PAI

PROFESSIONAL ANALYSIS, INC.

2155 Louisiana Blvd., NE, Suite 2100
Albuquerque, New Mexico 87110

Telephone (505) 883-0942
Fax (505) 883-1840

4/4/95

Gary Estepp
IRC
2618 Coors SW
Albuquerque, NM 87105

Re: GC Analysis of extracted wipe test samples

Dear Gary,

Gas Chromatograph FID analysis of sanded surface wipe test sample indicates that the peaks of concern are volatile in nature and therefore not pesticides. Furthermore, the peaks that are not volatile and which may be pesticide seem to be below 10 ppm.

A complete report for both the HPLC and GC analysis will follow.

Sincerely,

Greg Bybee

PAI

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IRC,inc
2618 Coors SW
Albuquerque, NM 87105

Dear Gary,

Using recycled material in any process present some unique challenges. A frequent concern is ensuring hazardous residue is not present in the final product. IRC contacted PAI to provide a simple, cost effective test method to check for pesticide residue on the sutface of their recycled plastic products. Given the cost constraints and the client request for a qualitative result, PAI employed a surface wipe method and HPLC (High Performance Liquid Chromatography) analysis.

The wipe procedure consisted of wiping the surfaces of two IRC products (one sanded, one non-sanded) with a cotton swab. Extractable matter was then removed with a 10% actone 90% methanol solution. The extracts were then injected on a isocratic Spectra Physics SP 8810 HPLC system with a reverse phase isothermal column and uv detector. Appropriate solvent, swab and trip blank samples were also extracted and analyzed.

The results of the HPLC analysis of the two IRC samples were as follows:

Sample ID	# of peaks	Retention time (min)
Non-sanded	3	2.07,2.36,5.68
Sanded	4	2.18,2.40,2.88,5.70

Based on prior HPLC analysis of Pyrethrin I&II and Piperonyl Butoxide insecticides, the detected peaks seem to be less than 10ppm (with the exception of the sanded surface sample peak 2.18 and 2.40). However, due to various detector responses for differnt compounds and the unknown nature of detected compounds, the true values can not be precisely determined.

In order to help identify the larger peaks (as either volatile or nonvolatile and therefor a pesticide) the samples will be injected on a Gas Chromatograph FID at no additional charge.

Sincerely,

Greg Bybee

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WIPE TEST ANALYSIS REPORT

1.0 INTRODUCTION

Using recycled material in any process presents some unique challenges. A frequent concern is ensuring hazardous residue is not present in the final product. IRC contacted PAI to provide a simple, cost effective test method to check for pesticide residue on the surface of their recycled plastic products. Given the cost constraints and the client request for a qualitative result, PAI employed a surface wipe method and HPLC (High Performance Liquid Chromatography) analysis.

2.0 PAI HPLC SYSTEM

2.1 Configuration

The HPLC system is composed of the following components:

Spectraphysics 8815 Isocratic High Pressure Pump
Rhedyne 7125 Injector Valve with 20ul loop
Timberline Prototype Column Oven at 30.0 C
Jones 25cm x 4.6cm C18 (5um) separation column
Linear Model 200 UV/VIS Detector
Spectraphysics SP4400 Intergrator
Generic Computer(386SX 25Mhz)

2.2 Standards and Sample Preparation

No standards were used for this qualitative test.

Wipe test samples were processed for analysis using the following procedure:

1. Wipe the surfaces of the item to be tested with a clean, dry cotton swab.
2. Place swab in a clean, sealed glass vial.
3. Extractable matter was removed from swab with a 10% acetone 90% methanol solution (5ml total).
4. The swab/extract solution was sonicated for 10 min. to aid in the removal of any matter from swabs.
5. Approximately 3ml of extract was then removed and filtered (Acrodisc CR PTFE 0.2um) to remove any particulate.

6. Filter sample into a small vial and mark vial with sample number.
7. Appropriate solvent,swab and trip blank samples were also extracted using the above method.

3.0 ANALYSIS PROCEDURE

3.1 HPLC Analysis Procedure

Liquid Chromatographic Parameters

Column	Jones 25cm x 4.6cm C18
Mobile Phase	80% Methanol/20% Water
Flow Rate	1.0 ml/min
Detector Sensitivity	0.2 AUFS
Volume Injected	50ul

3.2 Gas Chromatograph Procedure

Gas Chromatographic Parameters

Perkin Elmer Sigma I GC	
Column	J&W DB-5(30 m)
Detector	FID
Initial Temp	35 C
Final Temp	150 C

4.0 SUMMARY OF THE HPLC ASSAYS

SAMPLE ID	# OF PEAKS	RETENTION TIME (min)
Non-sanded	3	2.07, 2.36, 5.68
Sanded	4	2.18, 2,40, 2.88, 5.70

Based on prior HPLC analysis of Pyrethrin I & II and Piperonal Butoxide insecticides, the detected peaks seem to be less than 10ppm (with the exception of the sanded surface sample peak 2.18 and 2.40). However, due to variable detector responses for different compounds and the unknown nature of detected compounds, the true concentrations can not be precisely determined.

In order to help identify the larger peaks (as either volatile or nonvolatile and therefore probably not pesticides) both samples were shot on a Gas Chromatograph. All peaks had very short retention times, indicating high volatility. For this reason it is highly unlikely that they represent pesticide residue.

5.0 RECOMANDATIOM

After the initial analysis of IRC products, we at PAI feel that a quality assurance program should be set up utilizing HPLC to monitor for the presents of any pesticide residues. Part of this program would include identification of any pesticides which may be part of the recycled stream. Quantitative as well as qualitative analysis would be part of this program. The use of EPA approved standards would be needed for this program.

Innovative Recycling Corporation
2618 Coors Blvd. SW
Albuquerque, New Mexico 87121

Purchase Order

DATE	P.O. NUMBER
3/29/95	1

VENDOR

PAI
2155 Louisiana Blvd. NE
Suite 2100
Albuquerque, NM 875110

SHIP TO

Innovative Recycling Corporation
2618 Coors Blvd. SW
Albuquerque, New Mexico 87121

EXPECTED	FOB
3/29/95	

ITEM	DESCRIPTION	QTY	RATE	AMOUNT
200	Testing: wipe samples using high performance liquid chromatography	2	125.00	250.00

Pd. 4/24/95
Ck. 1182

Total $250.00

000294

172/175

Analysis of Polymers in Plastic Wood

by

Jim Machir

Brigham Young University
Analyses

Pro. A. Brent Strong
MFE 355, Polymer Processing
December 7, 1994

Analysis of Polymers in Plastic Wood

Introduction

Researchers developed plastic wood within the last four years. Due to its early popularity, a council has been recently organized to determine uses and standards. The council is comprised of men and women in industry who wish to see the full potential of plastic wood recognized. The American Society of Testing and Measurements (ASTM) also recognizes the potential of plastic wood and has developed new standards for testing this year. These organizations and the availability of these tests encourage further development and research for plastic wood products.

Some of the tests deal with environmental and health issues. This report briefly discusses test data which was collected by mass spectroscopy in order to determine the composition of gaseous releases of a burning sample. Since copies of the ASTM Standards for Plastic Wood were unavailable, a different test procedure was created (Appendix pg. 1-2). The professional accuracy of this test is questionable , but the outlined procedure was performed with as much precision as possible. The results indicate low toxicity for the burning sample which produces eye and throat irritation but would only be a major health hazard in large doses.

Problem

Plastics are invading markets traditionally held by glass and metals. This is true because of the ease of manufacturing plastics, as well as the material's high strength-to-weight ratio. Unfortunately this huge output of material generates new waste levels of disturbing proportions. Although some waste is quickly salvaged at the source (when trim and flashing are used as regrind), much concern surrounds plastic that has already entered the waste stream. This plastic requires more effort to recover but is feasible to reuse. One use is plastic wood. Synthetic wood is being used in a number of places

(landscape timbers, fencing, pylons, etc.) where environmental affects would normally age or rot natural wood.

Choices

The ability to analyze the chemical nature of plastics recovered from the waste stream is important. For plastic wood, material type is determined to predict property characteristics, to design compatible processes, and to understand health and safety factors. The analysis is not limited to one general test; many different techniques are available to determine the type of plastic and its properties. Some tests can be simple and quick if the plastic being considered is homogeneous. However, if the plastics are mixed in a compound more complex testing is required. Some tests are listed in Table 1:

Gas Chromatography	Fractionation
C^{13} NMR Spectroscopy	IR Spectroscopy
Pyrolysis-Gas Chromatography	Mass Spectrometry
Packed Column Chromatography	Thermal Degradation
Particle Induced X-Ray Diffraction	Differential Calorimetry
Electron Spin Resonance Spectroscopy	Proton NMR Spectroscopy
High Pressure Liquid Chromatography	

Table 1: Selected Analytical Tests Usable for Plastic Identification

Process Explanation

Other than plastic identification, mass spectrometry has the added potential of being able to determine the quantitative amounts of plastic. The mass spectrometer bombards molecules while in the vapor phase with a high-intensity electron beam. The machine records the result of these impacts on the basis of mass/charge. Different molecular arrangements produce characteristic peaks that can be identified by a trained technician. Figure 1 shows a diagram of a mass spectrometer.

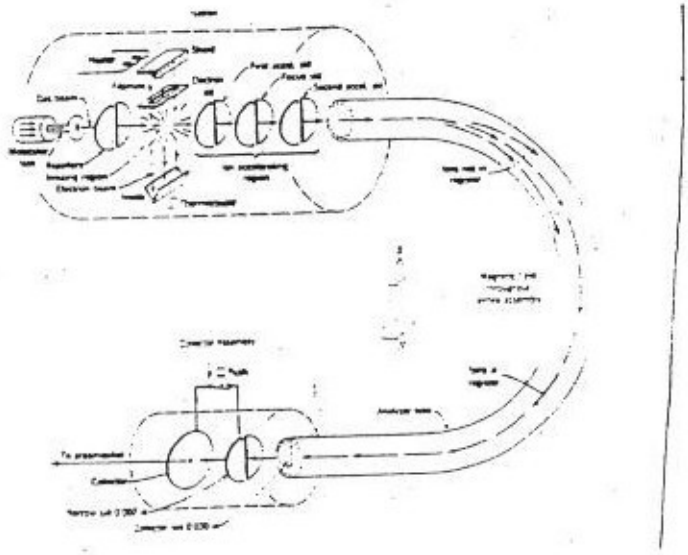


Figure 1: Basic Diagram for Mass Spectrometer, Magnetic Field Perpendicular to Page

The gaseous sample being tested was analyzed using mass spectrometry. Based on this procedure, the following concentrations were calculated:

Polymer Name	Percent of Total %**
Polyethylene	45
Polystyrene	12
Polypropylene	10
Phenolic	10
SBR	5
Other (PMMA, Polycarbonate, Epoxies, ABS, PTFE, Polyurethanes, Acetal, Fluorinated Polymers)	18

** *Disclaimer: The evaluator concedes the possibility of inaccuracy in these values based on inexperience.*

Figure 2: Calculated Concentrations based on Mass Spectrometry Analysis

Observations

Observations recorded during testing included the following:

- The sample burned with a red-orange flame with very little blue.
- Plastic self-extinguished if flame was removed for longer than 3 seconds.
- The burned plastic formed a thin layer(≈2 mm) of char; scraping it away revealed unburned plastic.

Analysis

The sample's total chromatogram can be seen in the appendix on page 3. For analysis purposes only eleven of the major peaks were selected for in-depth study. The breakdown of seven of these peaks, including the computer's library comparison, is included in the Appendix on pages 4-10. The peaks not included were omitted because of redundancy. Specifically, peak #4 was similar to peak #2, peaks #8 and #9 were similar to #1, and peak #11 was similar to peak #10.

The percentage results recorded previously were obtained by first determining the principle component in each peak. This component was assigned a numerical value equal to the calculated peak areas provided by the computer. When all the peaks were evaluated in this fashion, percentages were calculated in the normal manner. For example; the presence of SBR was identified in only one peak, peak #3. The peak area for peak #3 was provided as 165,132. This was 5% of the total area for the entire chromatogram and therefore SBR is listed as being 5% of the total composition.

Regardless of the exact compositional nature, it is evident from the breakdown of each peak that the sample is highly aromatic in nature. It is also evident that fluorine, or other heavier-than-air gases, is present by its self-extinguishing nature. This agrees with the computer's frequent suggestion of fluorinated compounds, but analysis of the molecular weights involved could not verify this fact. The absence of chlorine (and thus PVC) as one of these gases can possibly be explained by its removal with the CH_2Cl_2 solvent peak.

inexperience of the technician and, extending from that fact, the inability to clearly distinguish compounds from the products of combustion. The dominant combustion products were naphthalene and acenaphthylene, but various polymer segments were reminiscent of the listed polymer chains and thus are included under this title.

In the way of speculation, the presence of phenolic insinuates it could have been introduced as a binding material. The presence of an epoxy or polyurethanes as possible binders did not appear as significantly as phenolic, which support this hypothesis.

Availability

The ability to operate the machines necessary for such analysis requires significant training and skill. Most small businesses may never be able to afford a full-time analyst because of the cost. Larger companies (Dupont, FMC, Huntsman, etc.) can and do hire people who do this type of work. For businesses who cannot, there are companies set up who perform analysis for a small fee.

The machines themselves are expensive. A quick check places the price range on these machines from $12,000 (Packed column chromatograph) for small lab sizes up to as much as $160,000 (Differential Scanning Calorimeter) for large labs when it includes optional equipment.

Conclusion

There are many analysis techniques available for determining the plastic content of plastic wood. Most of them are cost prohibitive for small companies, but information can be obtained from consultants. The reasons companies need to consider this operation include property characteristics, design compatibility, health and safety factors, and it is the best means available for reverse engineering a competitors product.

Procedure: GC-MS Analysis

Equipment

GC Machine: Varian Model 3400
MS Machine: Finnigan MAT Model 700
MS Library: Finnigan MAT, 1986, Version D
Kitchen Matches
250 mL Pyrex Erlenmeyer Flask with Stopper
10 uL Syringe, Teflon tipped & gas tight
15 mL Dichloro Methane (CH_2Cl_2) Nanograde
Safety Hood
3 x 1 inch Sample of Plastic Wood
50 mL Pyrex Beaker
Graduated Cylinder

Preparation

1. Cut two equal 45° angles forming a point on one end of the plastic wood sample.
2. Set up the ring stand and clamp inside the hood.
3. Have all material within easy reach.

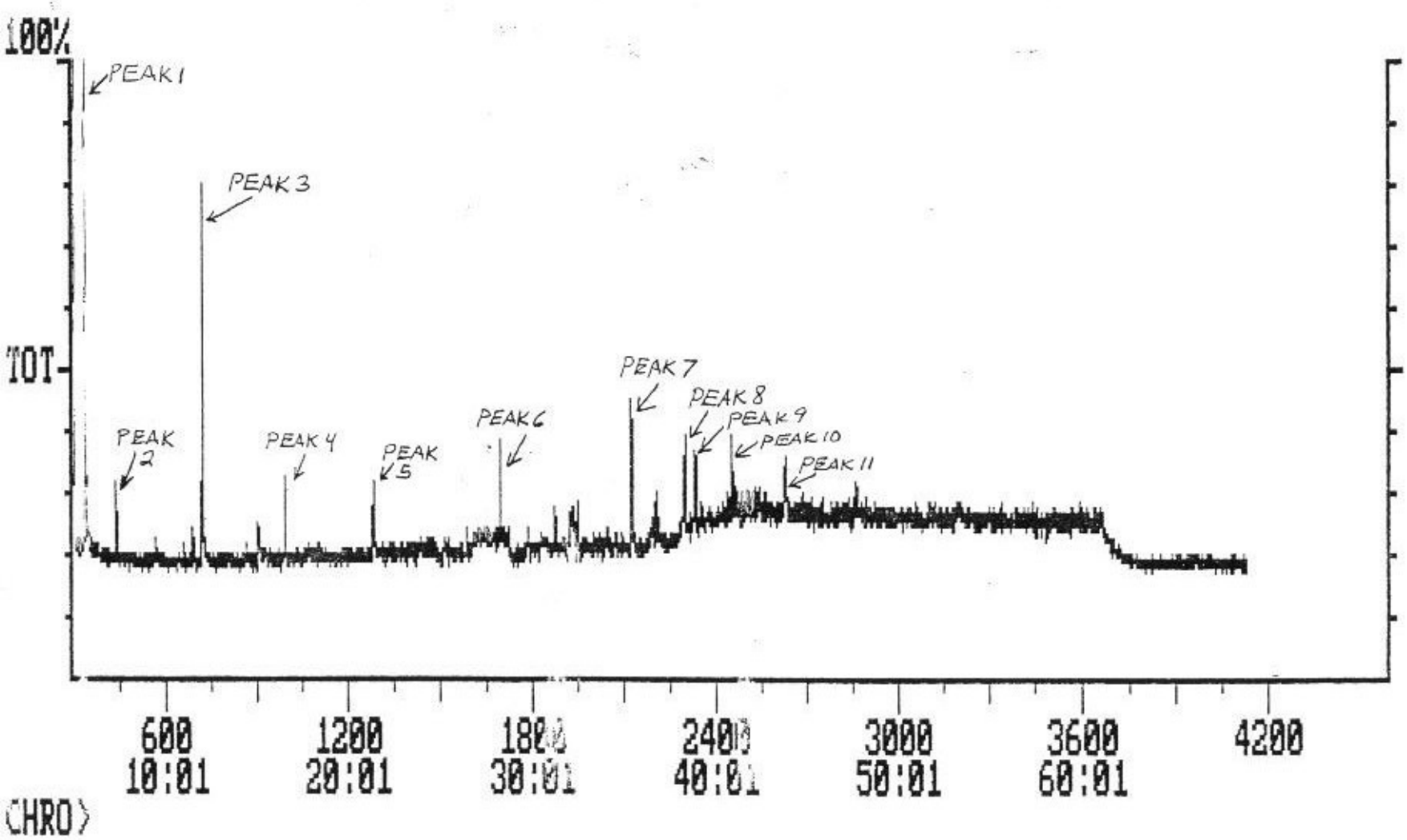
Procedure

1. Place prepared sample in the clamp with the pointed end down at a 45° angle from vertical.
2. Using kitchen matches, light the pointed end of the sample.
3. After the smoke from the match has cleared, invert the 250 mL flask over the burning sample, continue holding match under sample in order to supply heat. Light new matches away from sample in order to avoid collection of soot. The flask should be held in such a way to capture as much smoke from the plastic as possible.
4. When a thick, false atmosphere has been collected, quickly close the flask.
5. Extinguish the burning sample.
6. Measure out 15 mL of the Dichloro Methane into the 50 mL Beaker. Be sure this step is performed under the hood where the fumes can not collect.
7. As quickly as possible, open the flask and pour in the (CH_2Cl_2). Replace stopper.
8. Agitate the solution for 15 minutes by hand. At the end of 15 minutes check the color of the solution. It should have a cloudy gray appearance. If not, continue agitation.
9. When color is acceptable, carry remaining materials to where GC-MS machine is located.

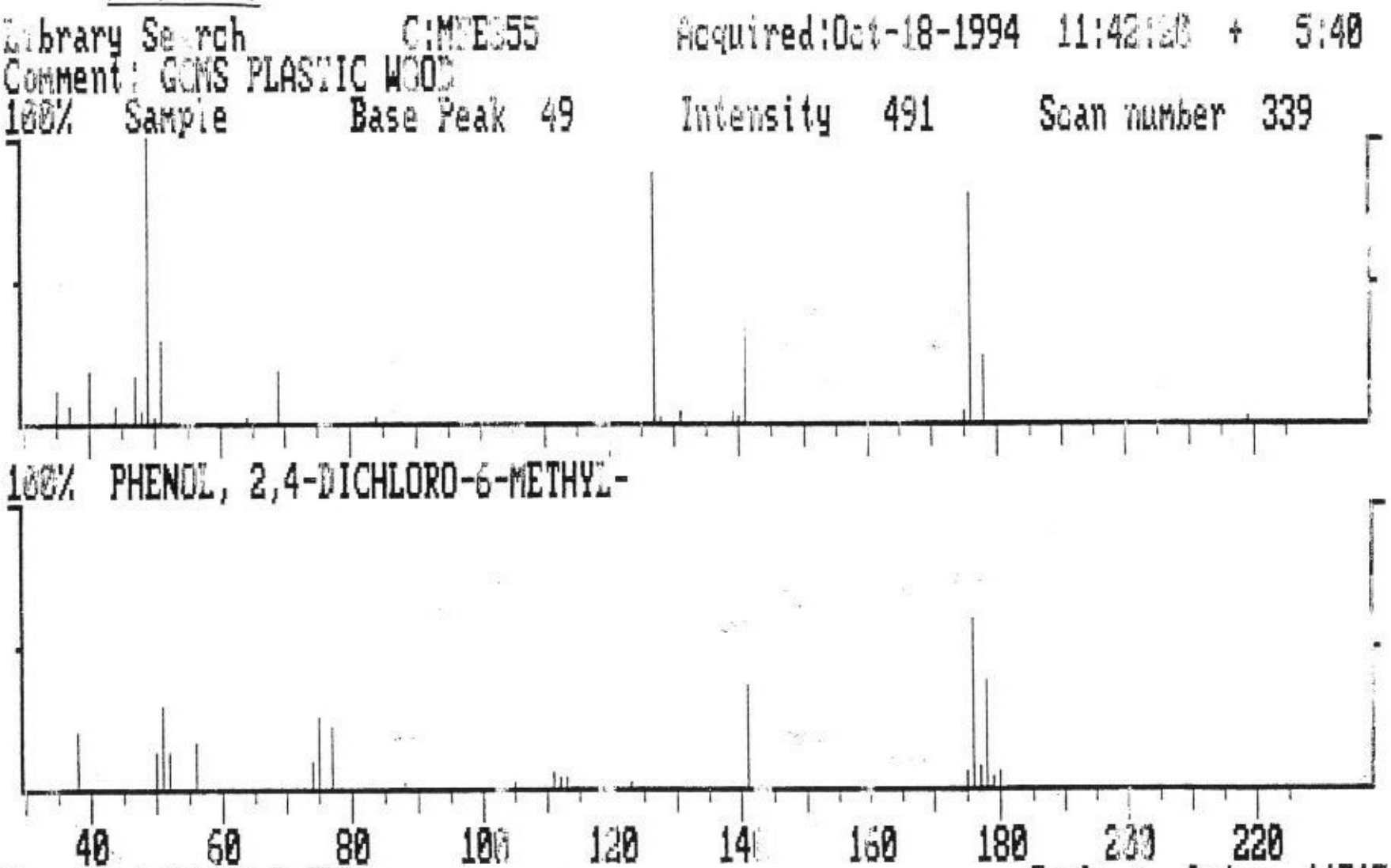
Procedure (continued)

10. Make parameter selection on the machine. (Suggest a 45 minute total run time for capture of possible longer, unburned polymer strands). Eclipse the beginning 100 seconds to allow for passage of solvent peak. Make evaluations on the shortest time intervals available.
11. Using syringe, measure 2 uL of solution and inject into appropriate port on machine.
12. When completed, save as many backups as deemed necessary.

Chromatogram C:MFE355 Acquired: Oct-18-1994 11:42:20
Comment: GCMS PLASTIC WOOD
Scan Range: 301 - 4060 Scan: 304 Int = 105895 @ 5:05 RIC: 100% =437877



PEAK #1



PEAK #2

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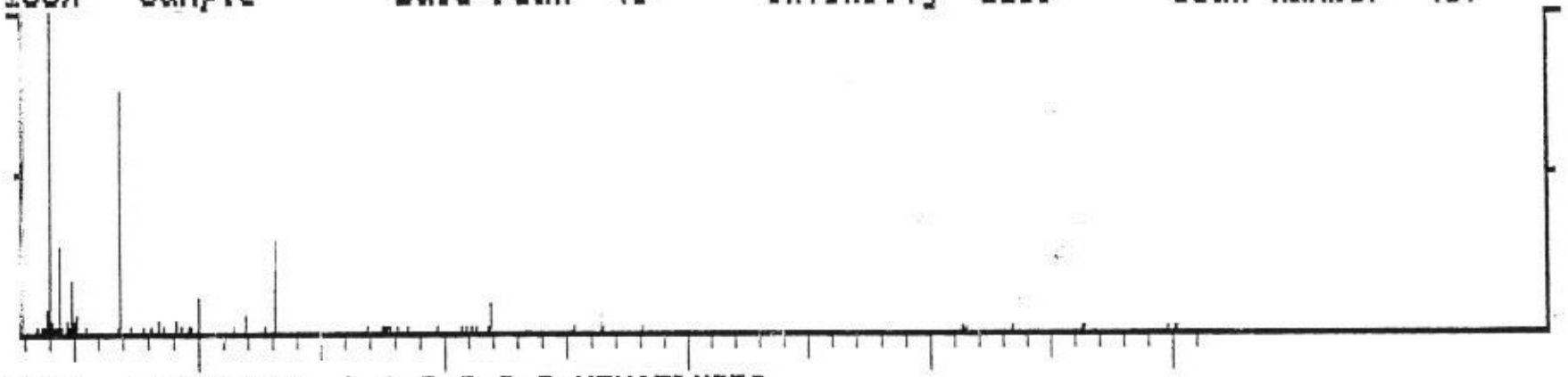
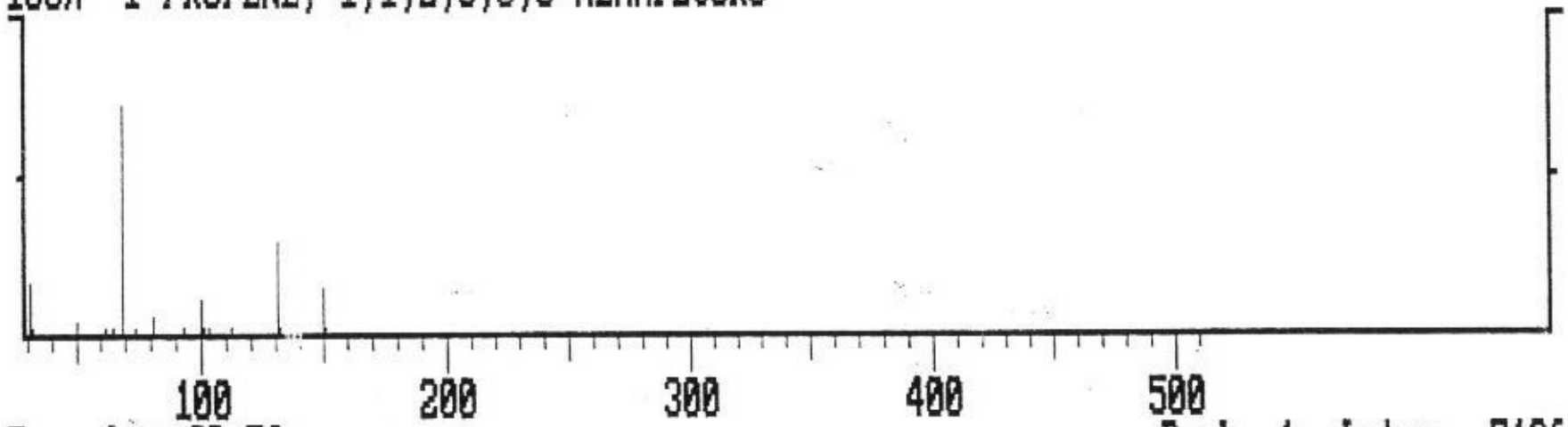
100% Sample Base Peak 40 Intensity 1159 Scan number 437



100% 1-PROPENE, 1,1,2,3,3,3-HEXAFLUORO-



Formula: C3.F6. Rank 1 Index 7126
Molecular weight 150 Purity [illegible] Fit [illegible] Rfit [illegible] Cas# 0-00-0
LIB (Purity, mass range 30 - 507, weight range 50 - 200)

PEAK #3

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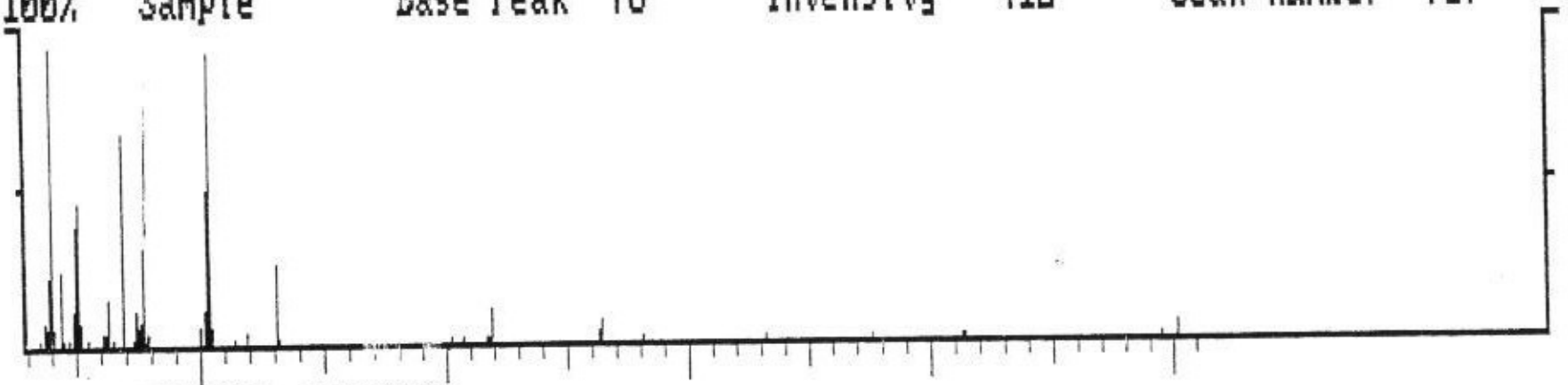
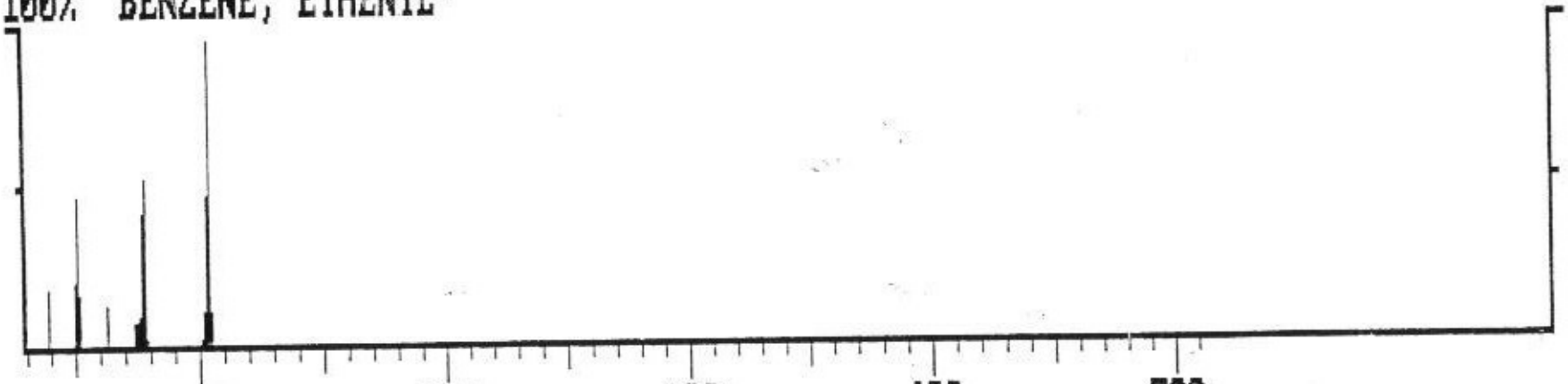
Comment: GCMS PLASTIC WOOD
100% Sample Base Peak 78 Intensity 412 Scan number 717



100% BENZENE, ETHENYL-



Formula: C8.H8,
Rank 3 Index 1613
Molecular weight 104 Purity [redacted] Fit [redacted] Rfit [redacted] Cas# 0-00-0
LIB (Purity, mass range 30 - 507, weight range 50 - 200)

PEAK #5

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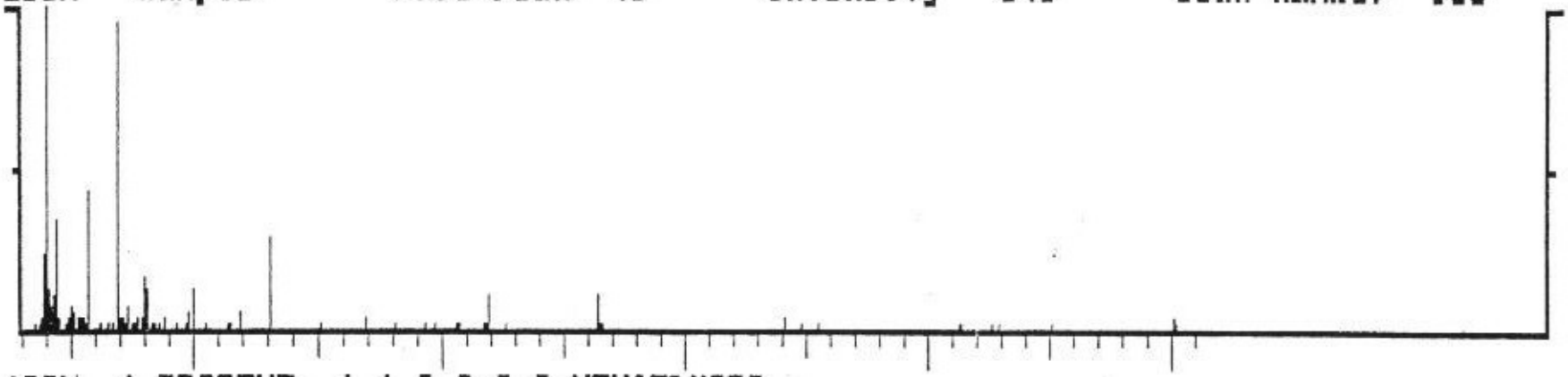
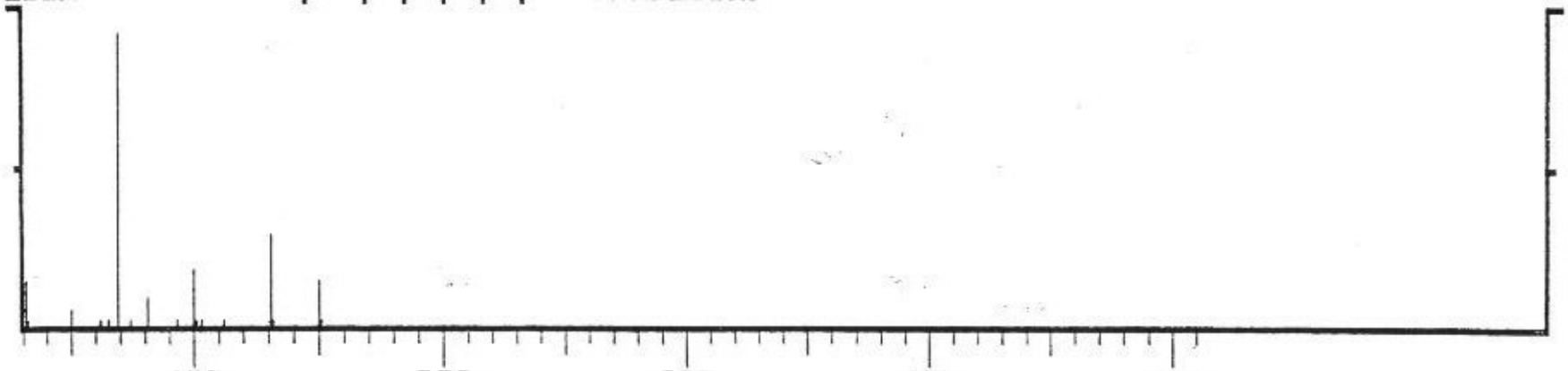


100% 1-PROPENE, 1,1,2,3,3,3-HEXAFLUORO-



Formula: C3.F6, Rank 1 Index 7126
Molecular weight 150 Purity [illegible] Fit [illegible] Rfit [illegible] Cas# 0-00-0
LIB (Purity, mass range 30 - 507, weight range 50 - 200)

PEAK #6

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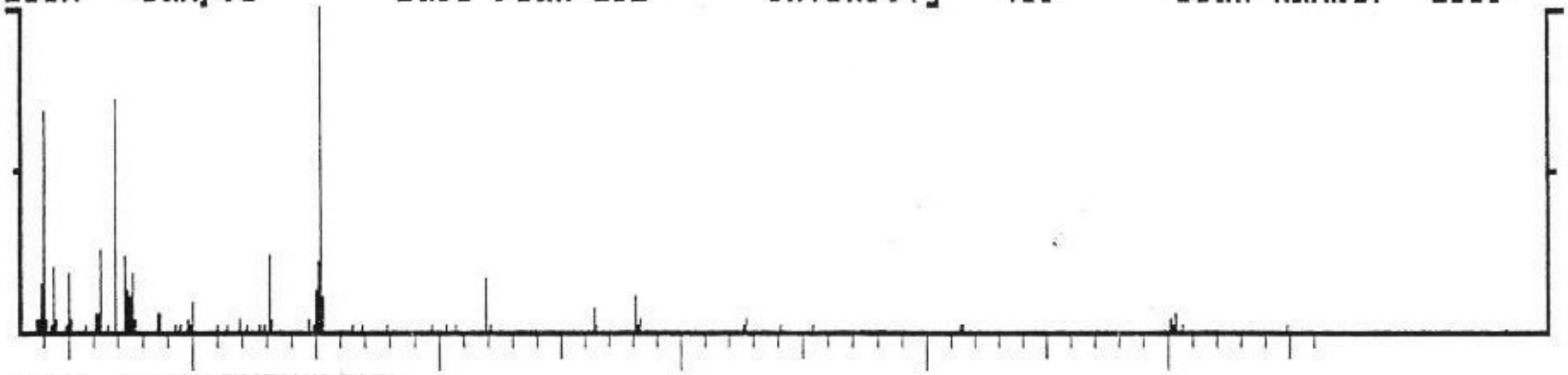
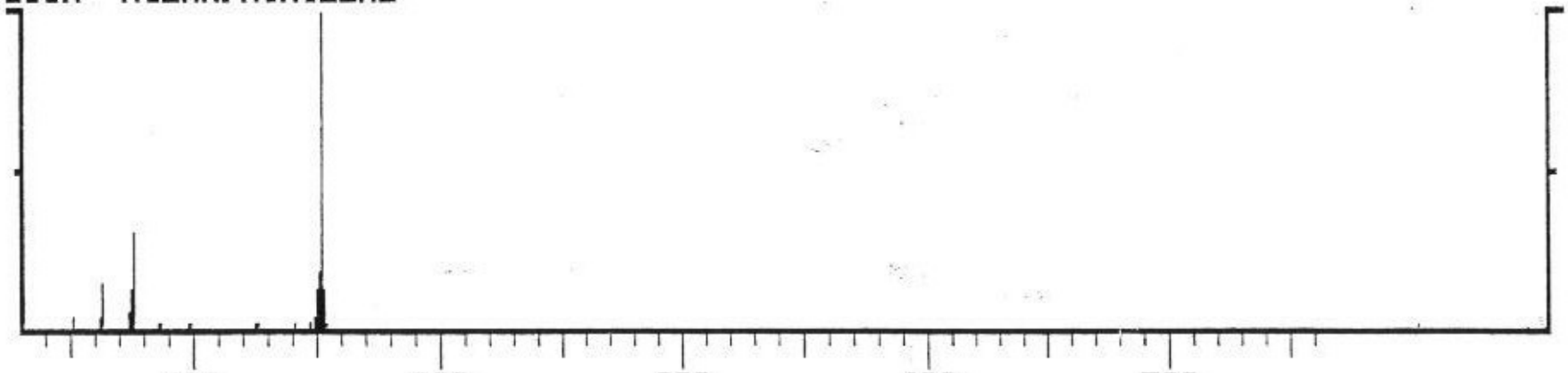


100% ACENAPHTHYLENE



Formula: C12.H8. Rank 1 Index 7462
Molecular weight 152 Purity [illegible] Fit [illegible] Rfit [illegible] Cas# 0-00-0
LIB (Purity, mass range 32 - 554, weight range 50 - 200)

PEAK #7

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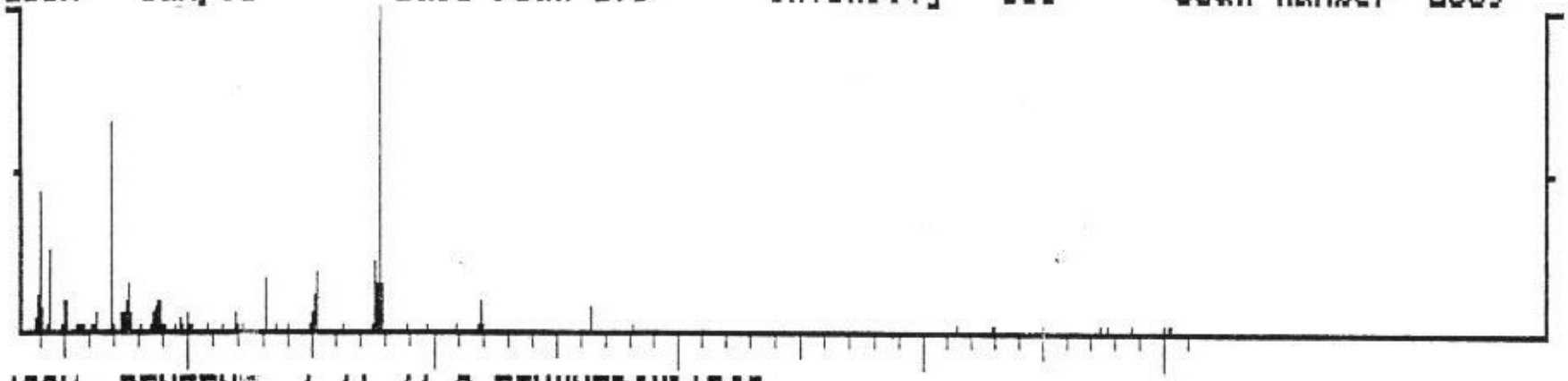
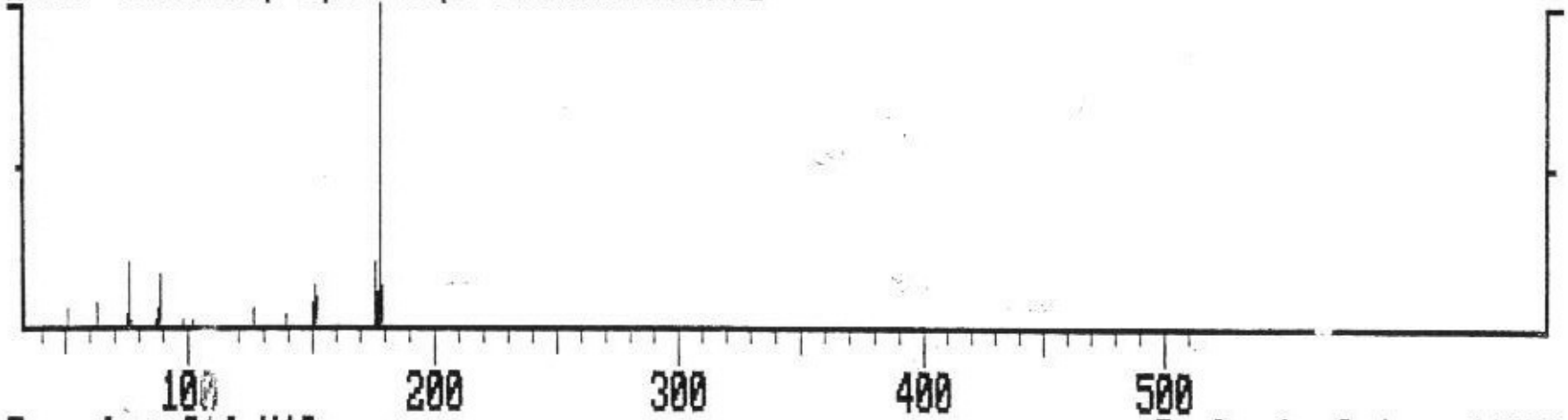


100% BENZENE, 1,1'-(1,2-ETHYNEDIYL)BIS-



Formula: C14,H10, Rank 1 Index 11971
Molecular weight 178 Purity [illegible] Fit [illegible] Rfit [illegible] Cas# 0-00-0
LIB (Purity, mass range 33 - 508, weight range 50 - 200)

000310

PEAK #10

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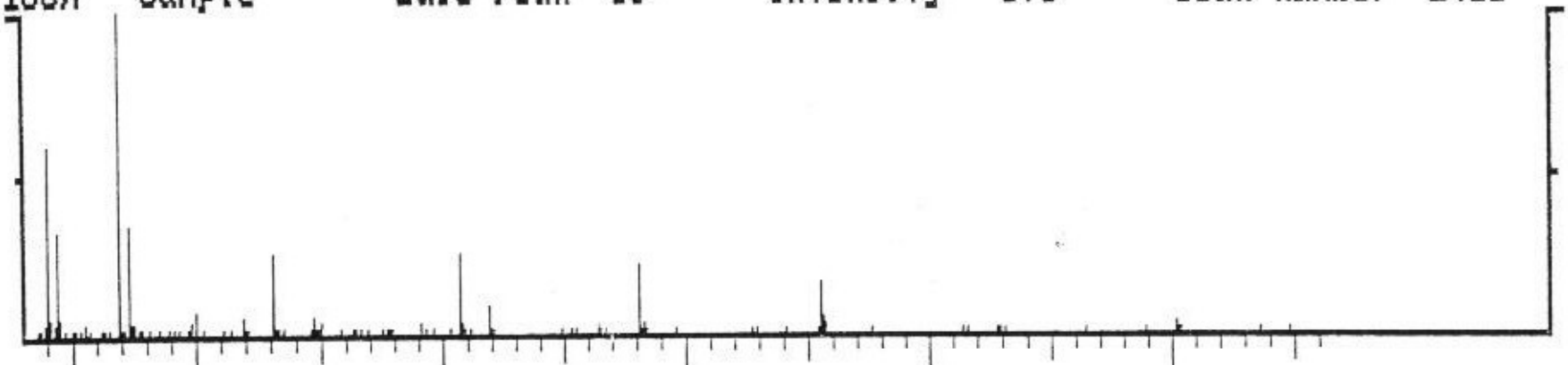
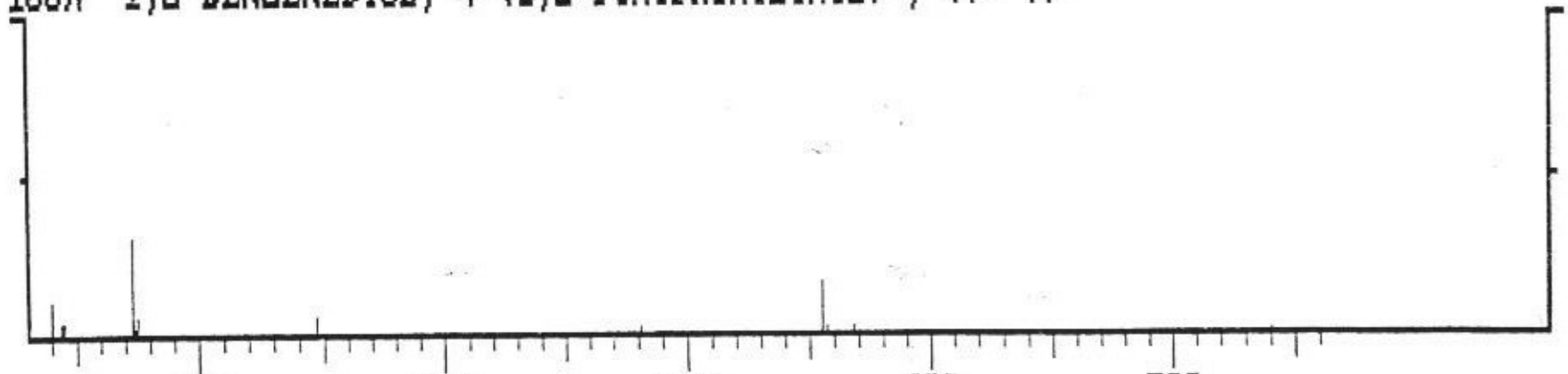


100% 1,2-BENZENEDIOL, 4-(1,2-DIHYDROXYETHYL)-, (.+-.)-



Formula: C8.H10.O4, Rank 1 Index 10819
Molecular weight 170 Purit[redacted] Fit[redacted] Rfit[redacted] Cas# 0-00-0
LIB (Purity, mass range 31 - 553, weight range 50 - 200)

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