

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Thiazopyr

MRID #: 444994-01

Matrix: Water/Soil

Analysis: GC/ECD

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STUDY TITLE

Preliminary Analytical Method for the Determination of Thiazopyr and Its Monoacid Metabolite in Water and Soil

DATA REQUIREMENT

Guideline 164-1

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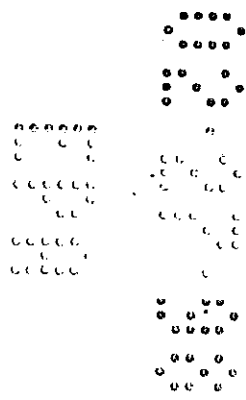
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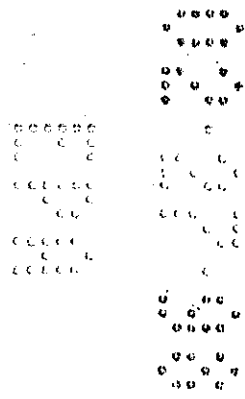
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Good Laboratory Practice Compliance Statement

Good Laboratory Practice regulations do not apply to method development.
However, all data collected in this report were done in a GLP environment.

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Quality Assurance Statement

This report has been reviewed by the Quality Assurance Unit of the Rohm and Haas Company Agricultural Research Division. It has been a true and accurate representation of the data collected.

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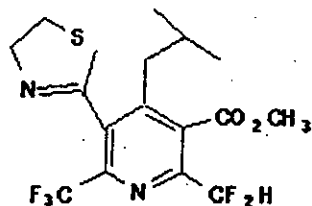
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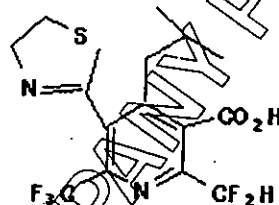
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1. Introduction

Thiazopyr (RH-123652) is a herbicide developed for citrus, grapes, tree nuts and other crops. The CAS registry number of thiazopyr is 117718-60-2 and its chemical name is 3-Pyridinecarboxylic acid, 2-(difluoromethyl)-5-(4, 5-dihydro-2-thiazolyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-, methyl ester. Its molecular structure is shown below. This analytical method describes the analysis of thiazopyr and its metabolite, monoacid, in water and soil.



Thiazopyr



Monoacid

2. Summary

Thiazopyr and monoacid are quantitated separately in this method. The two analytes are first separated by a hexane partition step (after extraction step for soil samples). Thiazopyr, which is extracted into the hexane phase, is quantitated by gas chromatography with an electron capture detector (GC/ECD). The monoacid in the aqueous phase is then acidified and partitioned into ethyl acetate. Derivatization is performed to convert monoacid to its methyl ester, thiazopyr. After a Florisil column clean-up, the monoacid, now in the form of thiazopyr, is quantitated by GC/ECD. The effectiveness of the analytical method is evaluated based on the recoveries of known concentrations of thiazopyr and monoacid fortified into the untreated water and soil samples which are then carried through the analytical procedure. The limit of quantitation (LOQ) is 0.1 ppb in water and 0.01 ppm in soil for both thiazopyr and monoacid. A full set of soil or water samples, including control and fortified control samples, is expected to be extracted and prepared for quantitation within eight hours.

Based on the limited fortification data shown in Table 1 and 2, the average recoveries for thiazopyr and monoacid in soil are 94.1% ± 10.3% and 89.9% ± 2.7%, respectively. The average recoveries for thiazopyr and monoacid in water are 100.5% ± 8.3% and 96.3% ± 7.6%, respectively. A confirmatory method using GC/MSD was also developed. Fortification recoveries are compared between the two procedures.

An enforcement analytical method will be issued after more fortification recovery data are available from the study sample analysis.

3. Chemicals and Supplies

Diazald kit	Aldrich, Z10,025-0, AL-131
Diazomethane	Lab made from the diazald kit.
Ethyl acetate (EtOAc), OPTIMA™ Grade	Fisher
Ethanol (EtOH), 200 proof	Midwest Grain, #6810-00-242-3645
Florisil	Fisher
Hexane, HPLC Reagent	Baker
Hydrochloric acid (HCl), 37% (12 N), Reagent Grade	Fisher
Isooctane (IO)	Fisher
Methanol (MeOH), OPTIMA™ Grade	Fisher
Sodium Chloride (NaCl)	Fisher
Sodium Sulfate (Na ₂ SO ₄)	Fisher
Sodium hydroxide (NaOH)	Fisher
Thiazopyr standard	Rohm & Haas Lot PIT-9001-1445-A
Thiazopyr monoacid standard	Rohm & Haas Lot HET-8912-1256-A
Trimethylsilyldiazomethane (TMS)	Aldrich, #36283-2
Water, Milli-Q	Laboratory

The following solutions may be prepared in advance:

0.4 N HCl, 0.24 N HCl, 0.5 N NaOH, 0.01 N NaOH, 10% NaCl solution by weight, 1:1 ethyl acetate/isooctane (EtOAc/IO) by volume, 70% methanol/0.4 N HCl by volume, 10% ethyl acetate/hexane by volume, and 20% ethyl acetate/hexane by volume.

Any equivalent suppliers may be used after demonstrating suitability.

4. **Equipment**

Büchner Funnel	Waring
Centrifuge Tubes (50 mL)	Pyrex
Chromatographic Column (14.5 cm id)	Pyrex
Glass Fiber Filter Paper	Whatman, #934-AH
Filter Flasks (500 ml)	Pyrex
Round Bottom Flasks, 24/40 (100, 250 and 300 ml)	Pyrex
Rotary Evaporator	Büchi
Separatory Funnels (500 ml)	Pyrex
Standard Laboratory Equipment: (Balances, Beakers, Vortex, and etc.)	Pyrex, Kimax, Mettler
Teflon FEP Round Bottles, 250mL	VWR
Wrist-Action Shaker	Burrell

Any equivalent equipment may be used after demonstrating suitability.

5. **Analytical Procedure**

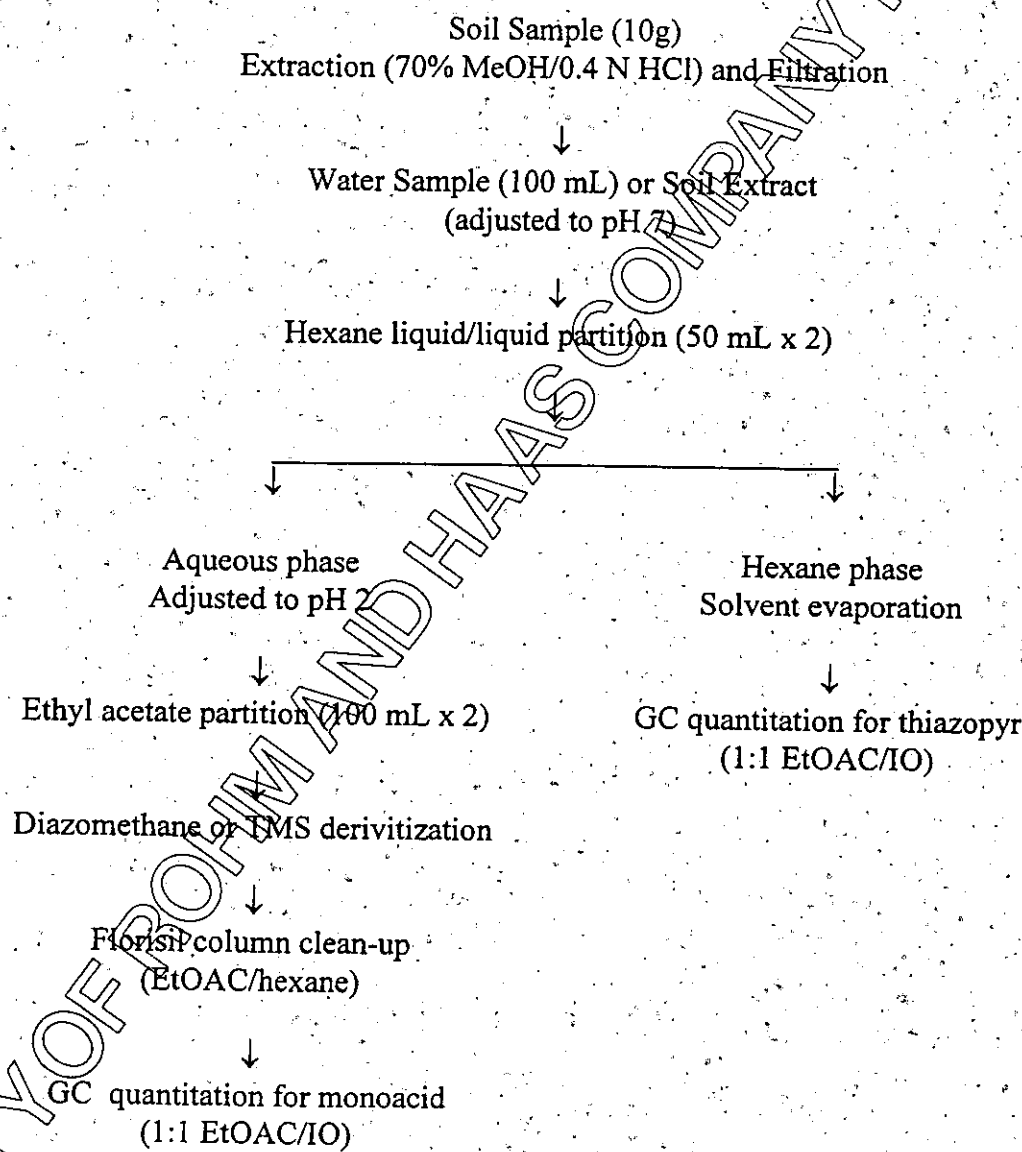
This analytical method describes the analysis of thiazopyr and its metabolite, monoacid, in water and soil. The recommended sample sizes are shown below. However, sample sizes and final volume can be varied depending on the expected analyte concentrations and the amount of sample available. The concentrations

and volumes of some of the chemicals, such as NaOH, HCl, diazomethane, and etc., may also be modified to achieve the best performance of the method.

A full set of soil or water samples, including control and fortified control samples, is expected to be extracted and prepared for quantitation within eight hours.

5.1 Flow Diagram

A flow diagram of the analytical procedure is shown below.



5.2 Extraction of Soil Samples

Weigh 10 grams of soil into a 250 mL Teflon round bottle, if required, spike the sample with the analyte(s) of interest, and add 50 mL of 70% methanol/0.4 N HCl. Shake for 15 minutes on a wrist-action shaker set at maximum speed. Filter the sample and rinse the flask with 20 mL of the same extraction solution twice and pass through the filter cake with vacuum. The extract is then rotary evaporated to less than 25 mL. Adjust volume to about 100 mL with Milli Q water.

5.3 Hexane Partition of Soil Extracts and Water Samples

Measure 100 mL of water sample or transfer the soil extract sample from 5.2 into a beaker. If required, spike the water sample with the analyte(s) of interest. Adjust the sample to a pH of 7 with 0.01 N NaOH for water samples and 0.5 N NaOH for soil extracts, then transfer to a 500-mL separatory funnel. Rinse the original water or soil extract container with 50 mL of hexane and transfer to the separatory funnel. Partition twice with 50 mL of hexane each time. Reserving the aqueous layer. Combine the hexane layers into a round bottom flask and rotovap to dryness. Dissolve the sample in an appropriate final volume of 1:1 ethyl acetate/isooctane. The final volume is usually 50 to 200 mL for soil samples and 5 to 20 mL for water samples. The sample is now ready for GC quantitation of thiazopyr.

Samples at this stage are stable at room temperature. If they are not expected to be injected right away, they should be sealed well to prevent solvent from evaporating.

5.4 Ethyl Acetate Partition

Transfer the aqueous phase from the previous step to a 250 mL beaker and adjust to pH 2 with 0.24 N HCl solution. Transfer to a 500 mL separatory funnel and extract twice with 100 mL ethyl acetate each time. Discard the aqueous layer. Combine the ethyl acetate layers in a 250 or 300 mL round bottom flask and evaporate to approximately 5 mL, then transfer to a 50 mL centrifuge tube. Rinse the round bottom twice with 2 mL of ethyl acetate and add to the centrifuge tube.

5.5 Derivatization

Evaporate the ethyl acetate solution to about 1 mL by nitrogen. Add about 100 μ L of MeOH and 1 mL of diazomethane or 0.5 mL trimethylsilyl-diazomethane (TMS) until the solution turns yellow. Cap and vortex to mix. Allow to stand for at least 30 minutes and transfer to a separatory

funnel containing 40 mL of MeOH and 60 mL of 10% NaCl. Partition twice with 50 mL hexane each time. Discard the aqueous layer. Combine hexane layers in a 250 mL round bottom flask and evaporate to approximately 5 mL.

Note: Special care needs to be taken when making and storing diazomethane. Instructions from the diazald kit should be closely followed.

5.6. Florisil Column Clean-up

Pre-activate Florisil, 60-100 mesh, by heating in oven for 24 hours at 200°C. Insert a small glass wool plug into a 145 mm ID chromatographic column and slurry pack the column with 15 cc of activated Florisil in hexane. Top the column with approximately 5 cc of anhydrous sodium sulfate. Rinse the column with 20 mL of hexane. Apply the hexane layer from step 5.5. Then rinse the round bottom flask with 30 mL of hexane which is then applied to the column. Wash the column again with 30 mL of 10% ethyl acetate/hexane. Discard the washes. Elute the column with 40 mL of 20% ethyl acetate/hexane and collect the eluent in a 300 mL round bottom flask. Concentrate eluent to dryness under vacuum at ~45°C using a rotary evaporator. Dissolve sample in an appropriate final volume of 1:1 isooctane/ethyl acetate. The final volume is usually 50 to 200 mL for soil samples and 5 to 20 mL for water samples. The sample is now ready for GC quantitation of the monoacid (converted to thiazopyr).

Samples at this stage are stable at room temperature. If they are not expected to be injected right away, they should be sealed well to prevent solvent from evaporating.

6. Instrumentation

Gas chromatography (GC) with an electron capture detector (ECD) is used for quantitation. The conditions on an HP 5890 GC/ECD are shown below.

Column:	RTX-5, 30 meters x 0.53 mm ID x 1.5 µm film
Carrier gas:	Helium
Flow Rate:	10 ml/min
Injection Volume:	5 µl
Injection Mode:	Splitless
Injection Temp:	250°C
ECD Temp:	300°C
Column Temp:	160°C for 1.0 minutes, 160 - 260°C at 6°/minute, 260°C for 5 minutes

Under these conditions, the typical retention time for the thiazopyr is about 7.4 minutes. Control (untreated) samples are run concurrently with the analytical samples to determine the presence of matrix interference. A solvent blank may be injected with the samples as part of an analytical set to confirm the cleanliness of the solvent used.

Similar conditions may be used after demonstrating suitability.

7. Analytical Standards

Analytical standard solutions are prepared for fortifying control matrices to determine analytical recoveries and for calibrating the response of the analyte in the gas chromatographic system.

7.1 Standard Stock Solutions

100 $\mu\text{g/ml}$ thiazopyr solution

Weigh 0.0100 grams (weight adjusted for purity) of analytical grade thiazopyr into a 100 ml volumetric flask, dilute to volume with ethanol and mix well to insure complete dissolution. This solution contains 100 $\mu\text{g/ml}$ of thiazopyr.

100 $\mu\text{g/ml}$ monoacid solution

Weigh 0.0100 grams (weight adjusted for purity) of analytical grade thiazopyr monoacid into a 100 ml volumetric flask, dilute to volume with ethanol and mix well to insure complete dissolution. This solution contains 100 $\mu\text{g/ml}$ of monoacid.

7.2 Fortification Solutions

Samples will be fortified at different analyte levels. The solutions used to fortify control samples are prepared in the following manner.

1.0 $\mu\text{g/ml}$ solution

Pipet 1.0 ml the 100 $\mu\text{g/ml}$ thiazopyr and/or monoacid solution into a 100 ml volumetric flask, dilute to volume with ethanol and mix well. This standard contains 1.0 $\mu\text{g/ml}$ of thiazopyr and/or monoacid.

0.10 $\mu\text{g/ml}$ solution

Pipet 10.0 ml the 1.0 $\mu\text{g/ml}$ thiazopyr and/or monoacid solution into a 100 ml volumetric flask, dilute to volume with ethanol and mix well. This standard contains 0.10 $\mu\text{g/ml}$ of thiazopyr and/or monoacid.

7.3 GC Calibration Standard Solutions

The GC calibration standard solutions are made at convenient concentrations of the analyte, thiazopyr, in a 1:1 EtOAC/IO solution. These standards are used to construct a calibration curve which is then used for quantitation of an analyte. Concentrations of thiazopyr in soil and water can be directly calculated based on the calibration curve.

When the standard solutions used for quantitation of monoacid, however, the thiazopyr concentrations of the standard solutions are multiplied by a conversion factor. Therefore, concentrations of monoacid in soil and water can be calculated directly from the reconstructed calibration curve for monoacid.

$$\text{Conversion factor} = \frac{\text{MW monoacid } 382.4}{\text{MW thiazopyr } 396.4} = 0.9647$$

Where MW is the molecular weight of monoacid (382.4) and thiazopyr (396.4), respectively.

1.00 µg/ml solution

Pipet 1.00 ml of the 100 µg/ml thiazopyr stock solution into a 100 ml volumetric flask, dilute to volume with 1:1 EtOAC/IO and mix well. This solution contains 1.00 µg/ml of thiazopyr. When used for the quantitation of monoacid, the monoacid equivalent concentration of this solution is $1.00 \times 382.4/396.4 = 0.965$ ppm.

0.10 µg/ml solution

Pipet 10.0 ml of the 1.00 µg/ml thiazopyr stock solution into a 100 ml volumetric flask, dilute to volume with 1:1 EtOAC/IO and mix well. This solution contains 0.10 µg/ml of thiazopyr. This is the working solution from which calibration standard solutions are made. When used for the quantitation of monoacid, the monoacid equivalent concentration of this solution is $0.10 \times 382.4/396.4 = 0.0965$ ppm.

The following is an example of calibration standard levels. Concentrations other than the ones shown below also may be prepared and used. The range of concentrations used in the method development is from 0.0010 to 0.010 µg/ml. When the standard solutions used for thiazopyr, the concentrations are listed in the column of Final Conc. Thiazopyr (ppm). When the standard solutions used for monoacid, the concentrations are listed in the column of Final Conc. Monoacid (ppm).

<u>Vol. of 0.100 ppm Std. Solution (mL)</u>	<u>Final Volume (mL)</u>	<u>Final Conc. Thiazopyr (ppm)</u>	<u>Final Conc. Monoacid (ppm)</u>
1.00	100	0.0010	0.00096
2.00	100	0.0020	0.00193
5.00	100	0.0050	0.00482
10.00	100	0.0100	0.00945

Dilute each of the detector calibration standards to a final volume of 100 ml with 1:1 EtOAc/IO.

8. Calculations

Standard solutions are prepared in the concentration range of 0.0010 µg/mL to 0.010 ppm. If necessary, sample final volumes should be adjusted to give a response within the standard curve range.

Standards and samples should be quantified using peak areas or heights. Construct a calibration curve with every set of samples. A minimum of four standards should be used for every curve.

8.1 Residue Concentration

Equation 1,

$$\text{ppm} = \frac{(\mu\text{g/mL detected})(\text{final volume, mL})}{\text{sample weight, g}}$$

In the case of the metabolite, monoacid is converted to thiazopyr for quantitation. Since the calibration standards used for monoacid quantitation are already translated to monoacid equivalent concentrations (Section 7), equation 1 can be used directly for calculating ppm of monoacid in the original samples.

8.2 Fortification Recovery

The analytical recovery for an individual fortification is calculated as below.

Equation 2,

$$\% \text{ recovery} = \frac{[(\mu\text{g/mL detected} \times \text{final volume, mL}) - \mu\text{g detected in control}] \times 100}{\mu\text{g fortified}}$$

8.3 Total Thiazopyr Concentration

When this method is used for residue analysis, total residue may be reported as thiazopyr equivalent by use of equation 3.

Equation 3,

$$\text{Total ppm of Thiazopyr} = \text{ppm of Thiazopyr} + (\text{ppm of Monoacid} \times 1.04)$$

Where,

$$\text{Conversion factor} = \frac{\text{MW thiazopyr } 396.4}{\text{MW monoacid } 382.4} = 1.04$$

9. Results and Discussion

The limits of quantitation of this method (LOQ) for both thiazopyr and monoacid are 0.010 ppm in soil and 0.10 ppb in water determined by fortifications at these levels. The limits of detection are 0.003 ppm and 0.03 ppb, respectively. Based on the limited fortification data shown in Tables 1 and 2, the average recoveries for thiazopyr and monoacid in soil are 94.0% ± 10.2% and 89.9% ± 2.7%, respectively. The average recoveries for thiazopyr and monoacid in water are 100.6% ± 8.3% and 96.3% ± 7.5%, respectively. An enforcement analytical method will be issued later when more fortification recovery data are available from the study sample analysis.

Representative standard chromatograms and curve on GC/ECD are shown in Figures 1-5. Control and fortified sample chromatograms can be found in Figures 6-13 for soil and Figures 14-21 for water.

10. Confirmatory Procedure

A confirmation procedure is developed to ensure the nature of a detected peak, if necessary. This is done by preparing the soil or water samples as described in section 5. The final volume is usually 10 to 20 mL for soil samples and 1 to 2 mL for water samples. However, the samples are injected to a gas chromatographic system (GC) with a mass spectrometer detector (MSD). The conditions on a Hewlett Packard 6890 GC with a 5973 MSD are shown below.

Under these conditions, the typical retention time for the thiazopyr is about 8.7 minutes. Control (untreated) samples are run concurrently with the analytical samples to determine the presence of matrix interference. A solvent blank may be injected with the samples as

part of an analytical set to confirm the cleanliness of the solvent used. Similar conditions may be used after demonstrating suitability.

Column:	HP-5MS, 30 meters x 0.25 mm ID x 0.25 μ m film
Carrier gas:	Helium
Flow Rate:	2 ml/min for 5 min, decrease to 1 ml/min
Injection Volume:	2 μ l
Injection Mode:	Pulsed Splitless, 25 psi for 1 min
Injection Liner:	2 mm ID cyclo double gooseneck - splitless
Purge Flow to Split Vent:	25 ml/min.
Injector Purge Delay:	1 min
Injection Temp:	250°C
Dwell Time:	100 msec
Transfer Line Temperature:	300°C
Ions Monitored:	363 and 396
Column Temp:	120°C for 10 minutes 120 - 280°C at 15°/minute 280°C for 4 minutes

Tables 3 and 4 show the fortification recoveries from the same sets of soil and water samples on GC/ECD and GC/MS. Results are satisfactory. Representative standard chromatograms and the calibration curve on GC/MSD are shown in Figures 22-26. Control and fortified sample chromatograms can be found in Figures 27-34 for soil and Figures 35-42 for water.

Table 1. Recoveries of Thiazopyr and Monoacid in Soil

<u>Sample</u>	<u>Fortification Level (ppm)</u>	<u>Analysis Date</u>		<u>Recovery (%)</u>		
		Thiazopyr	Monoacid	Thiazopyr	Monoacid	
Soil	0.01	11/11/97	11/10/97	149	90.1	
	0.01	11/11/97	11/10/97	87.4	86.0	
	0.05	11/11/97	11/10/97	88.9	89.4	
	0.05	11/11/97	11/10/97	88.0	88.4	
	0.10	11/11/97	11/10/97	84.6	85.5	
	0.10	11/11/97	11/10/97	90.4	90.8	
	0.01	11/11/97	11/13/97	111	92.2	
	0.01	11/11/97	11/13/97	90.7	88.4	
	0.05	11/11/97	11/13/97	93.7	93.7	
	0.05	11/11/97	11/13/97	91.2	93.1	
	0.10	11/11/97	11/13/97	89.3	88.4	
	0.10	11/11/97	11/13/97	94.7	92.4	
	Average				94.1 ± 10.3	89.9 ± 2.7

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Table 2. Recoveries of Thiazopyr and Monoacid in Water

<u>Sample</u>	<u>Fortification Level (ppb)</u>	<u>Analysis Date</u>		<u>Recovery (%)</u>		
		Thiazopyr	Monoacid	Thiazopyr	Monoacid	
Water	0.1	11/04/97	11/05/97	95.3	104	
	0.1	11/04/97	11/05/97	98.9	99.8	
	0.5	11/04/97	11/05/97	96.6	99.9	
	0.5	11/04/97	11/05/97	81.0	97.7	
	1.0	11/04/97	11/05/97	95.7	98.1	
	1.0	11/04/97	11/05/97	98.8	108	
	0.1	11/06/97	11/07/97	105	100	
	0.1	11/06/97	11/07/97	105	100	
	0.5	11/06/97	11/07/97	108	85.2	
	0.5	11/06/97	11/07/97	114	86.5	
	1.0	11/06/97	11/07/97	102	92.0	
	1.0	11/06/97	11/07/97	106	84.5	
	Average				100.5 ± 8.3	96.3 ± 7.6

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Table 3. Recovery Comparison of Thiazopyr and Monoacid in Soil: GC/ECD vs GC/MSD

Sample	Fortification Level (ppm)	Thiazopyr Analysis Date				Monoacid Analysis Date				Recovery (%)			
		GC/ECD	GC/MSD	GC/ECD	GC/MSD	GC/ECD	GC/MSD	GC/ECD	GC/MSD	GC/ECD	GC/MSD	GC/ECD	GC/MSD
Soil	0.01	11/11/97	11/11/97	11/11/97	11/11/97	119	87.1	90.1	87.1	90.1	87.1	87.1	
	0.01	11/11/97	11/11/97	11/10/97	11/11/97	87.4	95.4	86.0	95.4	86.0	86.2	86.2	
	0.05	11/11/97	11/11/97	11/10/97	11/11/97	88.9	100	89.4	100	89.4	96.3	96.3	
	0.05	11/11/97	11/11/97	11/10/97	11/11/97	88.0	105	88.4	105	88.4	96.1	96.1	
	0.10	11/11/97	11/11/97	11/10/97	11/11/97	84.6	95.5	85.5	95.5	85.5	83.4	83.4	
	0.10	11/11/97	11/11/97	11/10/97	11/11/97	90.4	98.5	90.8	98.5	90.8	90.4	90.4	
Average					93.1 ± 4.2	96.9 ± 6.0	88.4 ± 2.2	96.9 ± 6.0	88.4 ± 2.2	89.9 ± 5.4	89.9 ± 5.4		

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Table 4. Recovery Comparison of Thiazopyr and Monoacid in Water: GC/ECD vs GC/MSD

Sample	Fortification Level (ppm)	Thiazopyr		Monoacid		Recovery (%)			
		GC/ECD	GC/MSD	GC/ECD	GC/MSD	Thiazopyr GC/ECD	Thiazopyr GC/MSD	Monoacid GC/ECD	Monoacid GC/MSD
Water	0.01	11/04/97	11/07/97	11/05/97	11/07/97	95.3	94.5	104	92.4
	0.01	11/04/97	11/07/97	11/05/97	11/07/97	98.9	113	99.8	91.4
	0.05	11/04/97	11/07/97	11/05/97	11/07/97	96.6	97.3	99.9	107
	0.05	11/04/97	11/07/97	11/05/97	11/07/97	81.0	125	97.7	117
	0.10	11/04/97	11/07/97	11/05/97	11/07/97	95.7	109	98.1	94.6
	0.10	11/04/97	11/07/97	11/05/97	11/07/97	98.8	106	108	92.2
Average					94.4 ± 6.7	107 ± 11	101 ± 4.0	99.1 ± 10.5	

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Analysis of Thiazopyr

Run No.: 1

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj, Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: NA

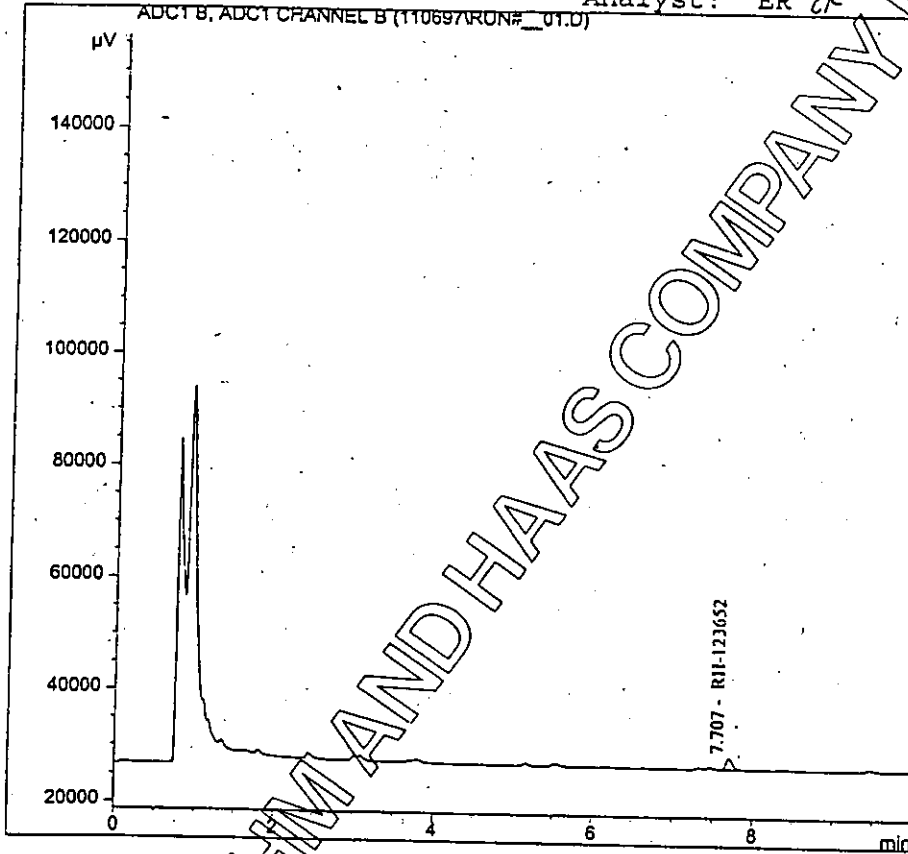
Sample Info: RH-123652 standard, 0.001 ug/mL C100197/5

Injected on: 11/6/97

9:19:13 AM

Method: NA

Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.71	RH-123652	2035	11948

Figure 1. 0.0010 ppm thiazopyr standard on GC/ECD.

TR 34-97-175

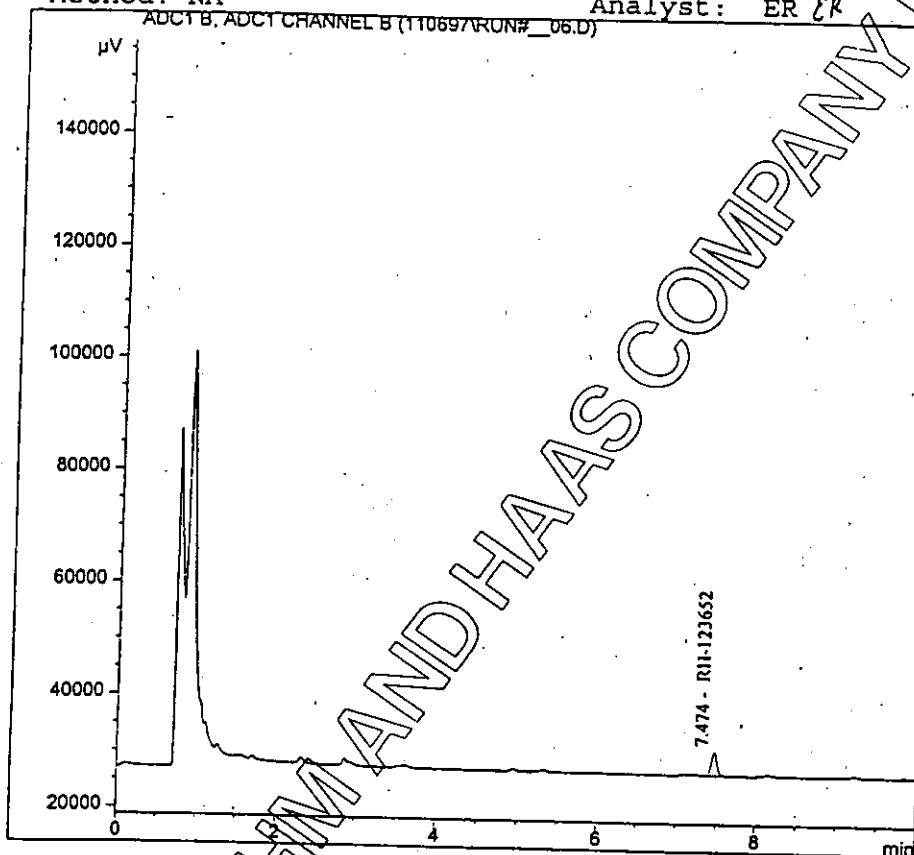
Analysis of Thiazopyr

Run No.: 6
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: NA
Sample Info: RH-123652 standard, 0.002 ug/mL C100197

Injected on: 11/6/97
Method: NA

11:25:30 AM
Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.47	RH-123652	4291	20595

Figure 2. 0.0020 ppm thiazopyr standard on GC/ECD.

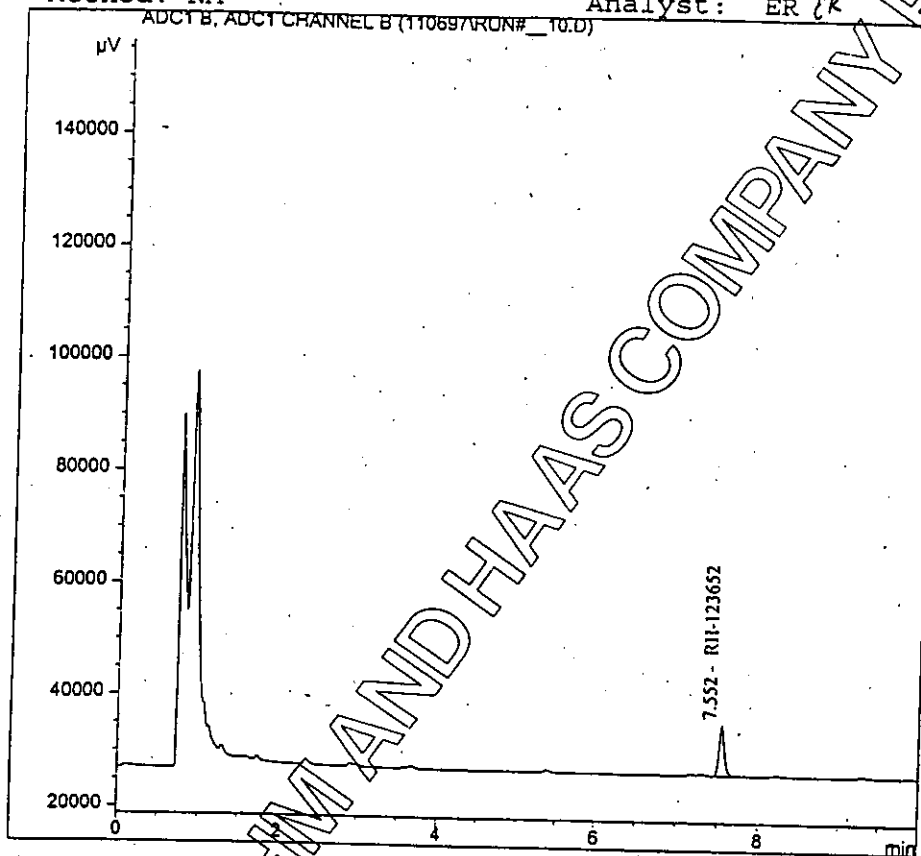
TR 34-97-175

Analysis of Thiazopyr

Run No.: 10
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: NA
Sample Info: RH-123652 standard, 0.005 ug/mL C1001973

Injected on: 11/6/97 1:06:28 PM
Method: NA Analyst: ER CR



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.55	RH-123652	9169	43810

Figure 3. 0.0050 ppm thiazopyr standard on GC/ECD.

Analysis of Thiazopyr

Run No.: 14

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: NA

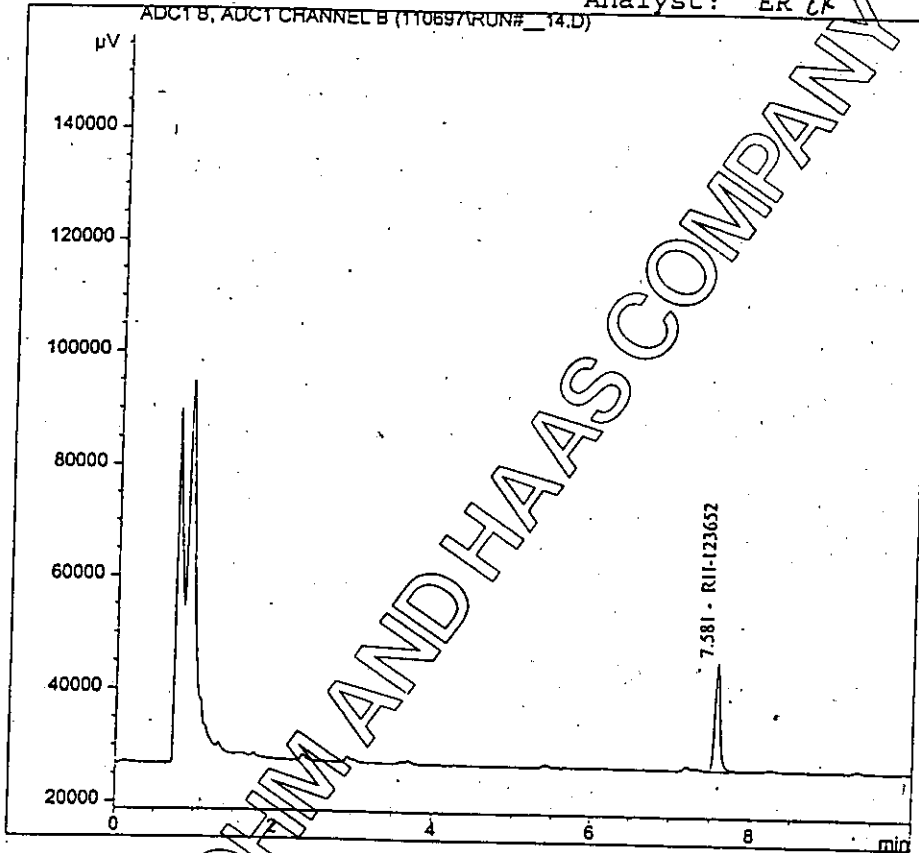
Sample Info: RH-123652 standard, 0.01 ug/mL C100197

Injected on: 11/6/97

2:47:26 PM

Method: NA

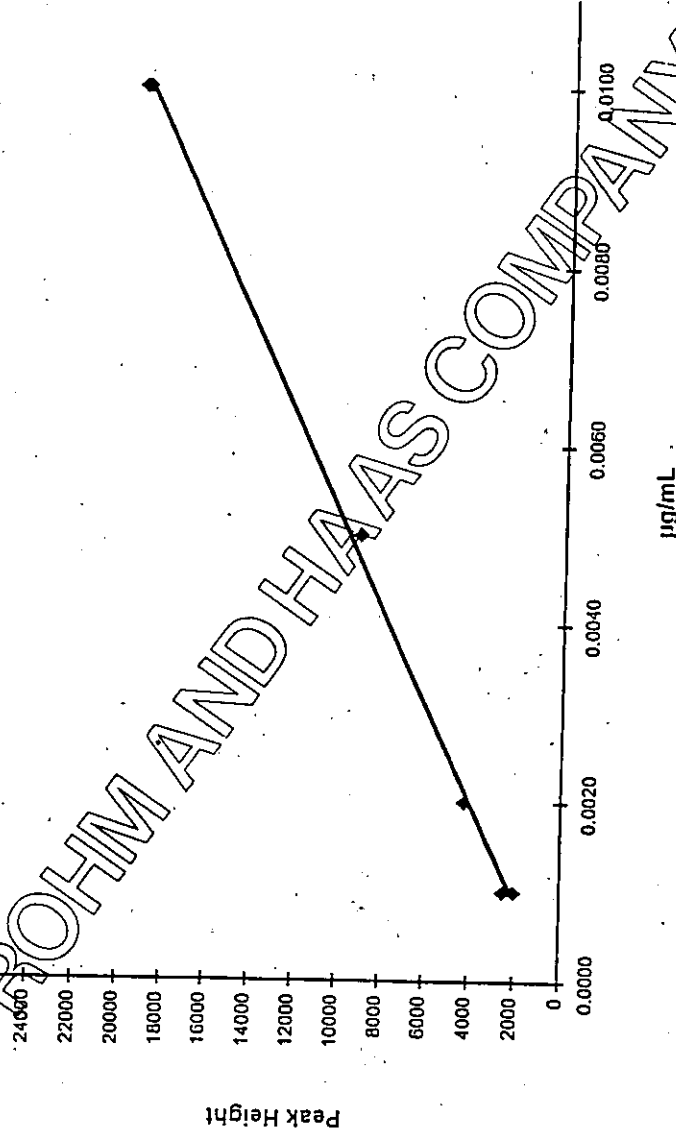
Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.58	RH-123652	19290	94527

Figure 4. 0.010 ppm thiazopyr standard on GC/ECD.

RH-123652 CALIBRATION CURVE
11/6/97



Concentrations in report are calculated from the equation:
Peak Height = 1869158.2*(µg/mL) + 326.41
obtained by least-squares fit of standard injection data.

Figure 5. Thiazopyr standard calibration curve on GC/ECD.

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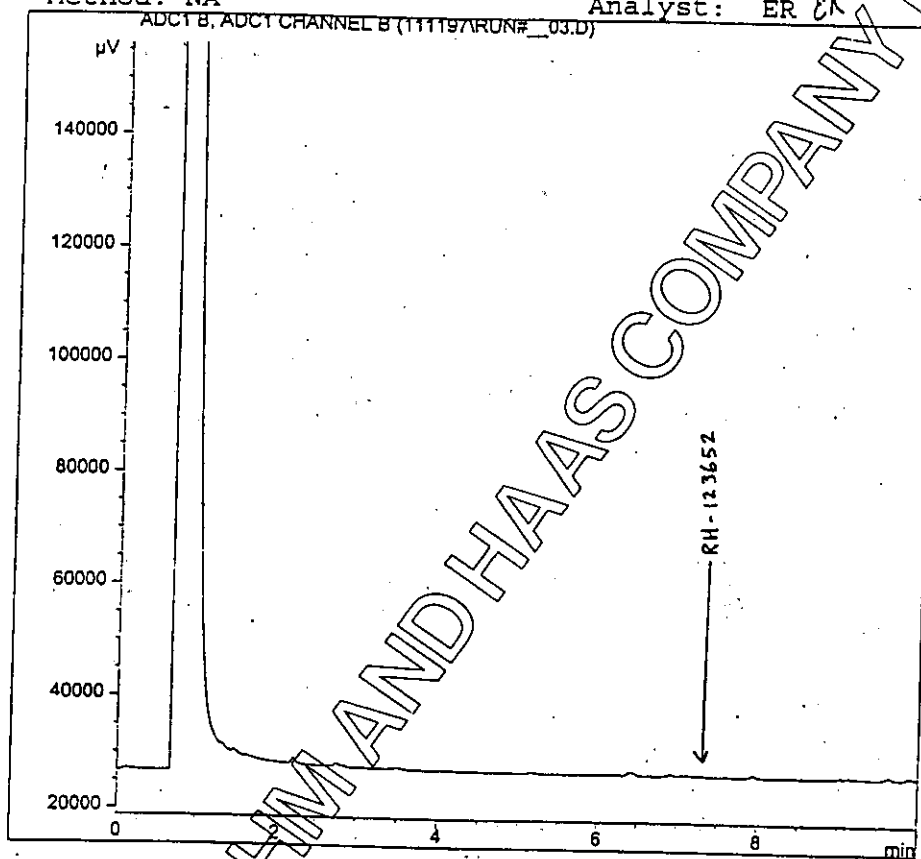
TR 34-97-175

Analysis of Thiazopyr

Run No.: 3
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
Sample Info: Blk, Sample Wt. = 10 g, FV= 50 mL

Injected on: 11/11/97 10:40:04 AM
Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
0.00	RH-123652	0	0

Figure 6. Thiazopyr soil control on GC/ECD.

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Analysis of Thiazopyr

Run No.: 4

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

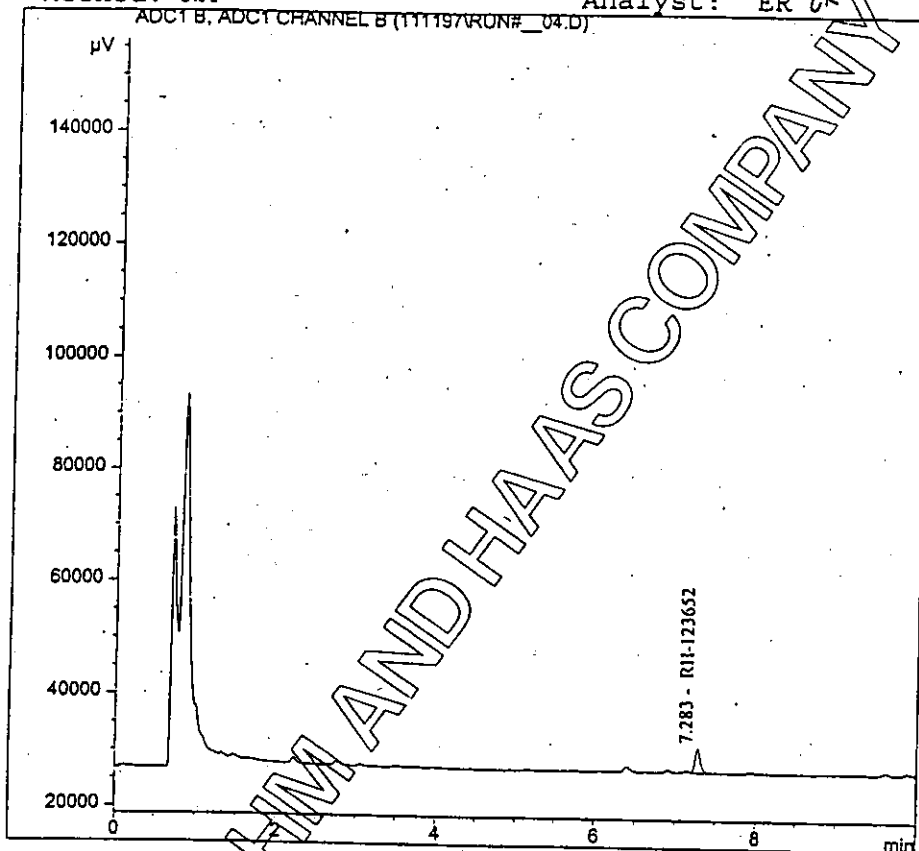
Sample No: HB-1
Sample Info: Spk A, Sample Wt. = 10 g, FV = 50 mL
0.1 μ g added

Injected on: 11/11/97

11:05:23 AM

Method: NA

Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.28	RH-123652	4350	20606

Figure 7. Soil sample fortified with 0.010 ppm thiazopyr on GC/ECD.

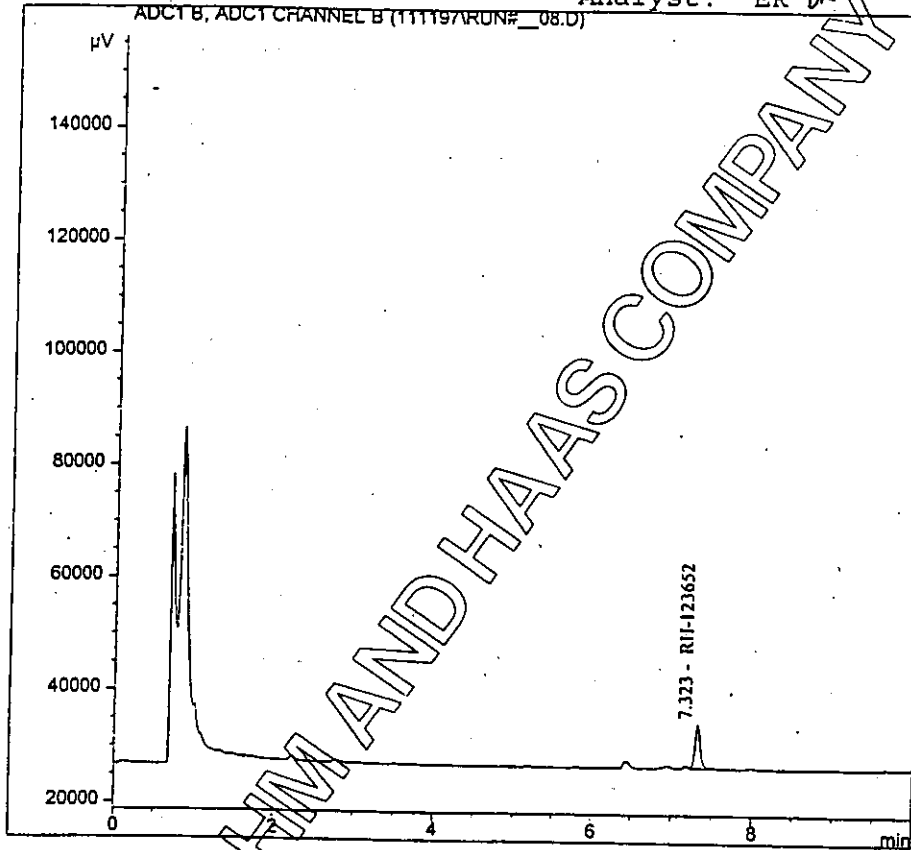
Analysis of Thiazopyr

Run No.: 8

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
 Sample Info: Spk C, Wt.= 10 g, FV= 100 mL
 0.5 μ g added

Injected on: 11/11/97 12:46:26 PM
 Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μV)	Peak Area (μV*s)
7.32	RH-123652	7941	37856

Figure 8. Soil sample fortified with 0.050 ppm thiazopyr on GC/ECD.

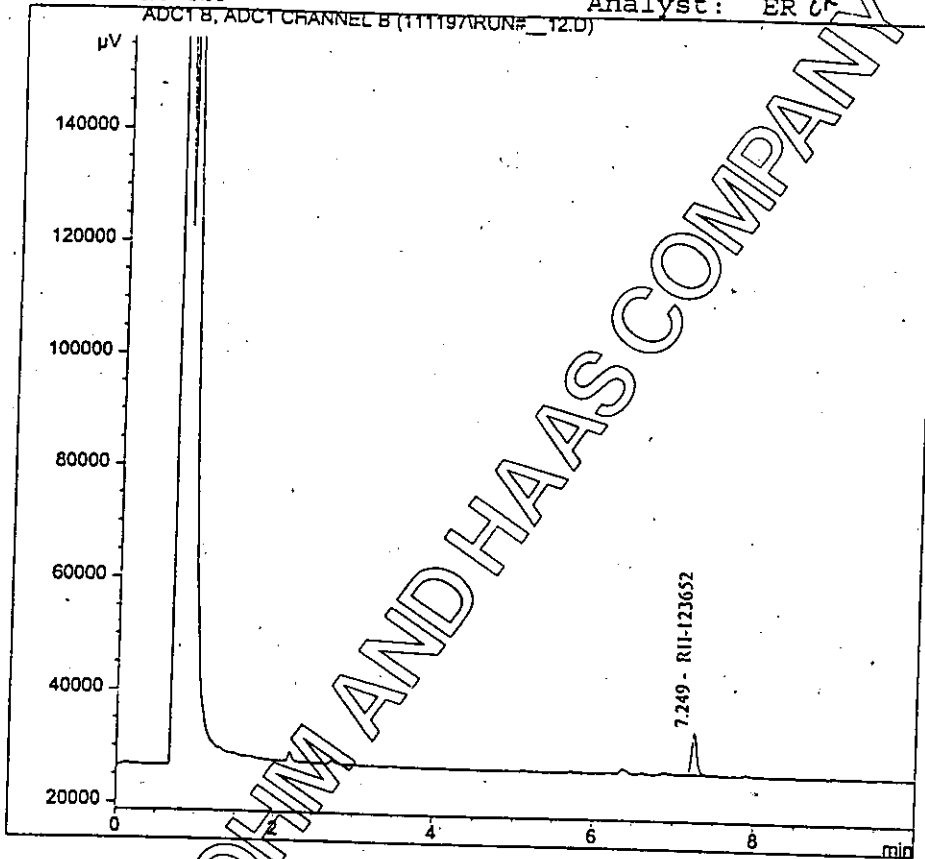
TR 34-97-175

Analysis of Thiazopyr

Run No.: 12
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
Sample Info: Spk E, Wt.=10 g, FV=200 mL
1.0 μ g added

Injected on: 11/11/97 2:27:30 PM
Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.25	RH-123652	7565	36125

Figure 9. Soil sample fortified with 0.10 ppm thiazopyr on GC/ECD.

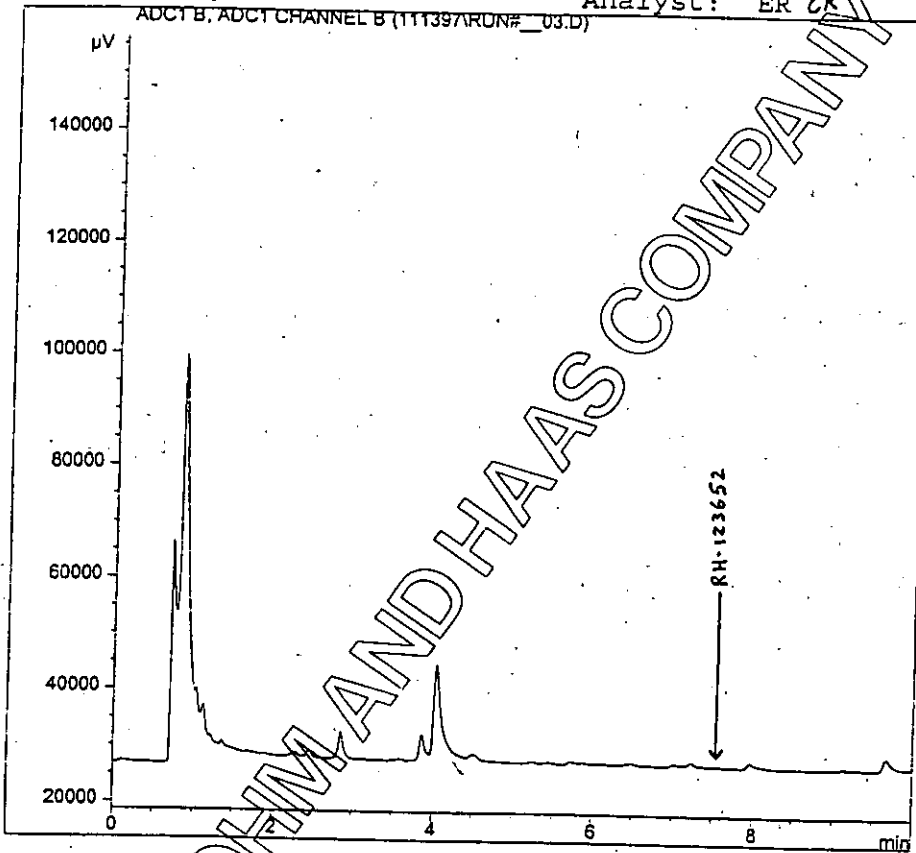
TR 34-97-175

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 3
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
Sample Info: Blk1, Sample Wt. = 10 g, FV= 50 mL

Injected on: 11/13/97 12:01:11 PM
Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
0.00	RH-123652	0	0

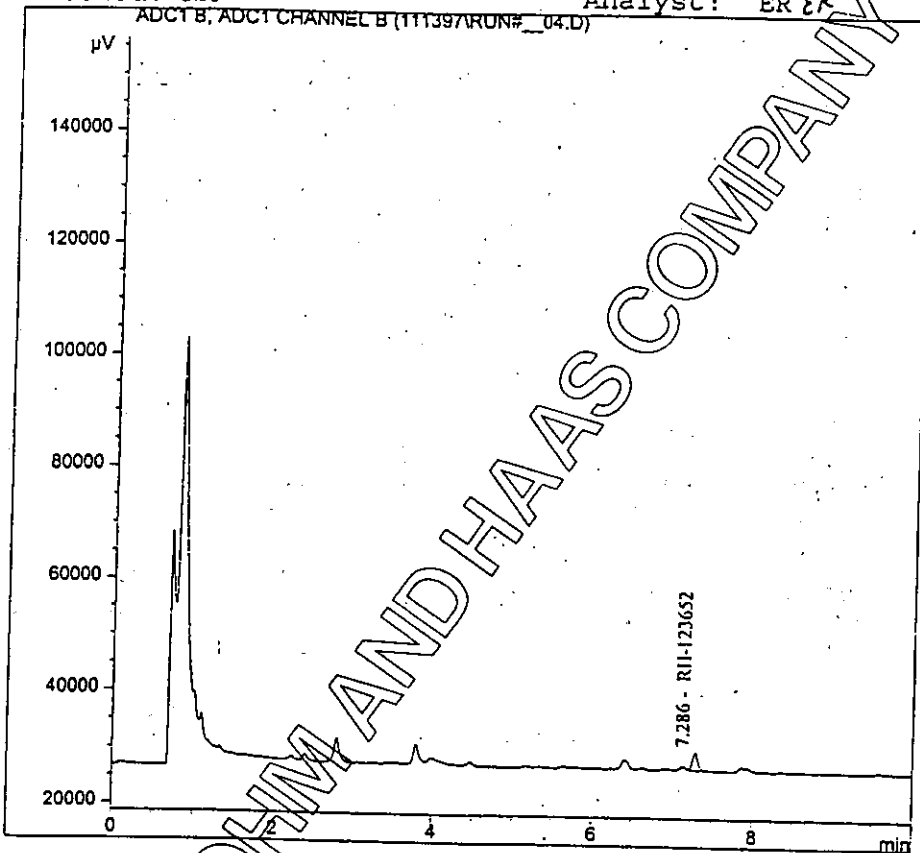
Figure 10. Monoacid soil control on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 4
 HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
 Sample Info: Spk A1, Sample Wt.= 10 g, FV= 50 mL
 0.1 μ g added

Injected on: 11/13/97 12:26:28 PM
 Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.29	RH-123652	3239	15173

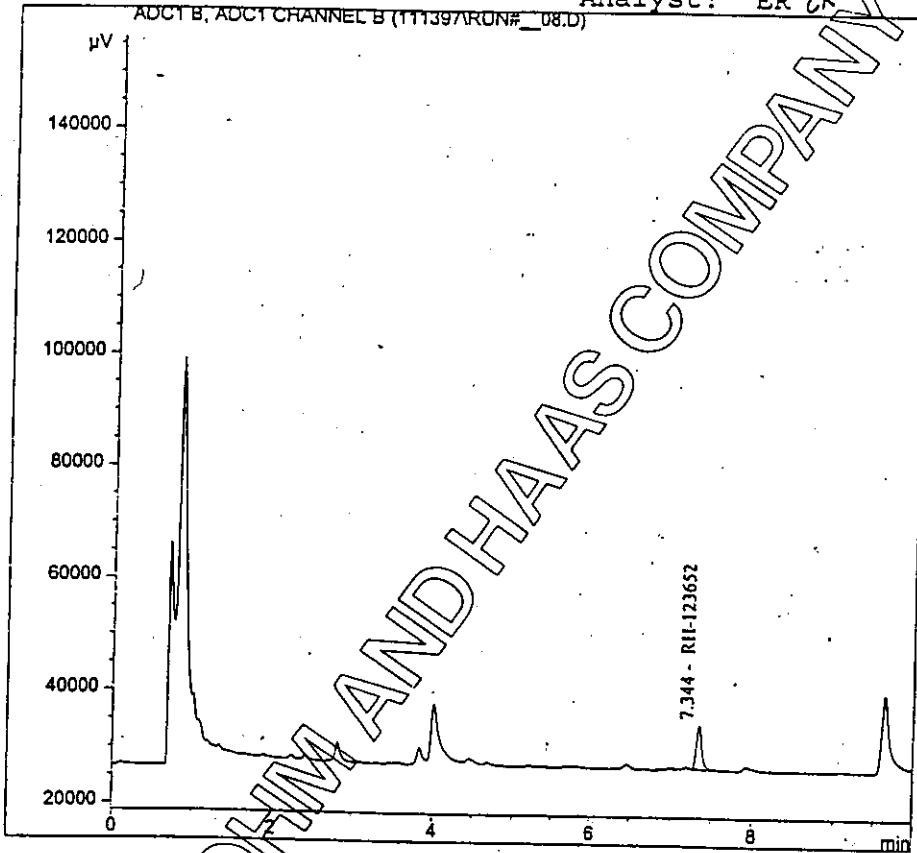
Figure 11. Soil sample fortified with 0.010 ppm monoacid on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 8
 HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
 Sample Info: Spk C1, Wt.= 10 g, FV= 100 mL
 0.5 μ g added

Injected on: 11/13/97 2:07:34 PM
 Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.34	RH-123652	7943	38442

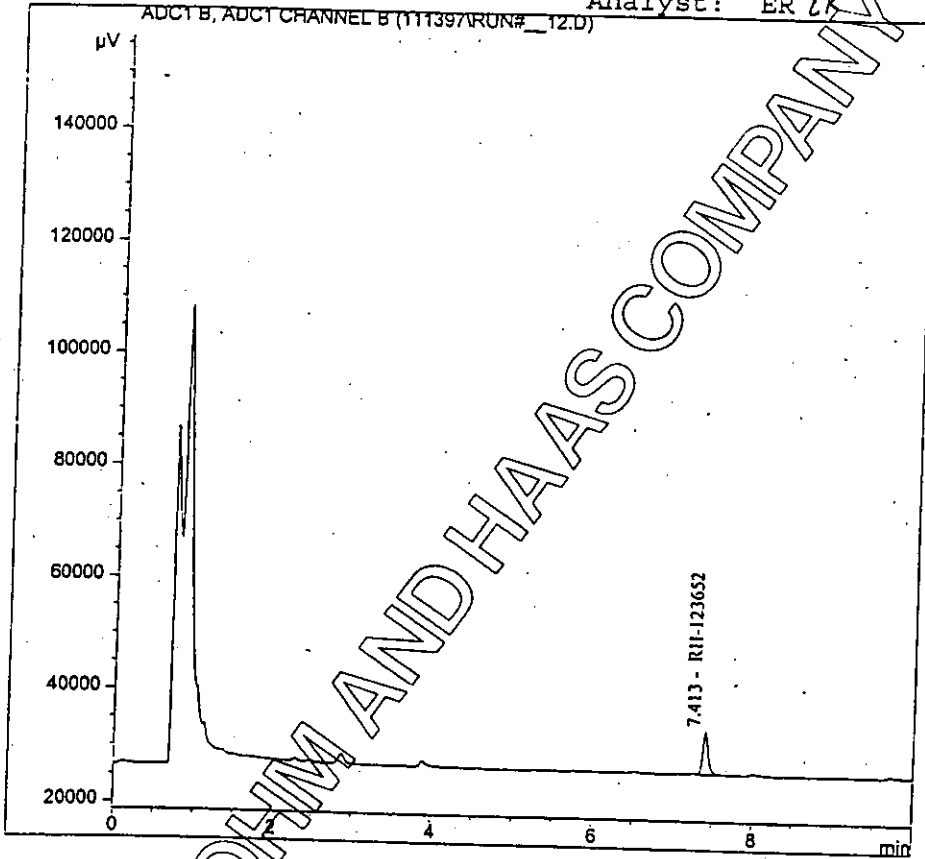
Figure 12. Soil sample fortified with 0.050 ppm monoacid on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 12
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: HB-1
Sample Info: Spk E1, Wt.=10 g, FV=200 mL
1.0 μ g added

Injected on: 11/13/97 3:48:36 PM
Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.41	RH-123652	7503	36856

Figure 13. Soil sample fortified with 0.10 ppm monoacid on GC/ECD.

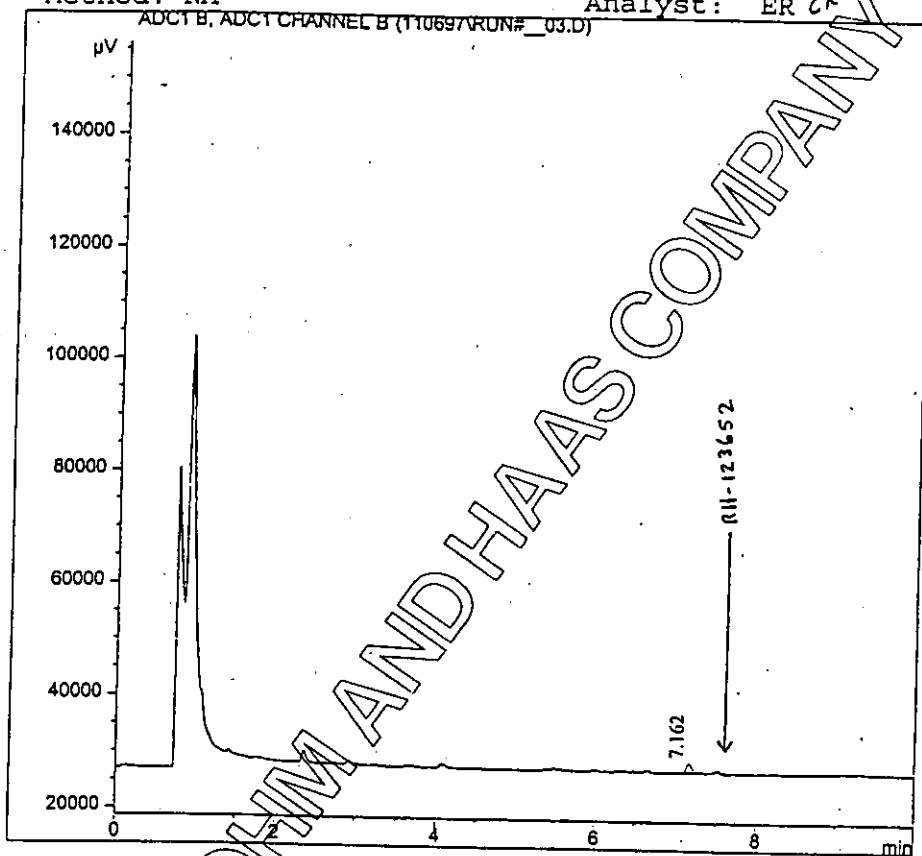
Analysis of Thiazopyr

Run No.: 3

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
 Sample Info: Blk, Sample Wt. = 100 mL, FV= 5 mL.

Injected on: 11/6/97 10:09:42 AM
 Method: NA Analyst: ER ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.16		1765	9228
0.00	RH-123652	0	0

Figure 14. Thiazopyr waterl control on GC/ECD.

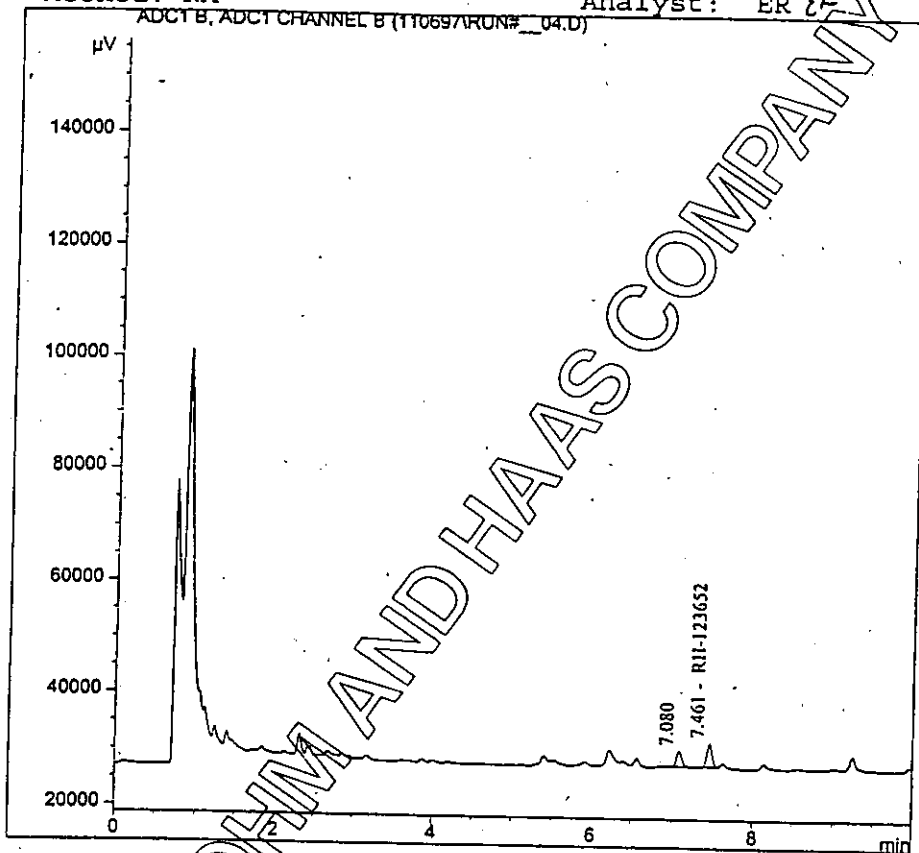
Analysis of Thiazopyr

Run No.: 4

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
 Sample Info: Spk A, Sample Wt. = 100 mL, FV = 5 mL
 0.01 μ g added

Injected on: 11/6/97 10:34:59 AM
 Method: NA Analyst: ER *ER*



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.08		2854	13681
7.46	RH-123652	4268	18915

Figure 15. Water sample fortified with 0.10 ppb thiazopyr on GC/ECD.

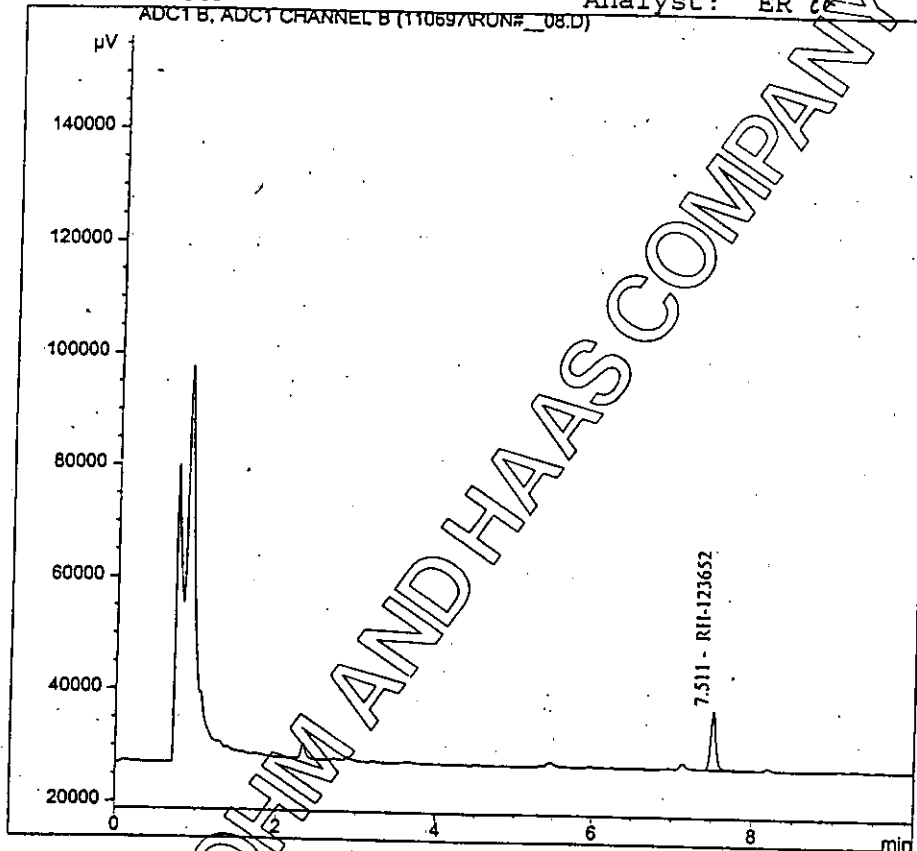
Analysis of Thiazopyr

Run No.: 8

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
 Sample Info: Spk C1, Wt. = 100 mL, FV = 10 mL
 0.05 μ g added

Injected on: 11/6/97 12:15:59 PM
 Method: NA Analyst: ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.51	RH-123652	10445	49539

Figure 16. Water sample fortified with 0.50 ppb thiazopyr on GC/ECD.

Analysis of Thiazopyr

Run No.: 12

HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

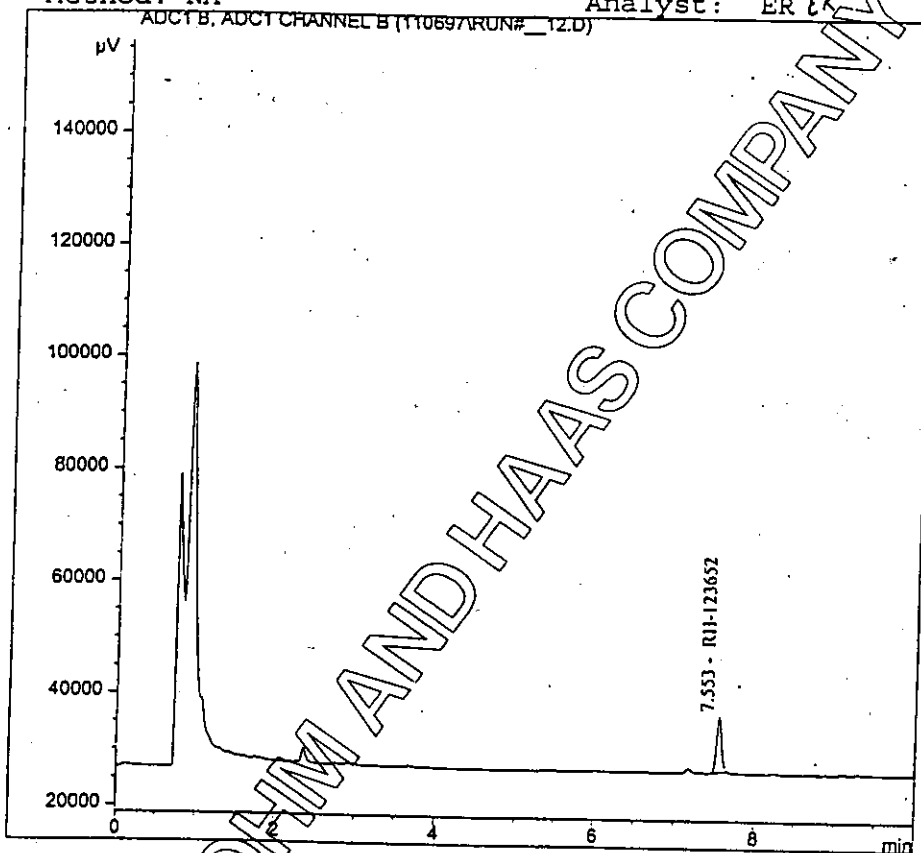
Sample No: 975898
 Sample Info: Spk E1, Wt.=100 g, FV=20 mL
 0.1 μ g added

Injected on: 11/6/97

1:56:59 PM

Method: NA

Analyst: ER *ll*



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.55	RH-123652	9848	43751

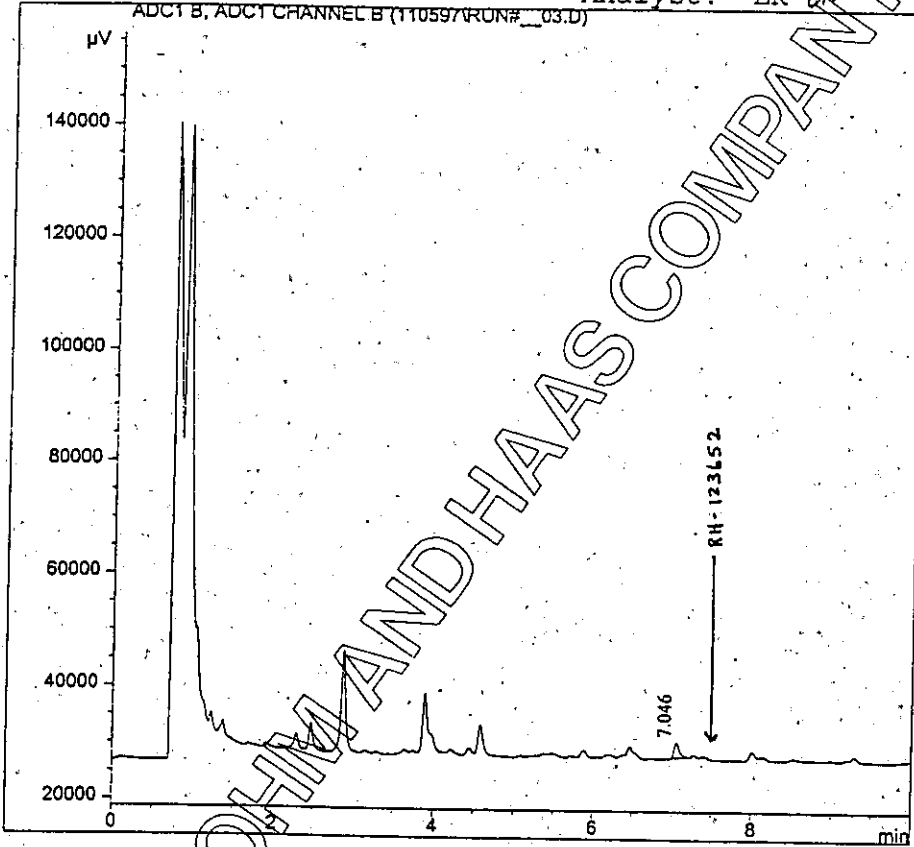
Figure 17. Water sample fortified with 1.0 ppb thiazopyr on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 3
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj. Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
Sample Info: Blk, Sample Wt. = 100 mL, FV= 5 mL

Injected on: 11/5/97 5:50:19 PM
Method: NA Analyst: ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.05		2682	12500
0.00	RH-123652	0	0

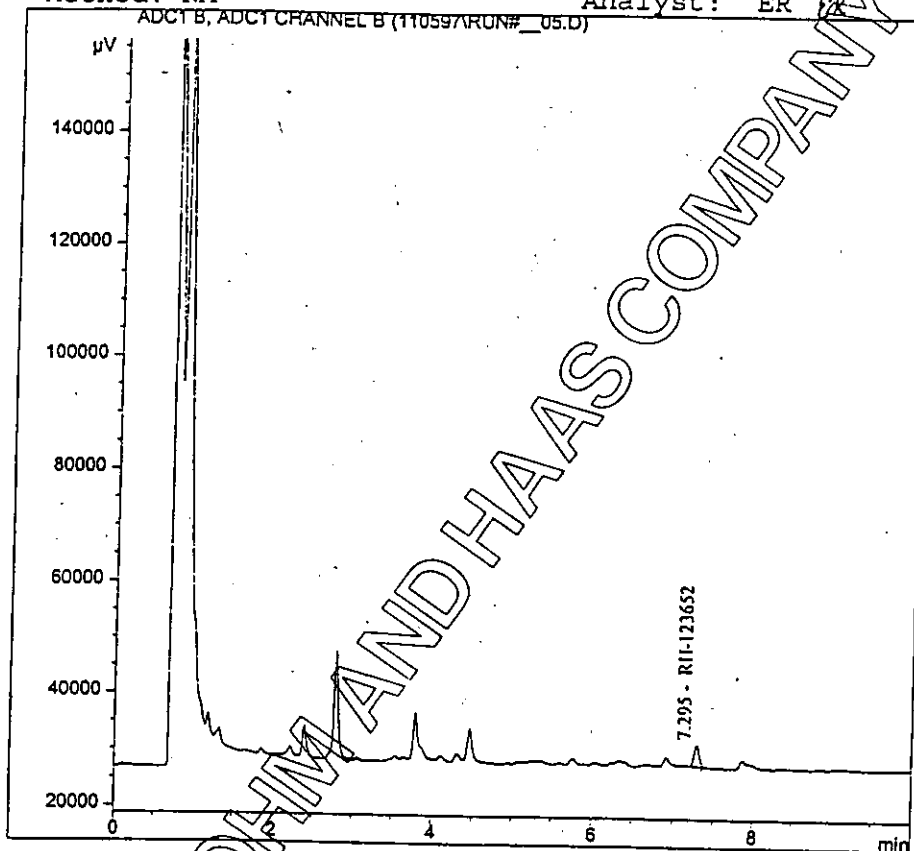
Figure 18. Monoacid water control on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 5
 HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m
 Carrier Gas: He, 10 mL/min. Inj, Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
 Sample Info: SPKB, Sample Wt. = 100 mL, FV = 5 mL
 0.01 μ g added

Injected on: 11/5/97 6:40:52 PM
 Method: NA Analyst: ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.30	RH-123652	3920	18071

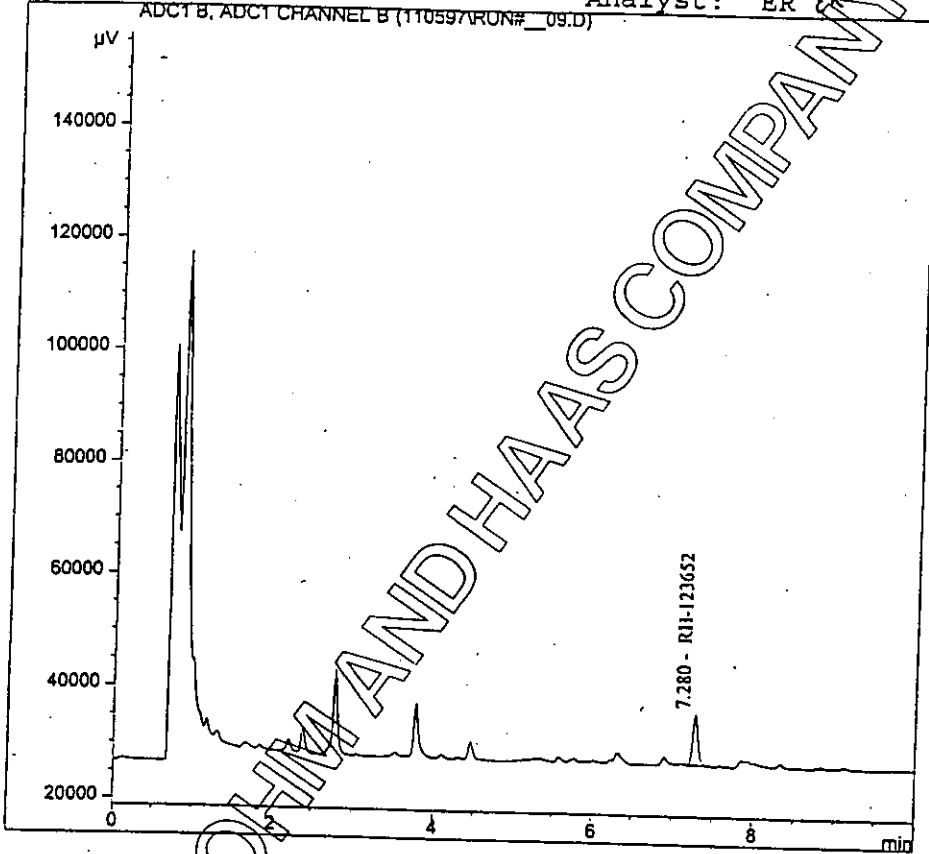
Figure 19. Water sample fortified with 0.10 ppb monoacid on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 9
 HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
 Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
 Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 4 min.,
 to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
 Sample Info: Spk D Wt. = 100 mL, FV = 10 mL
 0.05 μ g added

Injected on: 11/5/97 8:21:56 PM
 Method: NA Analyst: ER



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.28	RH-123652	9117	42150

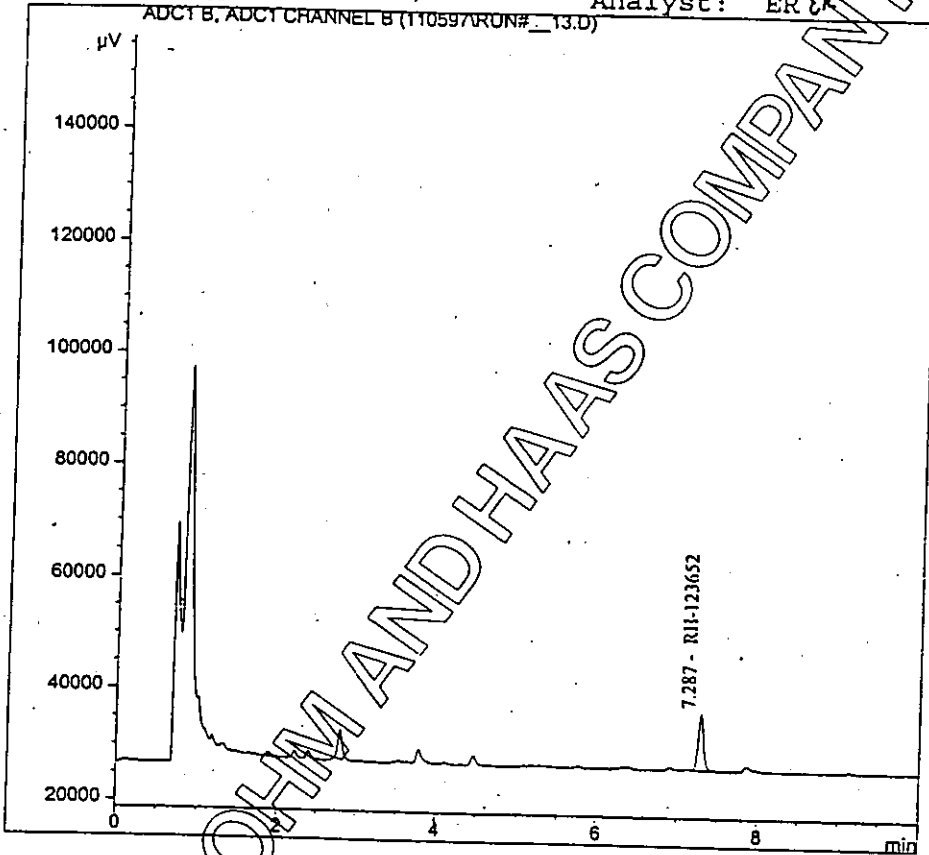
Figure 20. Water sample fortified with 0.50 ppb monoacid on GC/ECD.

Analysis of Monoacid (converted to Thiazopyr)

Run No.: 13
HP 5890 equ. with ECD. Column: RTX-5, 30 m x 0.53 mm x 1.5 μ m.
Carrier Gas: He, 10 mL/min. Inj. Temp.: 250°C.
Det. Temp. 300°C. Inj Vol. 5 μ L. Column Temp. 160°C for 1 min.,
to 260°C at 6°C/min., hold 5 min.

Sample No: 975898
Sample Info: Sample Wt. = 100 mL, FV = 20 mL
0.1 μ g added

Injected on: 11/5/97 10:02:53 PM
Method: NA Analyst: ER & K



Ret. Time (Min)	Compound Name	Peak Height (μ V)	Peak Area (μ V*s)
7.29	RH-123652	10008	45992

Figure 21. Water sample fortified with 1.0 ppb monoacid on GC/ECD.

File : C:\HPCHEM\1\DATA\N0797C\N0701.D
Operator : AS
Acquired : 7 Nov 97 4:02 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Thiazopyr Std. 0.005ug/mL, C110797-4
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 1

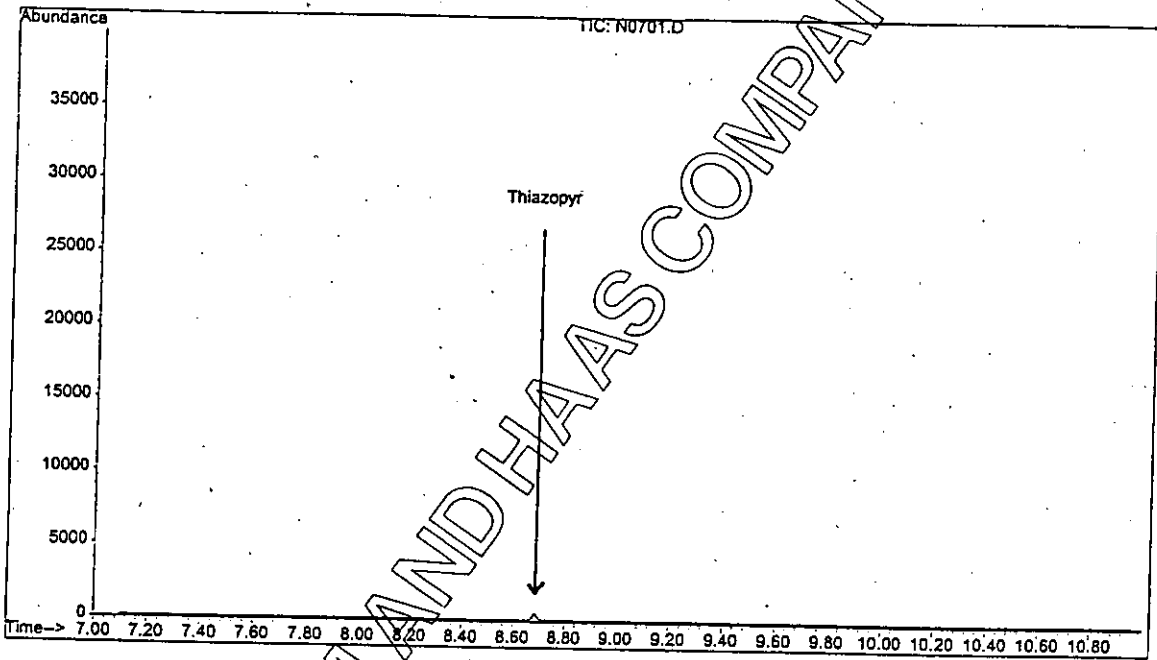


Figure 22. 0.0050 ppm thiazopyr standard on GC/MSD.

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File : C:\HPCHEM\1\DATA\N0797C\N0710.D
Operator : AS
Acquired : 7 Nov 97 6:50 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Thiazopyr Std. 0.01ug/mL, C110797-3
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 8

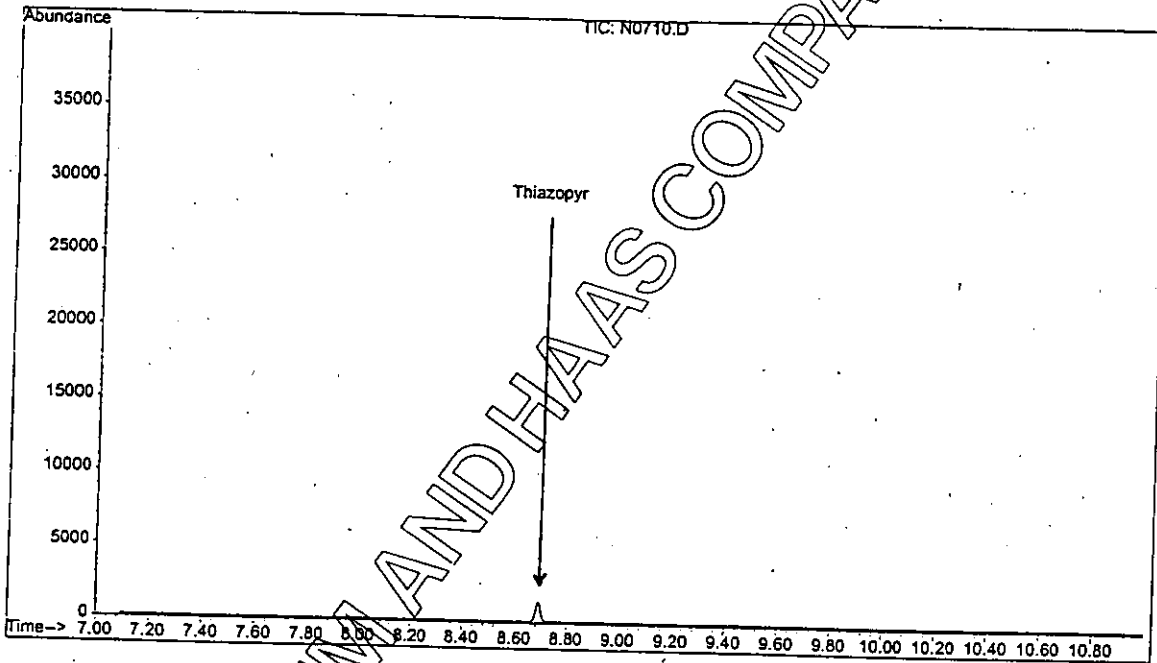


Figure 23. 0.010 ppm thiazopyr standard on GC/MSD.

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File : C:\HPCHEM\1\DATA\N0797C\N0716.D
Operator : AS
Acquired : 7 Nov 97 8:43 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Thiazopyr Std. 0.05ug/mL, C110797-2
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 13

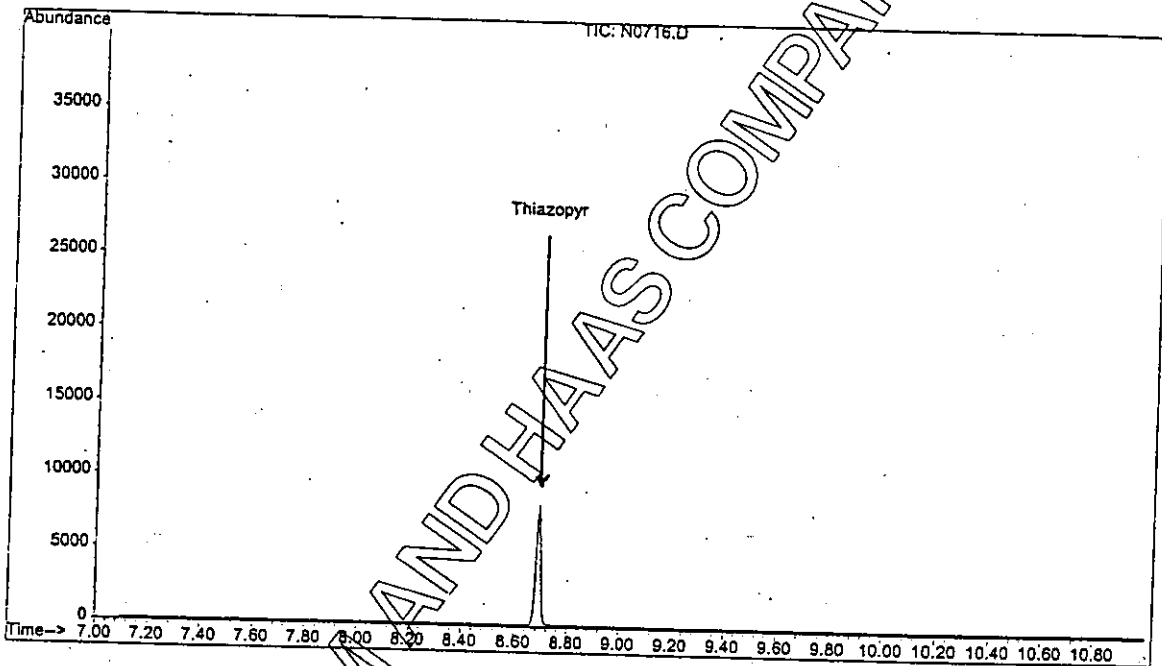


Figure 24. 0.050 ppm thiazopyr standard on GC/MSD.

TR 34-97-175

File : C:\HPCHEM\1\DATA\N0797C\N0722.D
Operator : AS
Acquired : 7 Nov 97 10:35 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Thiazopyr Std. 0.1ug/mL, C110797-1
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 18

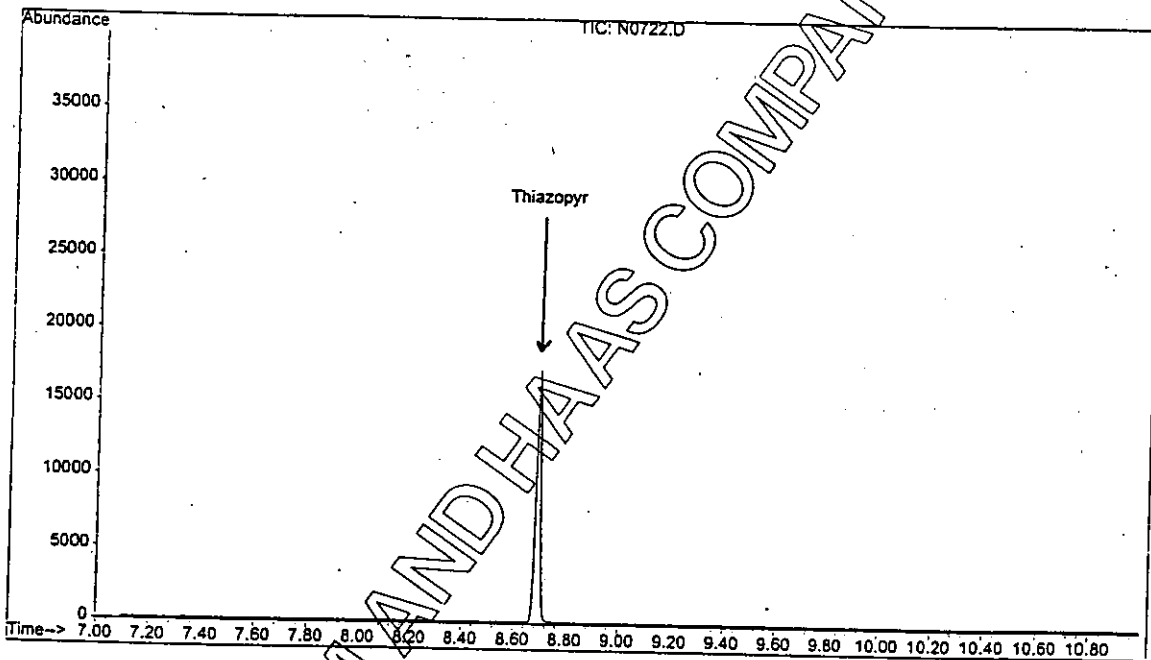
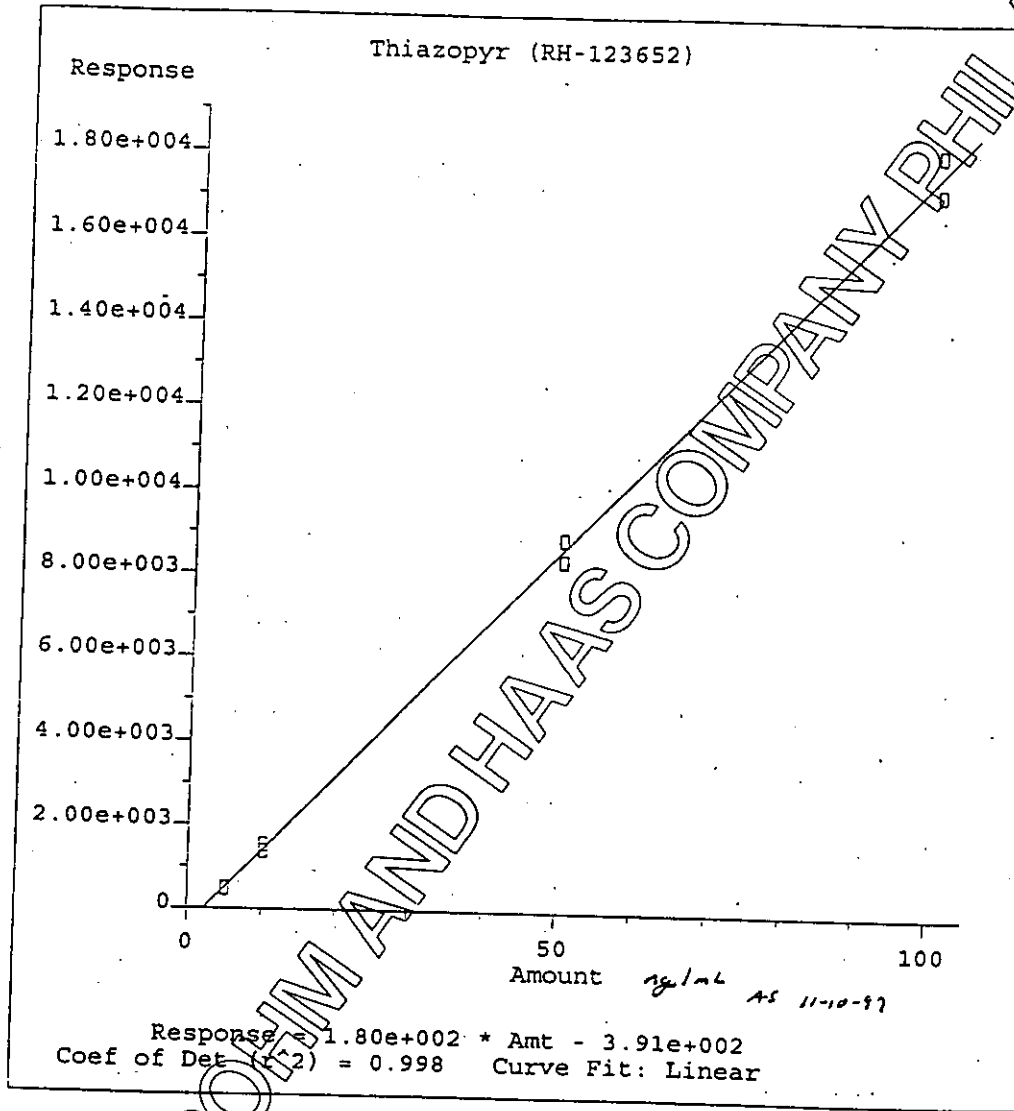


Figure 25. 0.10 ppm thiazopyr standard on GC/MSD.

Protocol No.: NA
CAL Study No.: 002-280
Extraction Date: 11/04/97
Analysis Date: 11/07/97



Method Name: C:\HPCHEM\1\METHODS\ALTHI.M
Calibration Table Last Updated: Mon Nov 10 08:22:41 1997
O.C.R. 11/8/97

Figure 26. Thiazopyr standard calibration curve on GC/MSD.

File : C:\HPCHEM\1\DATA\N1197A\N1103.D
Operator : AS
Acquired : 11 Nov 97 10:35 am using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Blk, HB-1, SW=10g, FV=10mL (T)
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 2

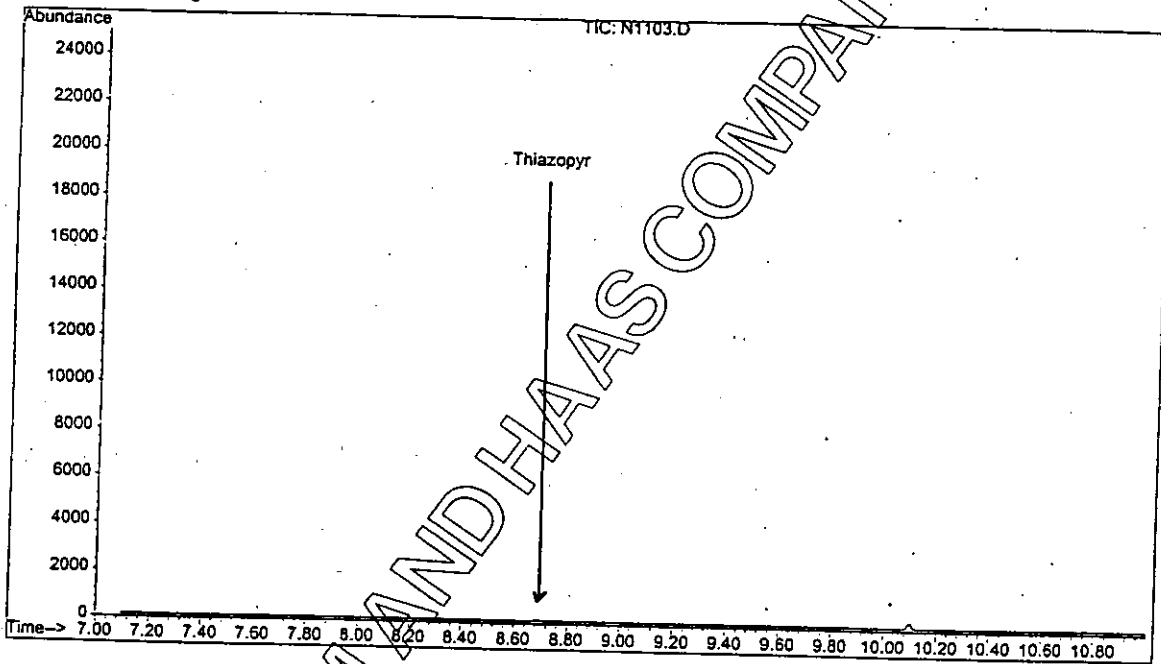


Figure 27. Thiazopyr soil control on GC/MSD.

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File : C:\HPCHEM\1\DATA\N1197A\N1105.D
Operator : AS
Acquired : 11 Nov 97 11:13 am using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk A, HB-1, SW=10g, FV=10mL (T)
Misc Info : CAL# 002-280, Pro# NA; 0.1ug added
Vial Number: 4

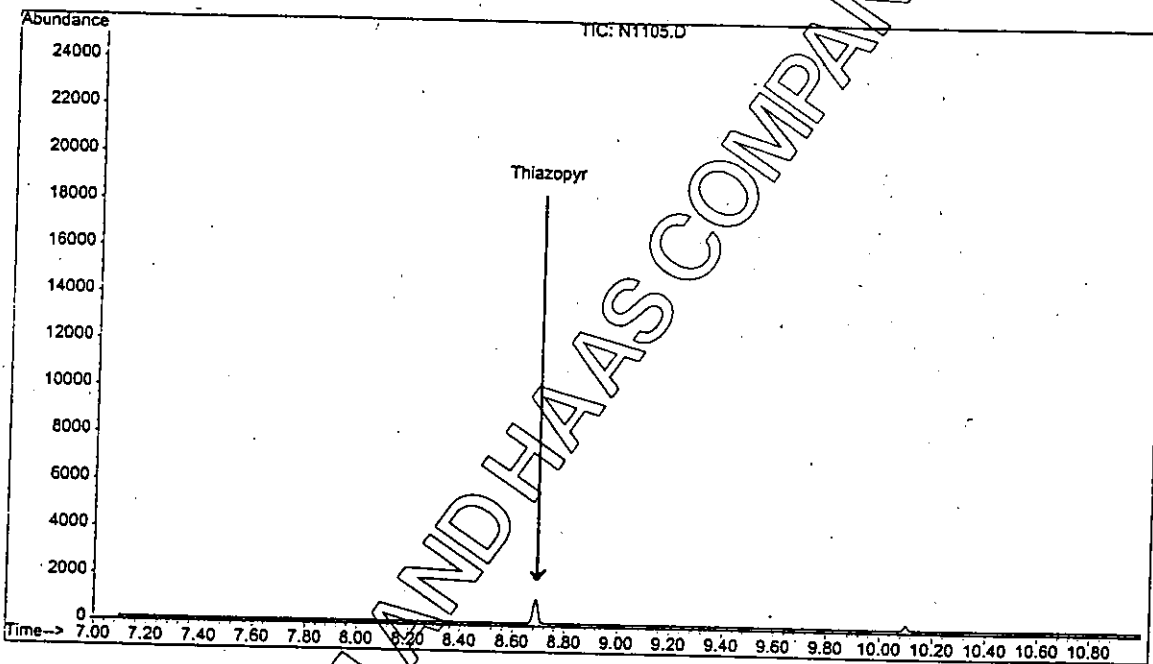


Figure 28. Soil sample fortified with 0.010 ppm thiazopyr on GC/MSD.

File : C:\HPCHEM\1\DATA\N1197A\N1111.D
Operator : AS
Acquired : 11 Nov 97 1:05 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk C, HB-1, SW=10g, FV=10mL (T)
Misc Info : CAL# 002-280; Pro# NA, 0.5ug added
Vial Number: 9

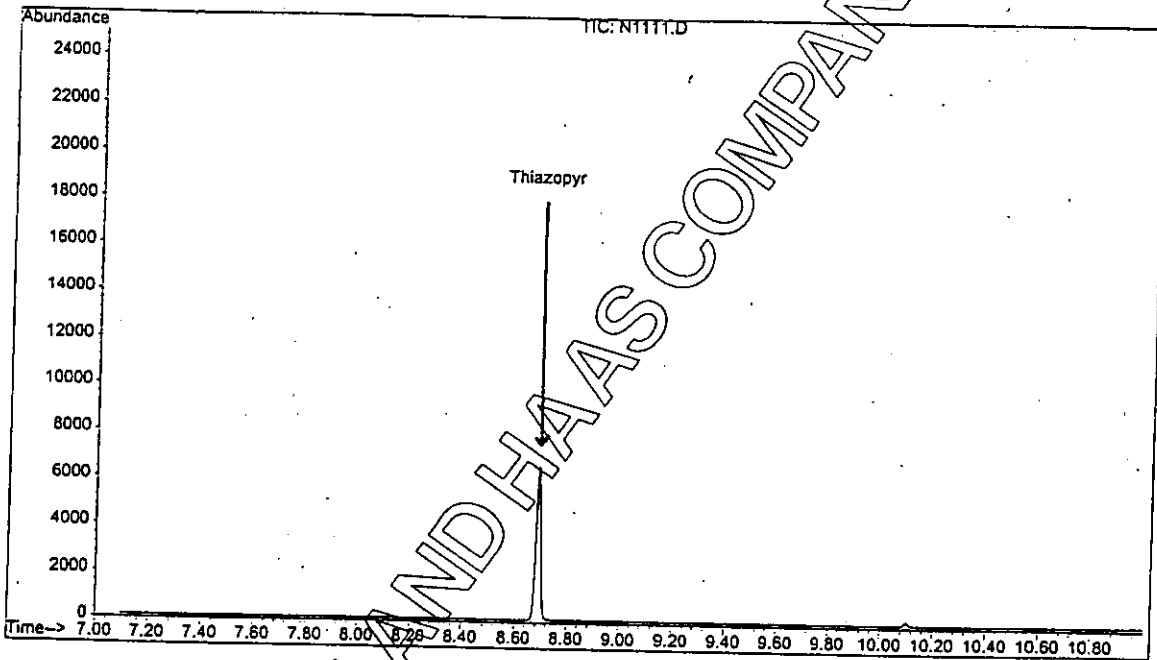


Figure 29 Soil sample fortified with 0.050 ppm thiazopyr on GC/MSD.

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File : C:\HPCHEM\1\DATA\N1197A\N1117.D
Operator : AS
Acquired : 11 Nov 97 2:57 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk E, HB-1, SW=10g, FV=20mL (T)
Misc Info : CAL# 002-280, Pro# NA, 1.0ug added
Vial Number: 14

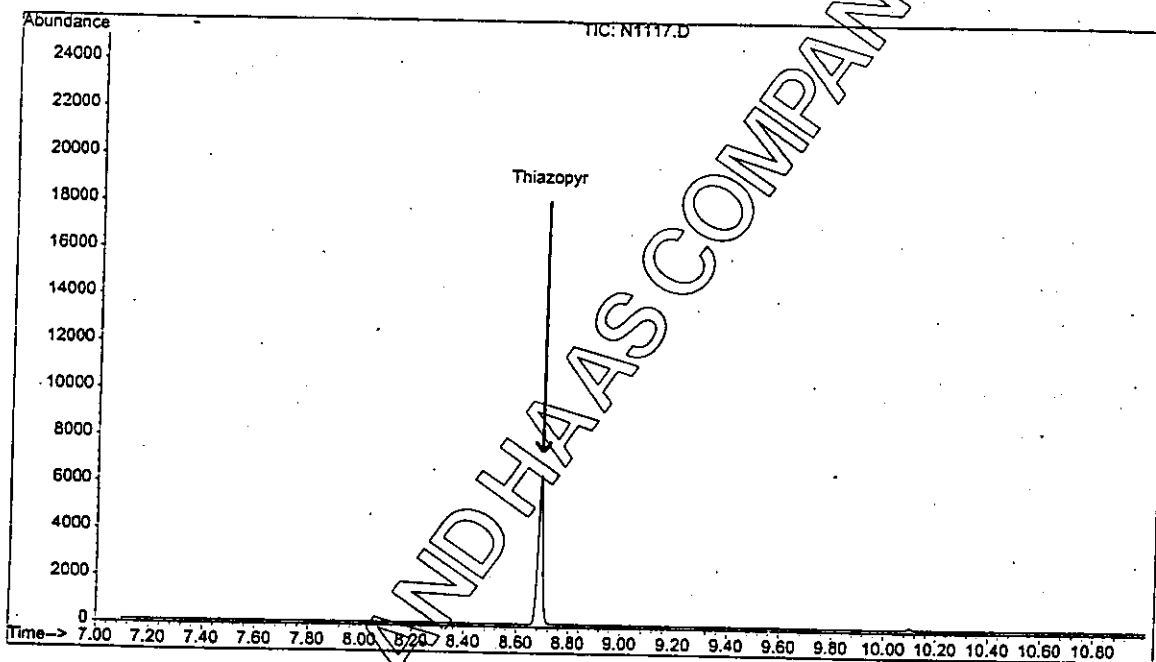


Figure 30 Soil sample fortified with 0.10 ppm thiazopyr on GC/MSD.

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File : C:\HPCHEM\1\DATA\N1197A\N1104.D
Operator : AS
Acquired : 11 Nov 97 10:54 am using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Blk, HB-1, SW=10g, FV=10mL (M)
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 3

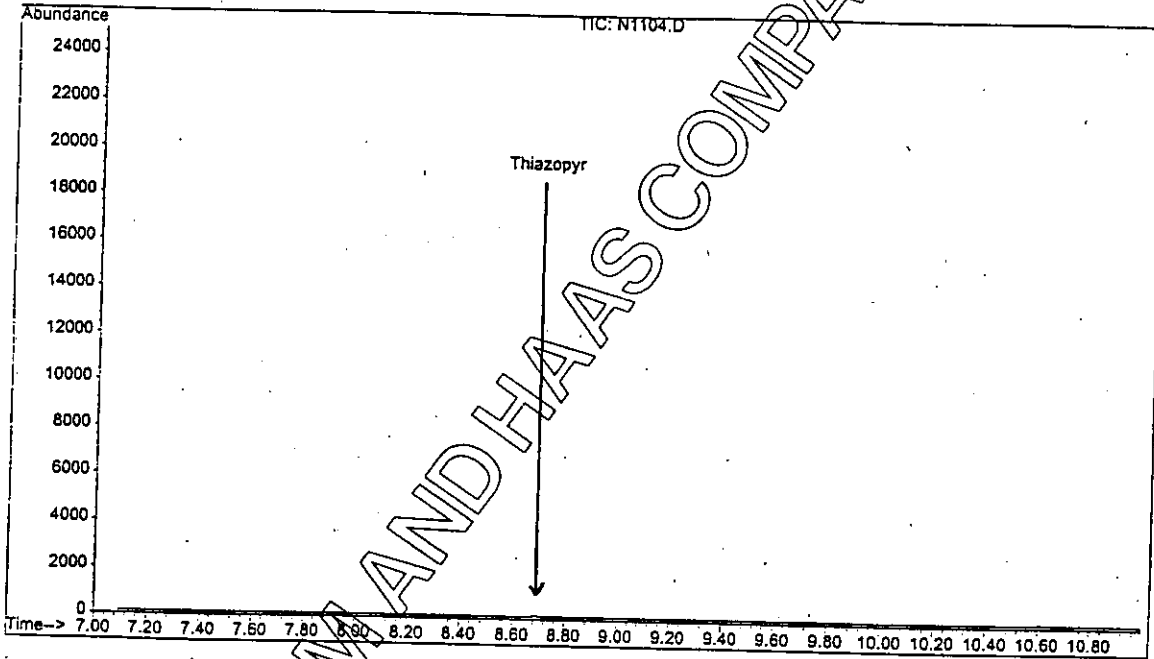


Figure 31. Monoacid soil control on GC/MSD.

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File : C:\HPCHEM\1\DATA\N1197A\N1106.D
Operator : AS
Acquired : 11 Nov 97 11:31 am using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk A, HB-1, SW=10g, FV=10mL (M)
Misc Info : CAL# 002-280, Pro# NA, 0.1ug added
Vial Number: 5

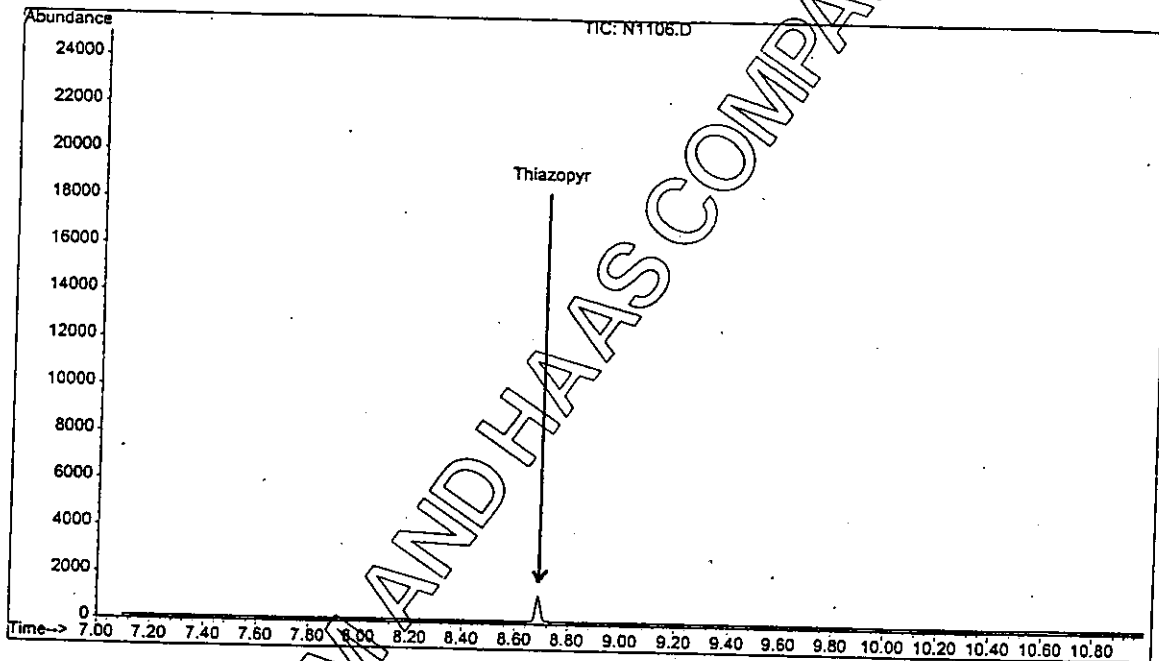


Figure 32 Soil sample fortified with 0.010 ppm monoacid on GC/MSD.

File : C:\HPCHEM\1\DATA\N1197A\N1112.D
Operator : AS
Acquired : 11 Nov 97 1:24 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk C, HB-1, SW=10g, FV=10mL (M)
Misc Info : CAL# 002-280, Pro# NA, 0.5ug added
Vial Number: 10

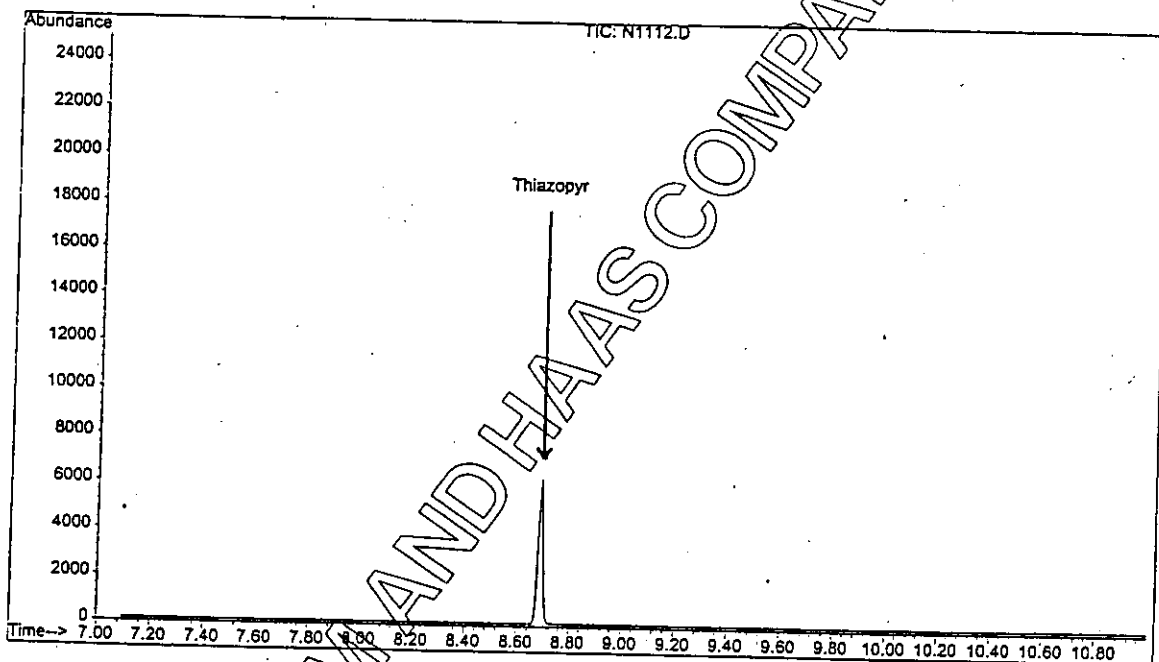


Figure 33 Soil sample fortified with 0.050 ppm monoacid on GC/MSD.

File : C:\HPCHEM\1\DATA\N1197A\N1118.D
Operator : AS
Acquired : 11 Nov 97 3:16 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk E, HB-1, SW=10g, FV=20mL (M)
Misc Info : CAL# 002-280, Pro# NA, 1.0ug added
Vial Number: 15

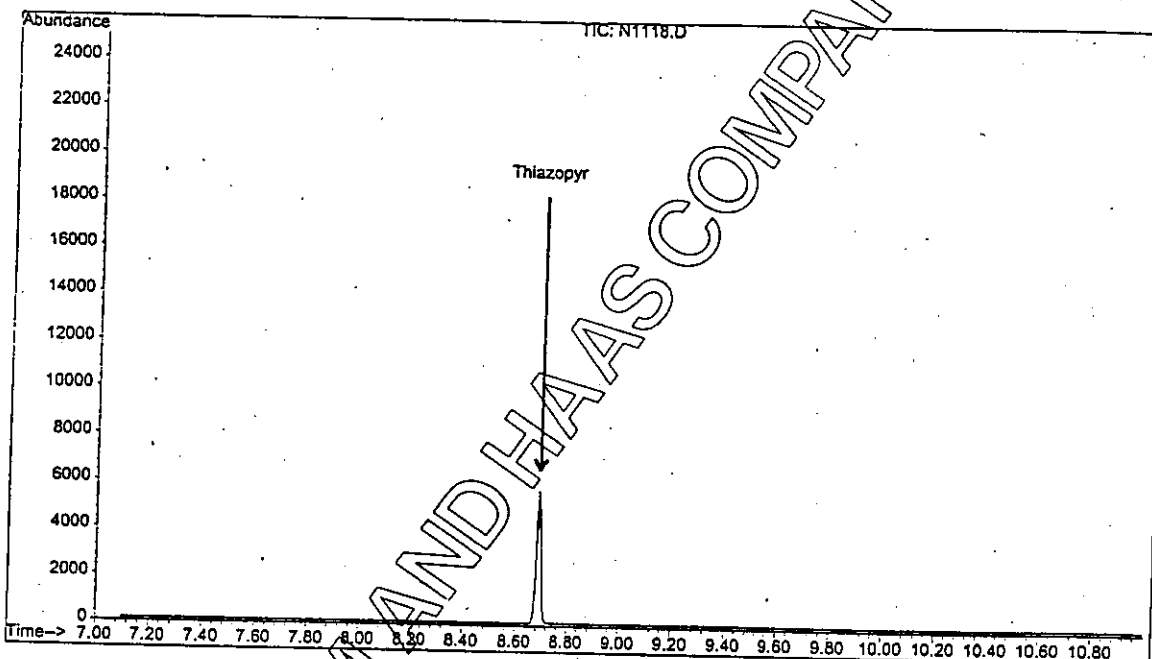


Figure 34 Soil sample fortified with 0.10 ppm monoacid on GC/MSD.

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File : C:\HPCHEM\1\DATA\N0797C\N0703.D
Operator : AS
Acquired : 7 Nov 97 4:39 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Blk, 975898, SW=100mL, FV=1mL (T)
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 2

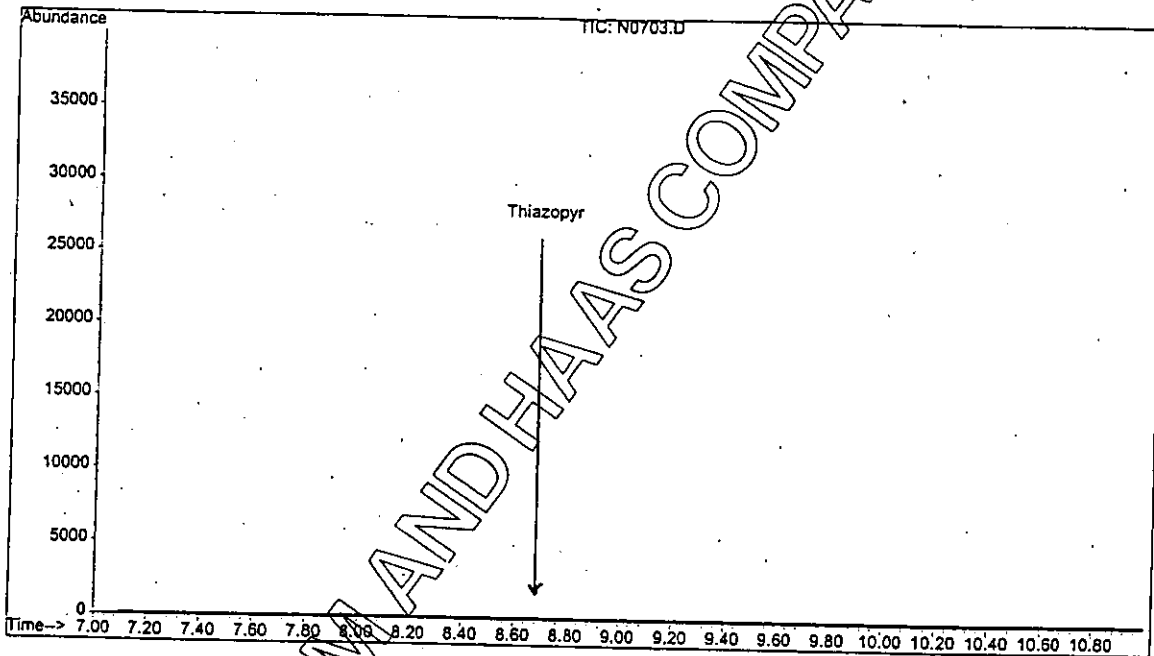


Figure 35. Thiazopyr waterl control on GC/MSD.

File : C:\HPCHEM\1\DATA\N0797C\N0705.D
Operator : AS
Acquired : 7 Nov 97 5:17 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk A, 975898, SW=100mL, FV=1mL (T)
Misc Info : CAL# 002-280, Pro# NA, 0.01ug added
Vial Number: 4

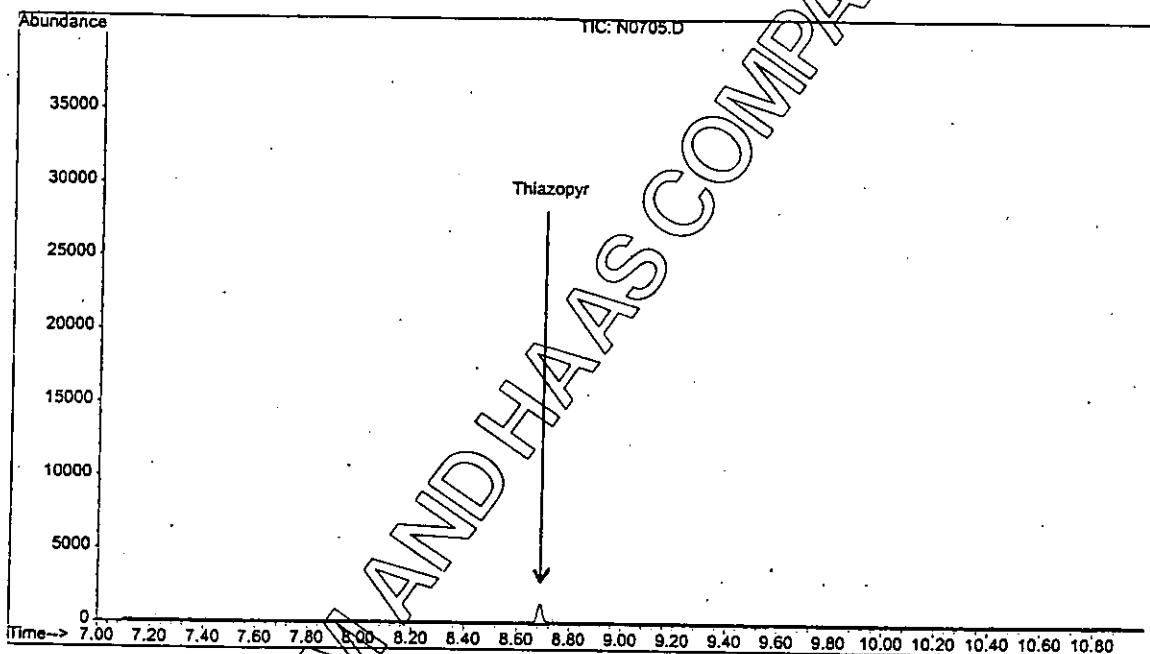


Figure 36 Water sample fortified with 0.10 ppb thiazopyr on GC/MSD.

File : C:\HPCHEM\1\DATA\N0797C\N0711.D
Operator : AS
Acquired : 7 Nov 97 7:09 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk C, 975898, SW=100mL, FV=1mL (T)
Misc Info : CAL# 002-280, Pro# NA, 0.05ug added
Vial Number: 9

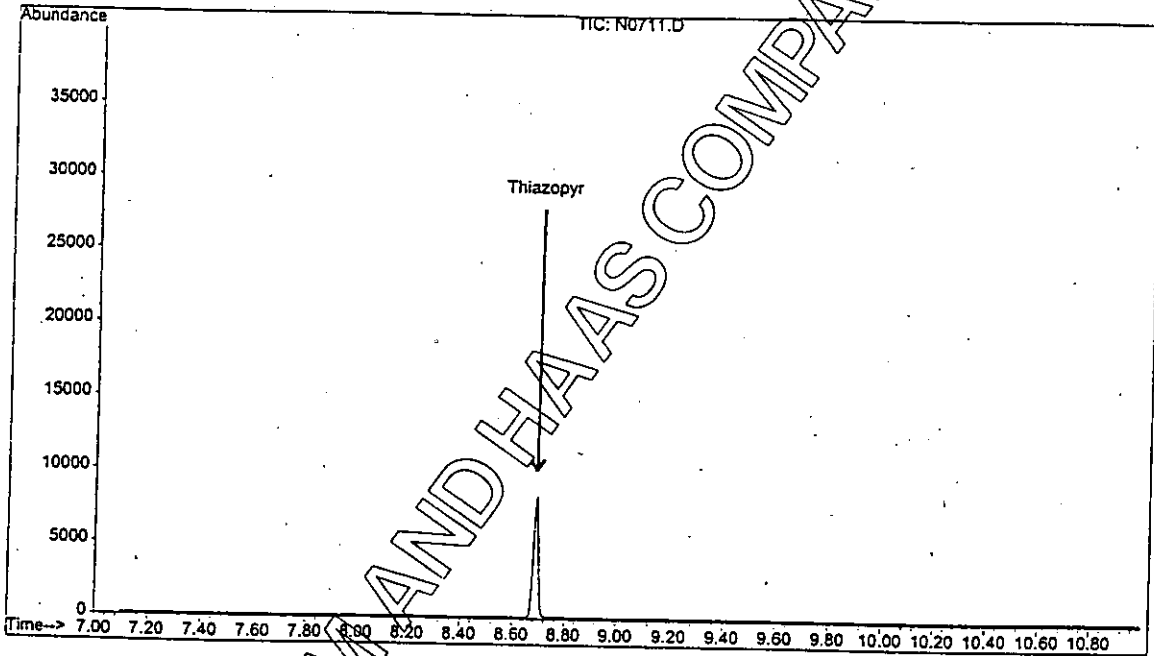


Figure 27 Water sample fortified with 0.50 ppb thiazopyr on GC/MSD.

File : C:\HPCHEM\1\DATA\N0797C\N0717.D
Operator : AS
Acquired : 7 Nov 97 9:02 pm using AcqMethod ALTHI
Instrument : GC/MS 7
Sample Name: Spk E, 975898, SW=100mL, FV=2mL (T)
Misc Info : CAL# 002-280, Pro# NA, 0.1ug added
Vial Number: 14

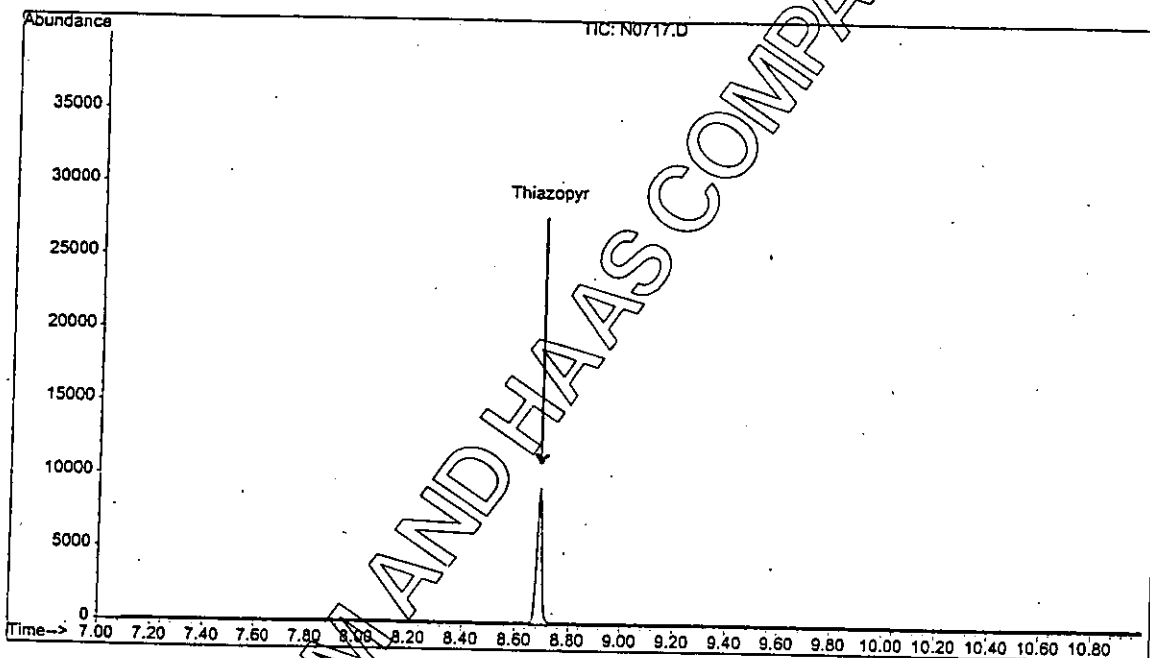


Figure 34. Water sample fortified with 1.0 ppb thiazopyr on GC/MSD.

File : C:\HPCHEM\1\DATA\N0797C\N0704.D
Operator : AS
Acquired : 7 Nov 97 4:58 pm using AcqMethod ALTH
Instrument : GC/MS 7
Sample Name: Blk, 975898, SW=100mL, FV=1mL (M)
Misc Info : CAL# 002-280, Pro# NA
Vial Number: 3

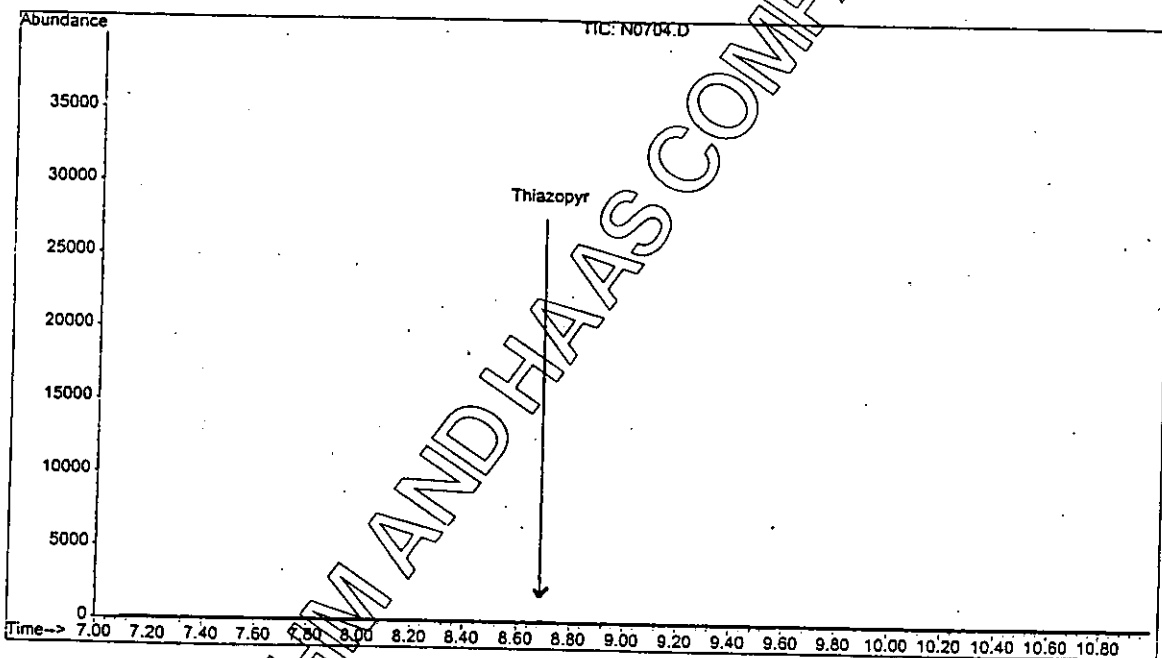


Figure 39. Monoacid water control on GC/MSD.

File : C:\HPCHEM\1\DATA\N0797C\N0706.D
Operator : AS
Acquired : 7 Nov 97 5:36 pm using AcqMethod ALTHI
Instrument : GC/MS 7.
Sample Name: Spk A, 975898, SW=100mL, FV=1mL (M)
Misc Info : CAL# 002-280, Pro# NA, 0.01ug added
Vial Number: 5

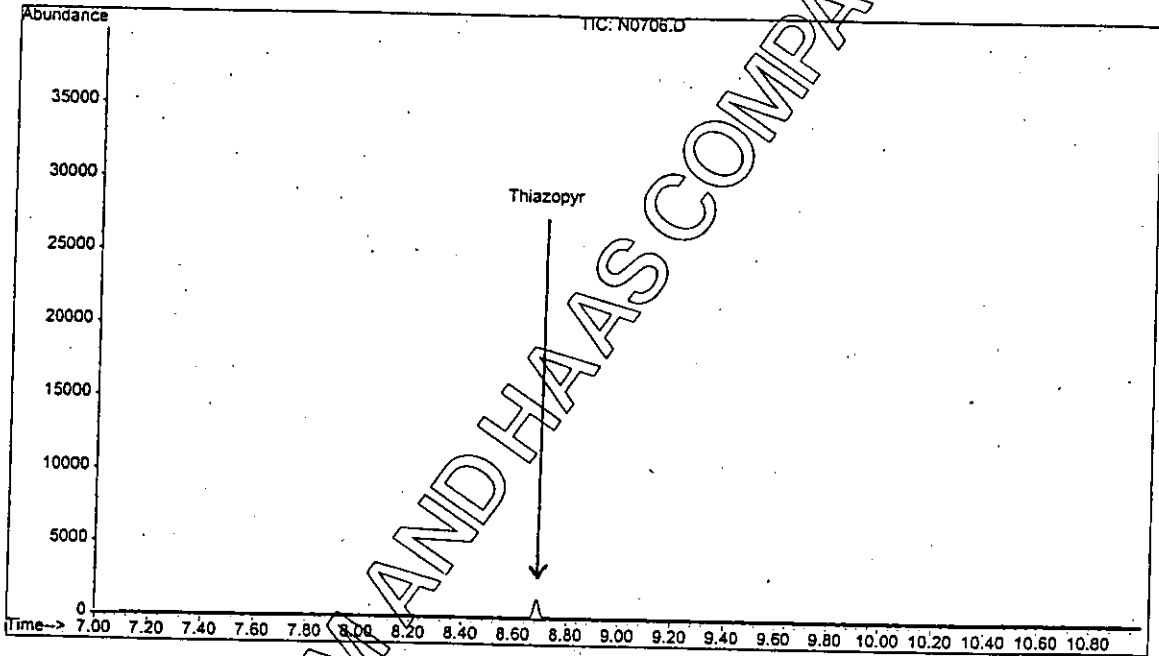


Figure 40. Water sample fortified with 0.10 ppb monoacid on GC/MSD.

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File : C:\HPCHEM\1\DATA\N0797C\N0712.D
Operator : AS
Acquired : 7 Nov 97 7:28 pm using AcqMethod ALTH
Instrument : GC/MS 7
Sample Name: Spk C, 975898, SW=100mL, FV=1mL (M)
Misc Info : CAL# 002-280, Pro# NA, 0.05ug added
Vial Number: 10

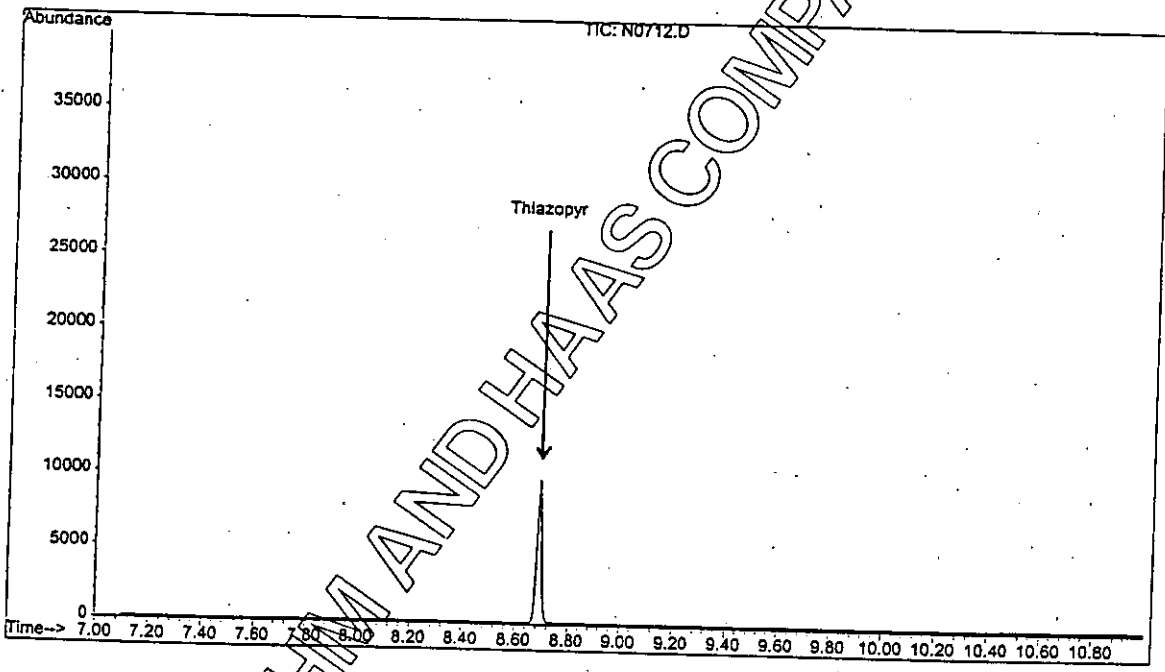


Figure 4. Water sample fortified with 0.50 ppb monoacid on GC/MSD.

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File : C:\HPCHEM\1\DATA\N0797C\N0718.D
Operator : AS
Acquired : 7 Nov 97 9:20 pm using AcqMethod ALTHY
Instrument : GC/MS 7
Sample Name: Spk E, 975898, SW=100mL, FV=2mL (M)
Misc Info : CAL# 002-280, Pro# NA, 0.1ug added
Vial Number: 15

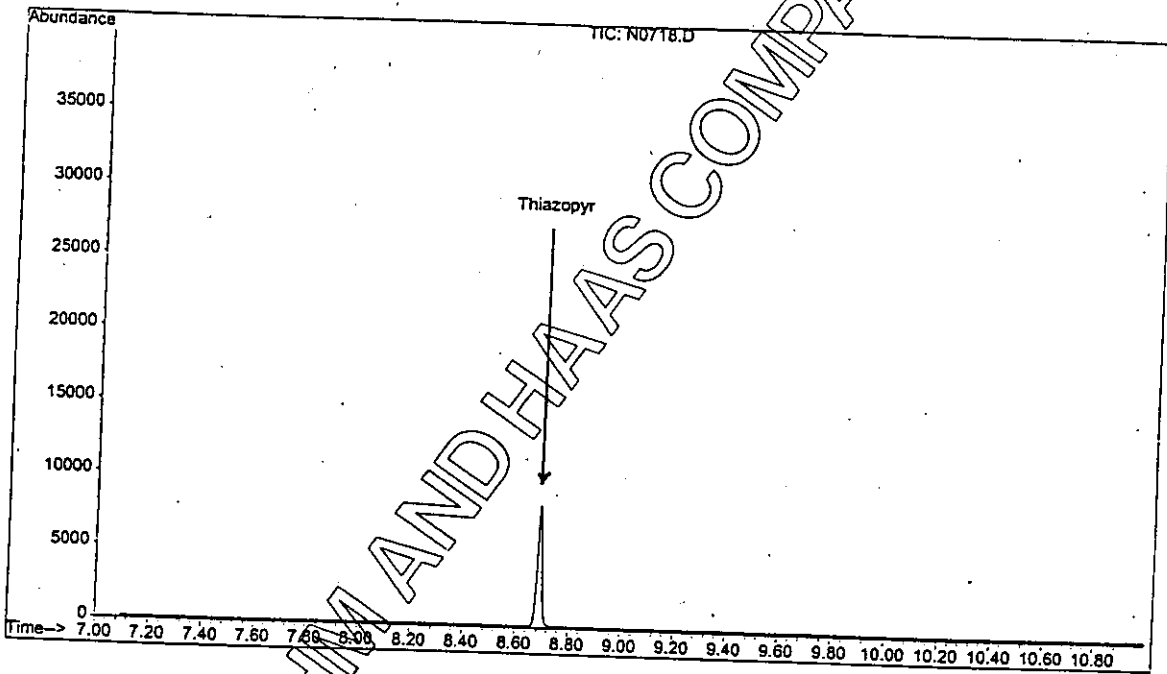


Figure 12 Water sample fortified with 1.0 ppb monoacid on GC/MSD.