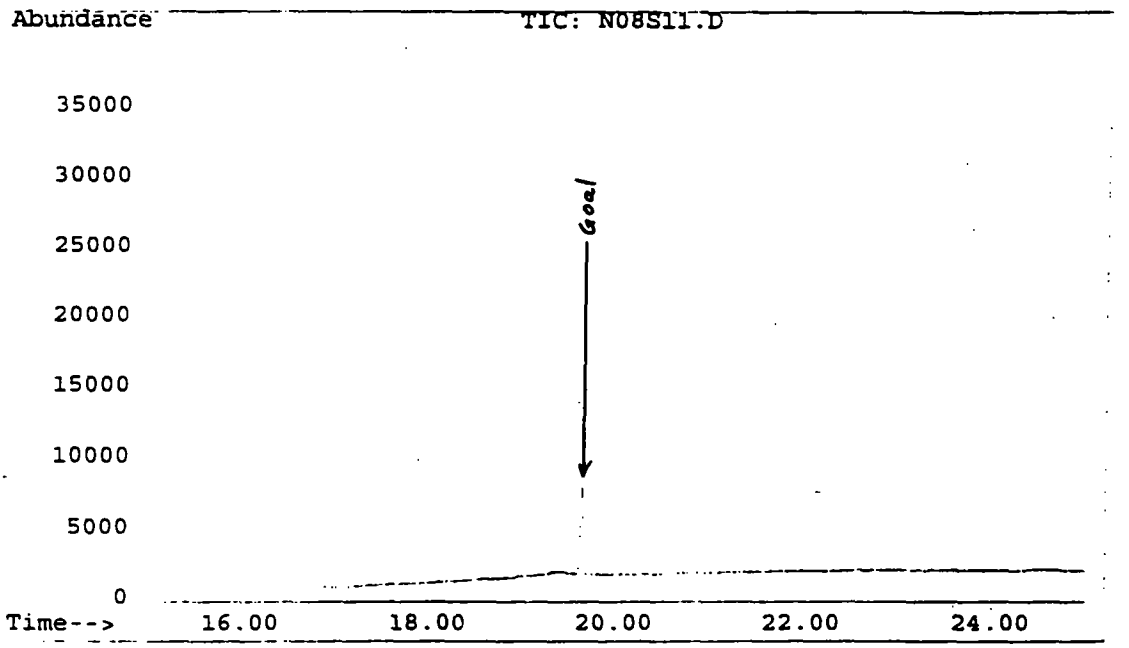


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000225

File : C:\HPCHEM\1\DATA\N08S11.D
Operator : sz
Acquired : 9 Nov 95 11:57 pm using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 92-0100-005
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 11



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S11.D
Operator : sz
Acquired : 9 Nov 95 11:57 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 11
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

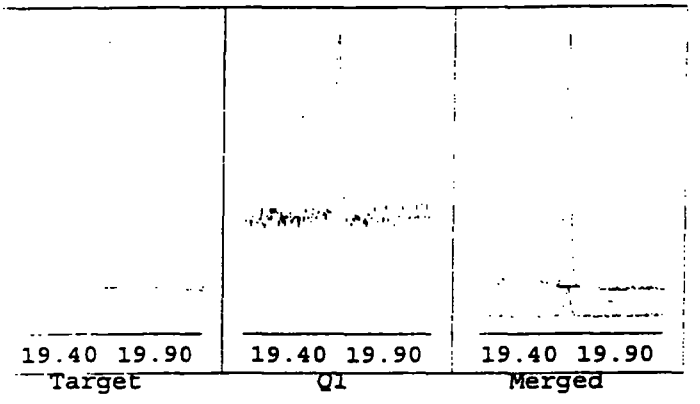
Compound	Expected RT	Actual RT

TR 34 95 111

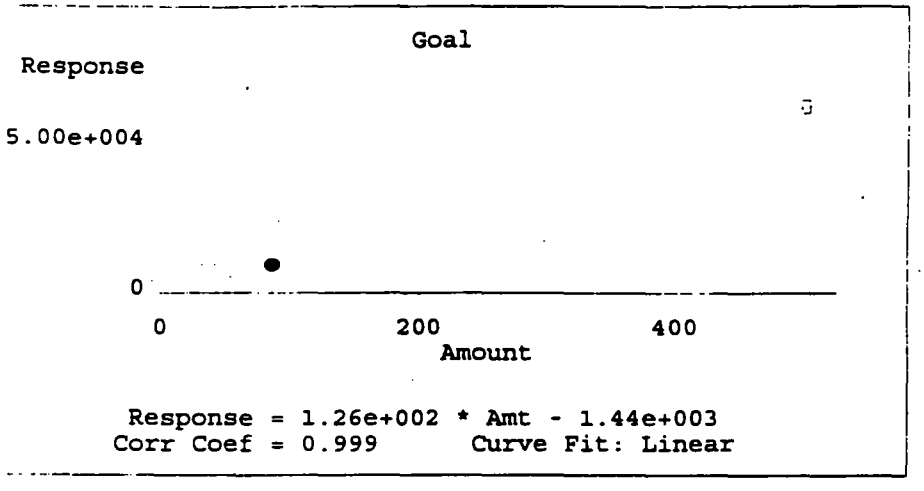
000227

File : C:\HPCHEM\1\DATA\N08S11.D
Operator : sz
Acquired : 9 Nov 95 11:57 pm using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkb SW=5g FV=2.5ml 0.05ppm
Vial Number: 11
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 85.98
Pk # and Type: 1 RTEINT used



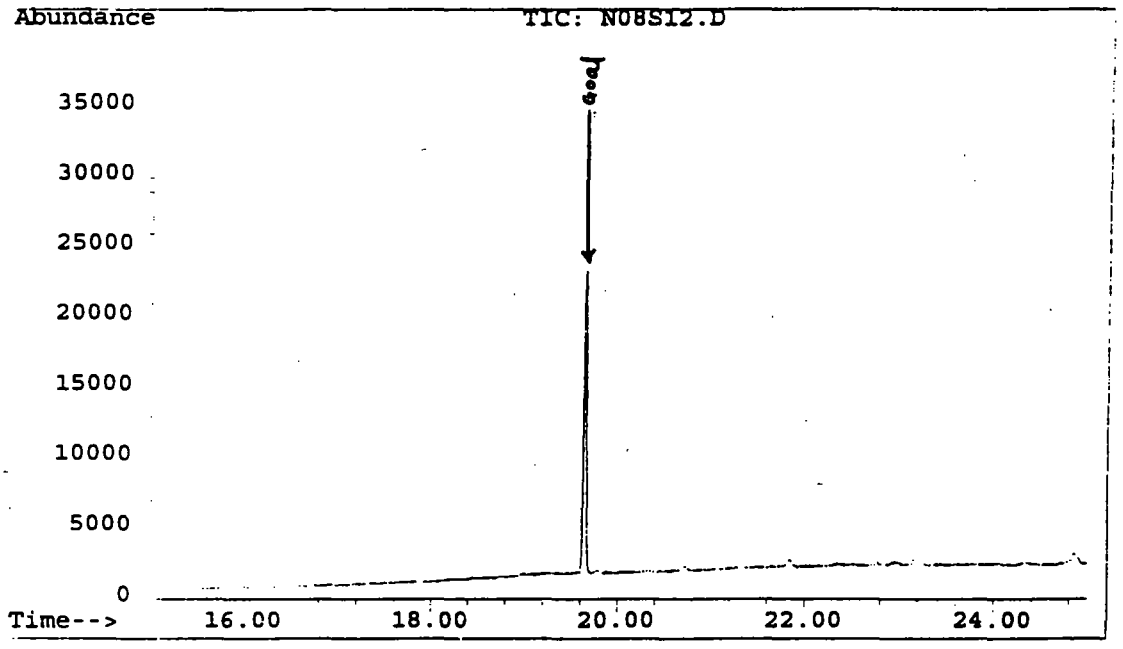
	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	9405	sys def
Q1	361.00	11.5	0.0-	22.0	19.65	to	1084	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def



TR 34 95 111

000228

File : C:\HPCHEM\1\DATA\N08S12.D
Operator : sz
Acquired : 10 Nov 95 12:35 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: 92-0100-005
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 12



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S12.D
Operator : sz
Acquired : 10 Nov 95 12:35 am using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 12
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

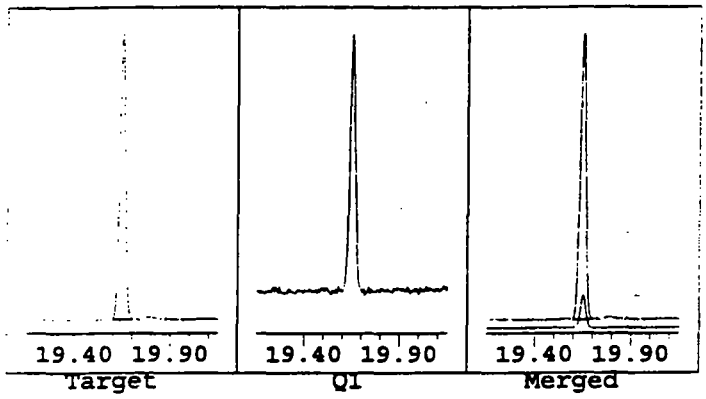
Time Reference Peaks:

Compound	Expected RT	Actual RT

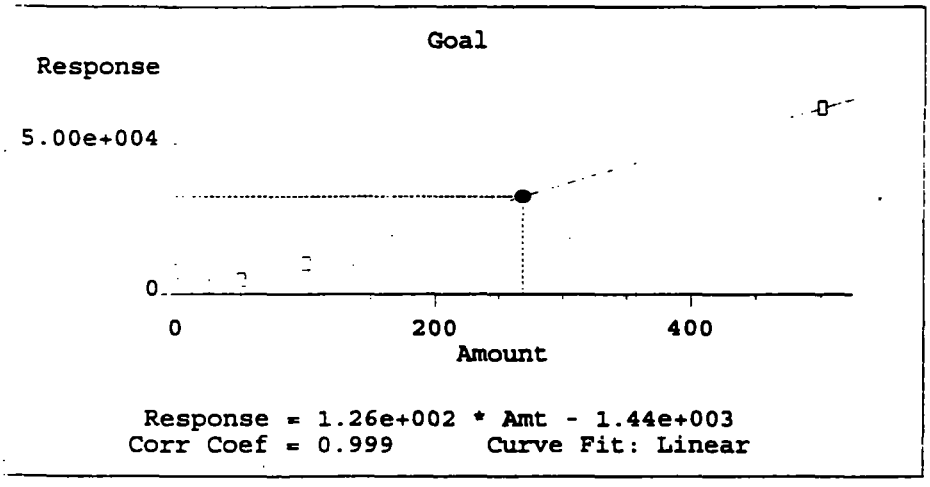
TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S12.D
Operator : sz
Acquired : 10 Nov 95 12:35 am using AcqMethod GOAL1
Sample Name: 92-0100-005
Misc Info : spkc SW=5g FV=2.5ml 0.125ppm
Vial Number: 12
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 269.02
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.65	19.16	32497	sys def
Q1	361.00	11.4	0.0-	22.0	19.65	to	3689	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

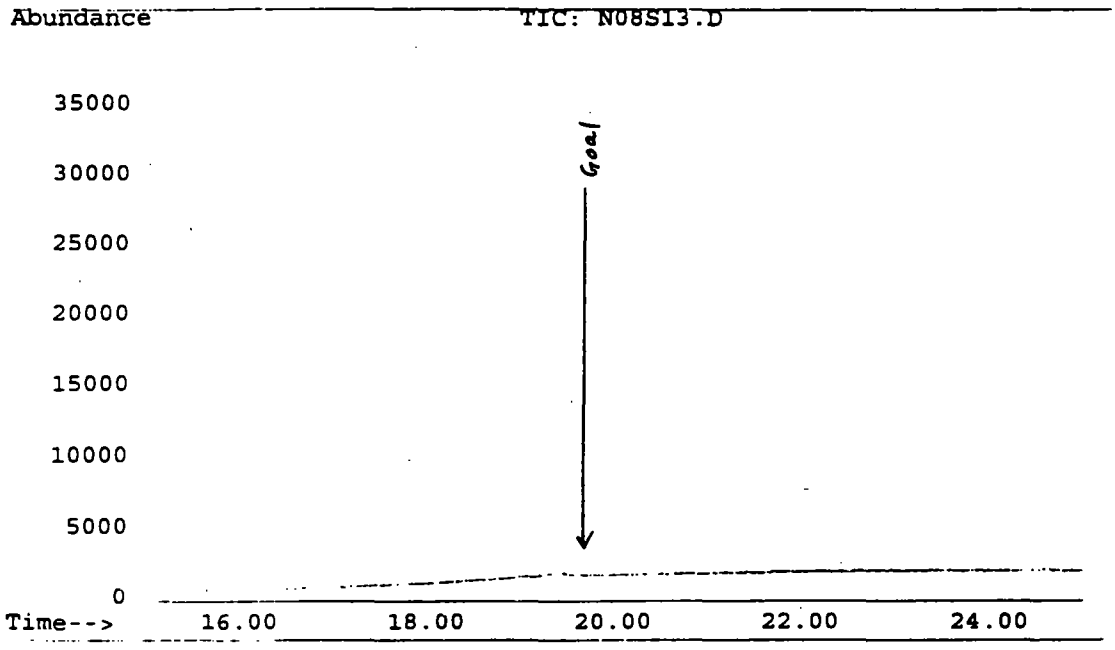


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000031

File : C:\HPCHEM\1\DATA\N08S13.D
Operator : sz
Acquired : 10 Nov 95 1:14 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: wash
Misc Info : na
Vial Number: 13



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S13.D
Operator : sz
Acquired : 10 Nov 95 1:14 am using AcqMethod GOAL1
Sample Name: wash
Misc Info : na
Vial Number: 13
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

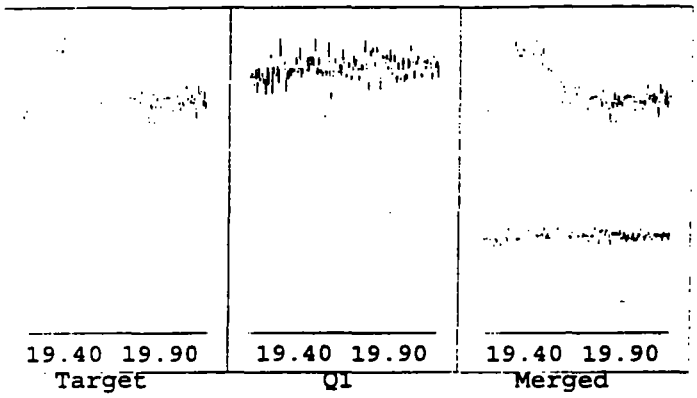
Compound	Expected RT	Actual RT

TR 34 95 111

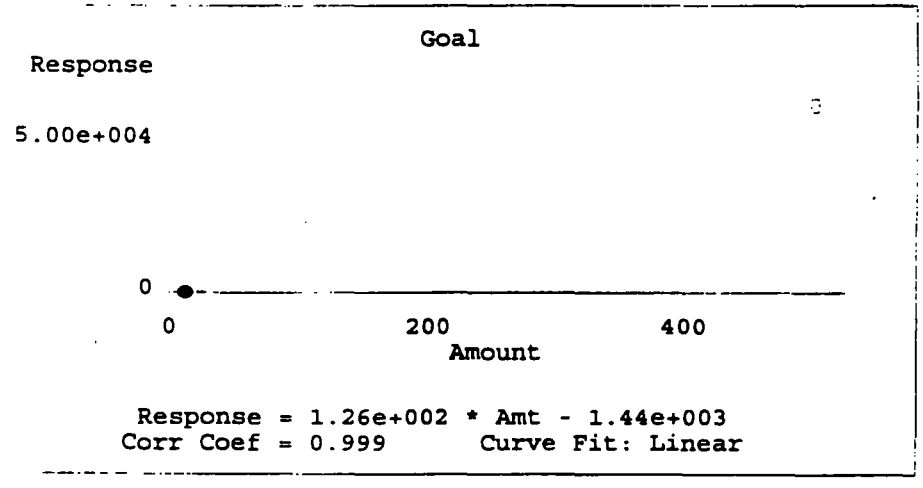
000233

File : C:\HPCHEM\1\DATA\N08S13.D
Operator : sz
Acquired : 10 Nov 95 1:14 am using AcqMethod GOAL1
Sample Name: wash
Misc Info : na
Vial Number: 13
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.85
Concentration: 12.42
Pk # and Type: 1 RTEINT used
Qualifiers Not Satisfied



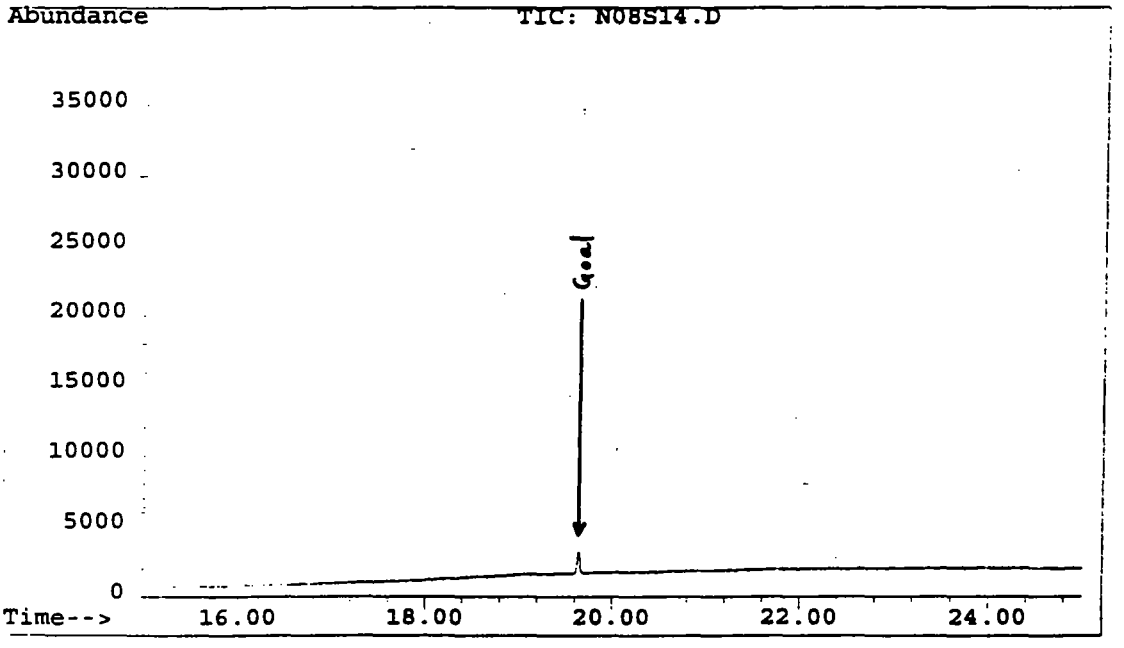
	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.85	19.16	125	sys def
Q1	361.00	0.0	0.0- 22.0	0.00	to	0	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def



TR 34 95 111

000234

File : C:\HPCHEM\1\DATA\N08S14.D
Operator : sz
Acquired : 10 Nov 95 1:52 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 14



Calibration Last Updated: Fri Nov 10 08:35:09 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S4.D
Operator : sz
Acquired : 9 Nov 95 7:30 pm using AcqMethod GOAL1
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 4
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

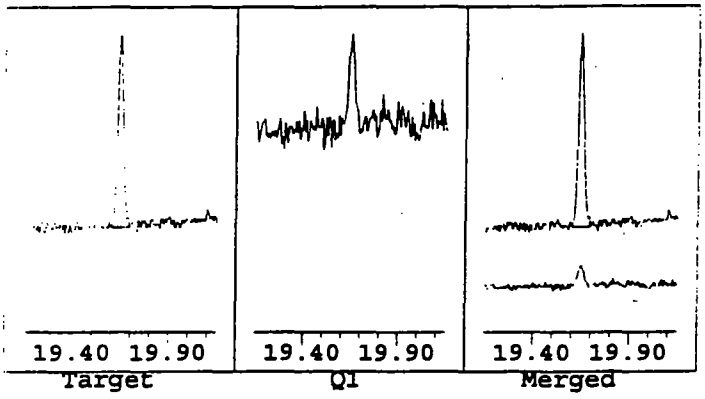
Number of Compounds in Database: 1

Time Reference Peaks:

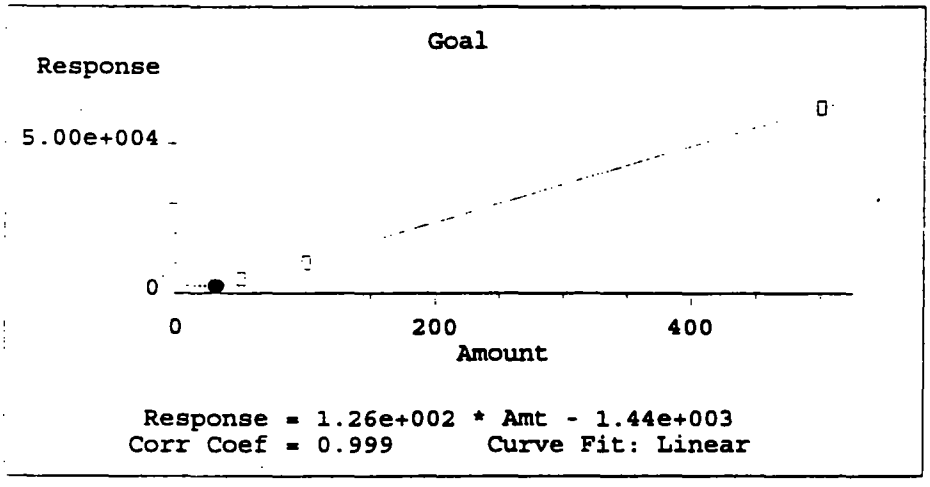
Compound	Expected RT	Actual RT

File : C:\HPCHEM\1\DATA\N08S14.D
Operator : sz
Acquired : 10 Nov 95 1:52 am using AcqMethod GOAL1
Sample Name: std 0.025ug/ml
Misc Info : C103195-5
Vial Number: 14
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 30.97
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.16	2465	sys def
Q1	361.00	13.3	0.0-	22.0	19.65	to	328	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.14	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def

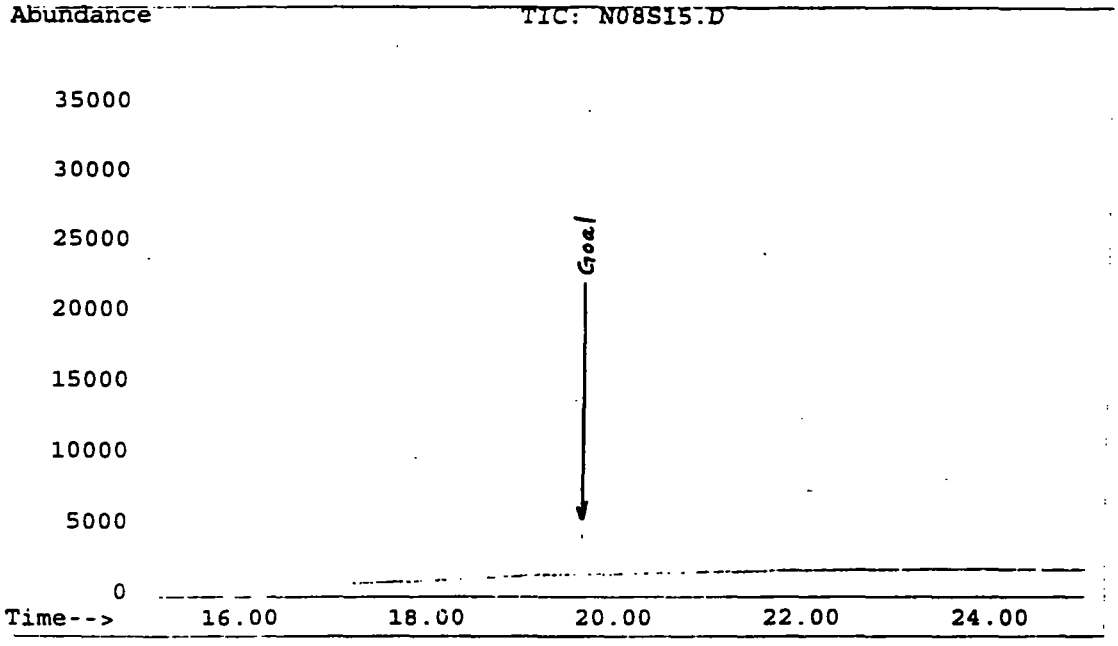


Response = 1.26e+002 * Amt - 1.44e+003
Corr Coef = 0.999 Curve Fit: Linear

TR 34 95 111

000237

File : C:\HPCHEM\1\DATA\N08S15.D
Operator : sz
Acquired : 10 Nov 95 2:30 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 15



Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S15.D
Operator : sz
Acquired : 10 Nov 95 2:30 am using AcqMethod GOAL1
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 15
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

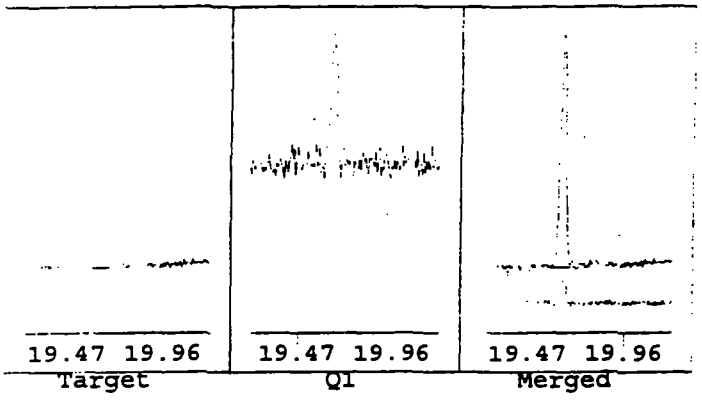
Number of Compounds in Database: 1

Time Reference Peaks:

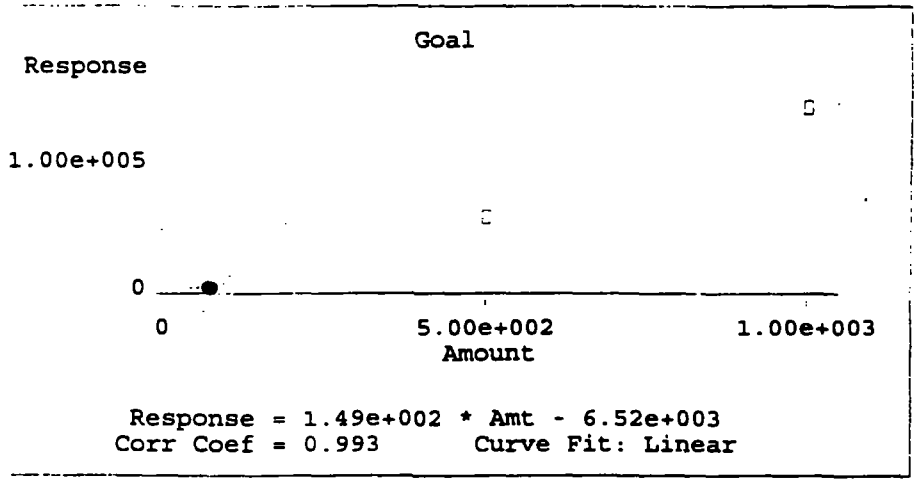
Compound	Expected RT	Actual RT

File : C:\HPCHEM\1\DATA\N08S15.D
Operator : sz
Acquired : 10 Nov 95 2:30 am using AcqMethod GOAL1
Sample Name: std 0.05ug/ml
Misc Info : C103195-4
Vial Number: 15
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 73.95
Pk # and Type: 1 RTEINT used



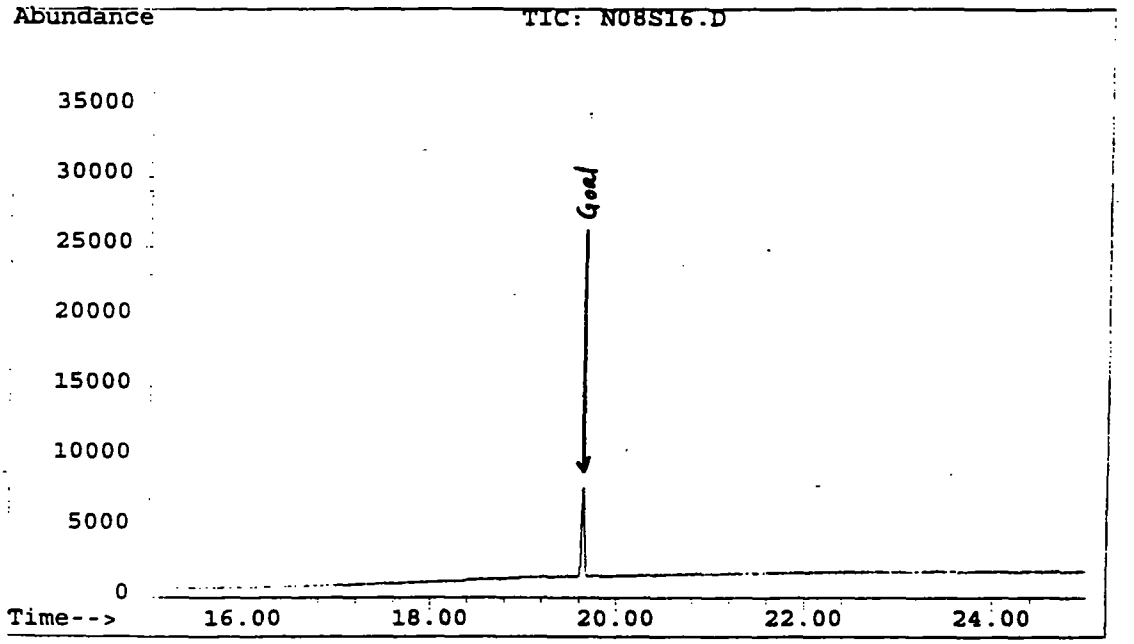
	Signal	Ratios	Limits		RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%			19.64	19.22	4496	sys def
Q1	361.00	13.3	0.0-	22.0	19.65	to	596	sys def
Q2	0.00	0.0	0.0-	0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0-	0.0	0.00		0	sys def



Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

TR 34 95 111

File : C:\HPCHEM\1\DATA\N08S16.D
Operator : sz
Acquired : 10 Nov 95 3:08 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 16



Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S16.D
Operator : sz
Acquired : 10 Nov 95 3:08 am using AcqMethod GOAL1
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 16
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

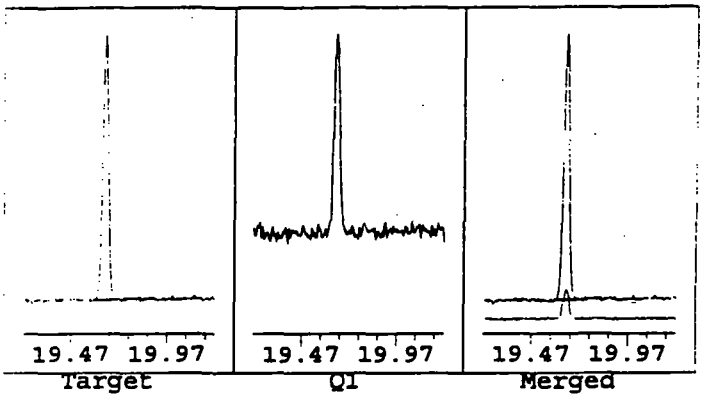
Compound	Expected RT	Actual RT

TR 34 95 111

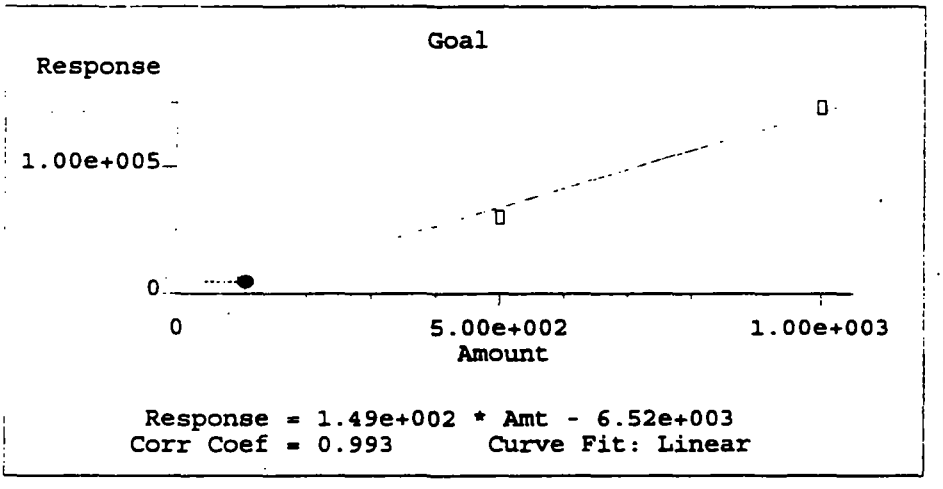
000242

File : C:\HPCHEM\1\DATA\N08S16.D
Operator : sz
Acquired : 10 Nov 95 3:08 am using AcqMethod GOAL1
Sample Name: std 0.1ug/ml
Misc Info : C103195-3
Vial Number: 16
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.64
Concentration: 106.49
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.64	19.22	9344	sys def
Q1	361.00	12.2	0.0- 22.0	19.64	to	1138	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def

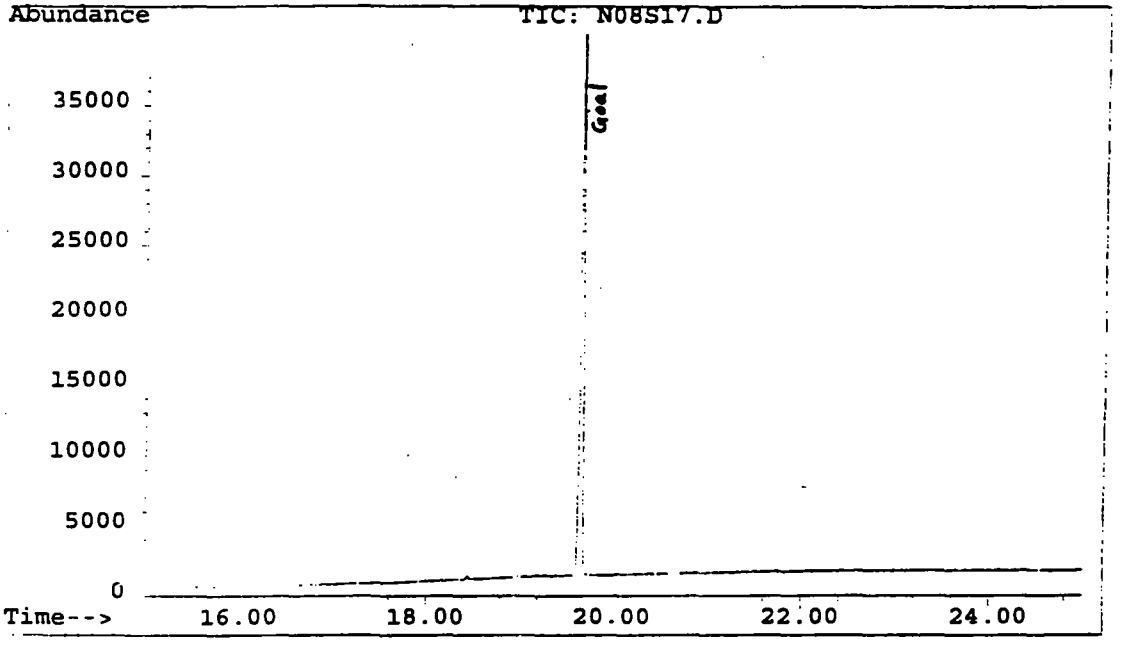


Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

TR 34 95 111

000243

File : C:\HPCHEM\1\DATA\N08S17.D
Operator : sz
Acquired : 10 Nov 95 3:46 am using AcqMethod GOAL1
Instrument : GC/MS # 4
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 17



Calibration Last Updated: Wed Nov 08 08:34:49 1995
Quantitation Report - Detailed

Information from Data File:
File : C:\HPCHEM\1\DATA\N08S17.D
Operator : sz
Acquired : 10 Nov 95 3:46 am using AcqMethod GOAL1
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 17
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Quantitation Settings:

Reference Peak Window:	10.00 Percent
Non-Reference Peak Window:	5.00 Percent
Correlation Window:	0.02 Minutes
Default Multiplier:	1.00
Default Sample Concentration:	0.00
Peak Type Decoding:	* -> Time Reference Peak
	m -> Manually re-integrated
	00X -> Compound references ISTD X

Number of Compounds in Database: 1

Time Reference Peaks:

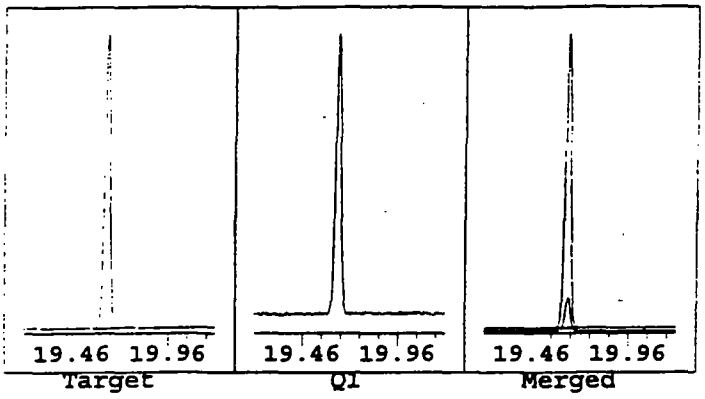
Compound	Expected RT	Actual RT

TR 34 95 111

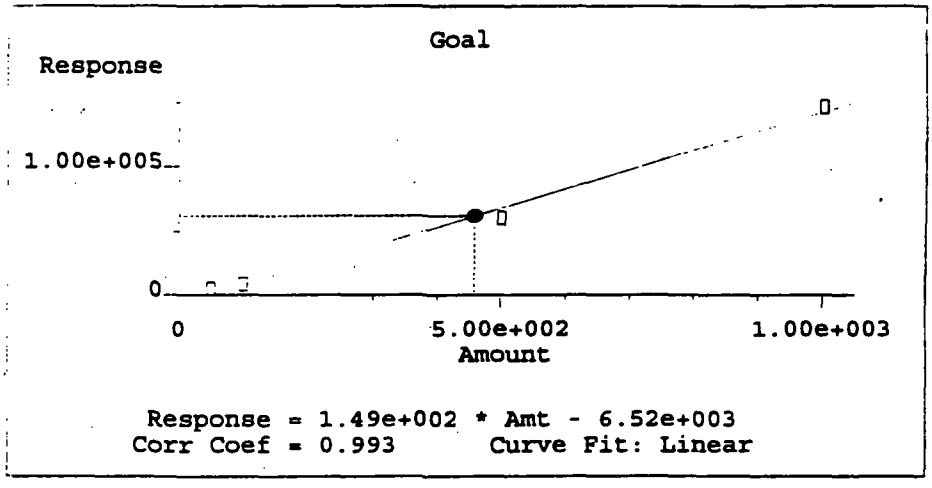
000245

File : C:\HPCHEM\1\DATA\N08S17.D
Operator : sz
Acquired : 10 Nov 95 3:46 am using AcqMethod GOAL1
Sample Name: std 0.5ug/ml
Misc Info : C103195-2
Vial Number: 17
CurrentMeth: C:\HPCHEM\1\METHODS\GOAL1.M

Compound: Goal
Ret Time: 19.65
Concentration: 458.40
Pk # and Type: 1 RTEINT used



	Signal	Ratios	Limits	RT	Limits	Resp	Integ Type
Tgt	252.00	100.0%		19.65	19.22	61774	sys def
Q1	361.00	11.2	0.0- 22.0	19.65	to	6923	sys def
Q2	0.00	0.0	0.0- 0.0	0.00	20.21	0	sys def
Q3	0.00	0.0	0.0- 0.0	0.00		0	sys def



Response = 1.49e+002 * Amt - 6.52e+003
Corr Coef = 0.993 Curve Fit: Linear

CAL STUDY NO. 002-165

SPONSOR PROTOCOL NO. 34P-95-92

DAILY STANDARD INJECTIONS ARE COMPARED AGAINST THE CURRENT CALIBRATION FILE. THIS FILE IS GENERALLY BASED ON THE PREVIOUS RUN'S STANDARDS. THE CALIBRATION FILE IS UPDATED BASED ON THE RUN'S STANDARD, AND SAMPLE ANALYSES ARE QUANTITATED USING THE UPDATED CALIBRATION FILE.

Shaozhi Zheng 1/4/96